

Ramagopal V. S. Uppaluri
Chandan Das
V. V. Goud
R. Anandalakshmi *Editors*

Agro and Food Processing Technologies

Proceedings of NERC 2022



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Ramagopal V. S. Uppaluri · Chandan Das ·
V. V. Goud · R. Anandalakshmi
Editors

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 Springer

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Foreword

It is a matter of great satisfaction for me that Indian Institute of Technology Guwahati successfully hosted North-East Research Conclave (NERC) 2022 during May 20–22, 2022. The NERC 2022 was conducted on the theme “Sustainable Science and Technology”. Concurrently, Assam Biotech Conclave (ABC) was also organized during May 21–22, 2022. Both the events attracted huge participation from policy-makers, researchers, industrialists, the army and students. Even the participation of schoolchildren was overwhelming.

NERC and ABC had many events including panel discussions, exhibitions, keynote lectures, competitions and paper presentations. Presentation of technical papers forms the core of any research conference. NERC attracted 879 research papers on various themes covering science, technology and humanities. Out of these, some select papers have been published by Springer Nature in the form of 15 volumes. These papers have been peer-reviewed and thoroughly edited by IIT Guwahati faculty members. I am sure that these volumes will prove to be excellent resource material for research. Most of the papers presented in these volumes highlight the special needs and aspirations of eight states of North-East India. I congratulate and thank the authors, reviewers, editors and publisher for bringing out the proceedings.

Motivation for organizing NERC came from none other than Honorable Minister of Education, Government of India, Shri Dharmendra Pradhan Ji. It helped to bring policy-makers, researchers, industrialists, academicians, students and children into one forum. It is perhaps the rarest Conclave covering almost all possible research themes. For better readability, the proceedings have been divided into 15 volumes, but each volume reflects diversity in terms of topics and researchers. The only common thread is the sustainable development of North-East India. Invariably, Sustainable North-East India is a prerequisite for sustainable India and the whole world. In that sense, these 15 volumes will serve guiding and stimulating light for all the stakeholders of the development. I am pleased to dedicate these volumes to our nation as a part of Azadi ka Amrit Mahotsav.



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Preface

Northeast India is a well-known region for its rich flora, fauna and endemic plant species possessing promising functional and medicinal properties. The region hosts horticultural resources such as seasonal and perishable fruits, vegetables, aromatic plants and endemic spices. To further enhance the economic security and livelihood of the region, agro and food processing technologies are inevitable to either enhance the shelf life or customize value-added product design and development. Agro and food processing technologies hold the key to the region's economic security and sustainability in a developing country such as India. Conventional approaches in agro and food processing technologies mimic the established processes and products in the landscape of a technology sustained landscape of Western countries. However, the customization of research methodology for the region's endemic produce needs to be emphasized for the broader application of commercial technologies and thereby enhance the transformation of agricultural produce into affordable consumer food products. To do so, scientific and technological research strategies are the need of the hour.

Three primary sub-themes are relevant in the broader domain of agro and food processing technologies. These are (a) Agro science and technology, a theme in which agricultural produce is explored from a physical and chemical parametric perspective and relates to the utility of relevant technology and scientific methodologies, (b) Agro process engineering, a theme that emphasizes the agricultural processing engineering through the optimization of process parameters of technologies such as drying, microwave, ultrasound and supercritical fluid extraction, and (c) Food product design and development, a theme that customizes toward the maturity of research methodologies for food product design such as chips, cookies and fruit leather. Notably, other sub-themes as well exist such as food process engineering, food process design and scale up and optimization of food process operations.

Invariably, the broad field of agro and food processing technologies involves a preliminary affirming of the science of agricultural produce in terms of constituents such as phenolics, phytochemicals, antioxidants and vitamins. Thereafter, the role of technology can be considered in the context of the retention of the valuables. Thus, the adopted research pedagogy in the above-mentioned major sub-themes advocates

the leverage of science and technology for the customization of relevant valuable constituents of agricultural produce and food raw materials and as value-added or valuable consumer food products. Product development through the achievement of either intermediate processed resources or end products through deeper formulation research remains important strategy in agro and food processing technologies. Considering these, the ongoing trend in this field is to customize research and development from the perspective of the development of affordable products with good consumer ratings.

In this volume entitled “Agro and Food Processing Technologies: Proceedings of NERC 2022”, the authors and editors strived to contribute thirteen book chapters in the mentioned sub-themes of agro science and technology, agro process engineering and food product design and development. In these sub-themes, the reader is introduced to mature characterization procedures being adopted for the agro and food intermediates and final products; low-cost and novel agro processing technologies for valuable constituent extraction from agro resources; mature and holistic research methodologies for value-added food product design and development. With rich and variegated research pedagogy, the volume will serve as a useful resource to understand and mature upon the knowledge frameworks in the broader field of agro and food processing technologies. Thereby, the presented research pedagogy is generic in nature and be conveniently applied to similar avenues in India and the world.

In the first sub-theme of Agro Science and Technology, scientific investigations that targeted the physical parameters of soybean cultivars and phytochemicals isolation from passion fruit bagasse have been delineated. These book chapters elaborate upon the role of science and technology in a systematic proliferation into agricultural process engineering and agro/food product design and development. The second sub-theme of agricultural process engineering presents a vivid description of several processing technologies such as thermal treatment, refractance window and tray drying, microwave drying, sonication, super-critical extraction and blanching, and tray drying for respective know-how in the fields of bio-chemical transformations in the production of black garlic, dried ginger and turmeric, drying kinetics and economic analysis of star fruit, papaya peel and pulp-based bioactive extraction, phenolics extraction from passion fruit rind and nutritional analysis-based abundant vegetable selection for soup mix formulation. Thus, the vast sub-theme aims to provide useful insights into the maturity of the adopted research methodologies for the retention or enhancement of nutritional constituents in desired media and/or shelf life enhancement of agricultural produce. In the final sub-theme of food product design and development, holistic process-product design approaches have been delineated to achieve fruit leather, grain- and fruit flour-based baked chips and cookies from horticultural produce such as guava, papaya and *mirika tenga*, atta/multigrain atta, aizong rice and squash, taro- and sweet potato-based composite flour, and squash- and grain flour-based cookies. In this sub-theme, formulation research and process optimality have been addressed in conjunction with the product characterization techniques for the development of nutritionally rich ready-to-eat food products.

In summary, this volume of Agro and Food Processing Technologies: Proceedings of NERC 2022 provides a detailed account of the quantitative and qualitative

studies of associated technologies for the processing of the horticultural produce of Northeast India. Notable benefits of the volume have been envisaged as follows. Firstly, for a business entrepreneur desiring to establish or venture into the affirming of relevant processing technologies for Northeast India, the volume will serve as a useful resource to vividly understand and realize associated techno-economic factors and indices. Secondly, for a research scholar aiming to enhance research and development in the agro-food sector of rural Northeast India, the volume will provide customized and mature research methodologies for systematic planning and execution of high-quality research in any chosen theme of the value-added processing of agricultural produce. Thirdly and most importantly, product design and development ranging from formulation to sensory analysis and associated research methodologies can be very thoroughly understood by studying the volume. In summary, this volume will serve as a very useful resource for agro-food scientists, technologists and entrepreneurs in their respective mission of scientific rationality in the value chain development of endemic produce and techno-economics of conventional and novel affordable processes for intermediate and final product development from agricultural produce and value-added product development, respectively. Thereby, betterment of the state-of-the-art, systematic and rigorous delineation into translational research and customization of the research and development for the development and economic security of Northeast India in a sustainable framework can be ensured.

Editorial Assistance: Prabhat Kumar Patel, Sneha Singh, Kumudhini Akasapu, Paushali Mukherjee and Preetisagar Talukdar.

Guwahati, India

Prof. Ramagopal V. S. Uppaluri
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About IIT Guwahati

Indian Institute of Technology (IIT) Guwahati established in 1994 has completed 25 years of glorious existence in 2019. At present, the Institute has 11 departments, seven inter-disciplinary academic centres and five academic schools covering all the major engineering, science, healthcare, management and humanities disciplines, offering B.Tech., B.De.s, M.A., M.De.s, M.Tech., M.Sc., and Ph.D. programmes. The institute presently offers a residential campus to 435 faculty members and more than 7500 students at present. Besides its laurels in teaching and research, IIT Guwahati has been able to fulfil the aspirations of people of the North East region to a great extent since its inception in 1994. The picturesque campus is on a sprawling 285 hectares plot on the north bank of the Brahmaputra, around 20 kms from the heart of the Guwahati city.

IIT Guwahati is the only academic institution in India that occupied a place among the top 100 world universities—under 50 years of age—ranked by the London-based Times Higher Education (THE) in the year 2014 and continues to maintain its superior position even today in various International Rankings. IIT Guwahati gained rank 37 globally in the ‘Research Citations per Faculty’ category and overall 384 rank in the QS World University Rankings 2023 released recently. IIT Guwahati has retained the 7th position among the best engineering institutions of the country in the ‘India Rankings 2021’ declared by the National Institutional Ranking Framework (NIRF) of the Union Ministry of Education. IIT Guwahati has been also ranked 2nd in the ‘Swachhata Ranking’ conducted by the Government of India. Recently, IIT Guwahati has been ranked as the top-ranked University in 2019 for IT developers by HackerRank in the Asia-Pacific region.

Among other frontier areas of research and innovation, IIT Guwahati is working towards augmenting critical science research initiatives in Genomics, Developmental Biology, Health Care and Bioinformatics, Flexible Electronics, Advanced Functional Materials, Sustainable Polymers, Rural Technologies, Renewable Energy, Artificial Intelligence, Disaster Resilience and Risk Reduction, and Water Resources and Management. In its silver jubilee year, IIT Guwahati is poised to scale newer heights through all-round growth and development.

Indian Institute of Technology Guwahati has dedicated itself to the cause of improving and empowering Northeast India through cutting-edge research, region relevant projects, innovations, individual and multilateral collaborations, and special initiatives. Being the only IIT in the entire Northeastern region, IIT Guwahati has an immense amount of responsibility to develop the region and empower the people of the region.

While the entire country is celebrating the “Azadi ka Amrit Mahotsav”—75 glorious years of Independence, and the great pride with which our nation of more than a billion people has been steadily growing today, IIT Guwahati is strongly committed to support that pace of growth for the entire NE so that we can keep pace along with the rest of the country. The specific areas of focus where IIT Guwahati has been contributing immensely to the region are:

- (a) Infrastructure development across multiple sectors.
- (b) Providing solutions for multiple natural disasters such as recurring floods, landslides, earthquakes, cyclones, hailstorms and other natural calamities,
- (c) Improving the education sector and creating opportunities for employment
- (d) Internet, telecommunication and cultural integration
- (e) Technological intervention in interdisciplinary areas
- (f) Healthcare services and education
- (g) renewable energy generation (solar, wind, biomass, hydro, geothermal)
- (h) overall industrialization, refining fossil fuels and setting up biorefineries.

Besides bringing in the state-of-the-art technical knowhow for most of the above sectors, the institute has been partnering with the local governments and enhancing the technological and educational interactions such that the next generation youth are empowered with knowledge, skills and necessary entrepreneurial ability. These measures in Assam as well as all other northeast states will usher in a new era of growth and the opportunities it will provide for interaction with the ASEAN countries as part of the Act East Policy of the Government of India will bring prosperity to this region.

Prof. Parameswar K. Iyer
Dean, PRBR, IIT Guwahati, India

North East Research Conclave-2022: Toward Sustainable Science and Technology

It is extremely important and imperative to have knowledge-driven growth based on innovation in the case of academic higher education institutes of high repute. The North Eastern region endowed with rich biodiversity comprises eight states. However, the climatic conditions, limited connectivity, lack of research infrastructure/institutes, territorial conflicts and the mountainous terrain of these regions are major impediments to the research ecosystem in the North-East. Quality higher education focusing on industry-academia collaboration and translational research is extremely beneficial for society. It has also been rightly pointed out by the Hon'ble Prime Minister Shri. Narendra Modi that, “*India cannot develop till Eastern India develops*”.



With this idea and as India marks 75 years of Independence, Indian Institute of Technology Guwahati organized “The North-Eastern Research Conclave” from 20th–22nd May 2022. This grand event was jointly conducted with Science, Technology and Climate Change Department and the Department of Education, Government of Assam at IIT Guwahati Campus.

The mission behind the conclave was to showcase the best R&D activities from educational and research institutions across North East India and to create an environment, conducive to development of local indigenous technologies and innovations, creating the scope and laying the foundation for entrepreneurship.

In order to attract people and spread awareness about the event, a roadshow was initiated from IIT Guwahati on 7th May 2022 in order to reach all the partnering academic institutes and make them an integral part of the mega event. The Director, IITG waved the NERC-2022 flag and sent off the road show vehicle from the institute. More than 400 students, staff and faculty participated actively in the roadshow.



A huge response was received by participants from throughout the country. The total no. of Participating institutions in this conclave included 7 IITs, 10 NITs, 5 IIITs and other CFTIs, 23 Research Labs, 17 Central Funded Universities, 47 other

Universities/Institutes along with about 100 schools. Eminent personalities from industries, start-ups, research councils and PSUs also joined in.

The presence of dignitaries from important Ministries was observed such as Shri Dharmendra Pradhan, Hon'ble Union Minister of Education and Minister of Skill Development and Entrepreneurship, GOI; Dr. Himanta Biswa Sarma, Hon'ble Chief Minister of Assam State; Dr. Ranaj Pegu, Hon'ble Minister of Education, Govt. of Assam; Dr. Rajkumar Ranjan Singh, Hon'ble Minister of State for Education, GOI; Dr. Subhas Sarkar, Hon'ble Minister of State for Education, GOI; Shri Keshab Mahanta, Hon'ble Minister of Science Technology & Climate Change, Government of Assam and many more.



The inauguration ceremony of the conclave was followed by the signing of an MoU between IIT Guwahati and the Government of Assam to establish 'The Assam Advanced Health Innovation Institute (AAHII)'. This MoU would prove to be a unique partnership between the Government of Assam and IIT Guwahati in order to set up a Research Institution to leverage advanced technologies to transform medical science. This joint venture company will be able to invite participation from intending parties including corporates/businesses/research institutions and philanthropic organizations.



The third edition of Assam Biotech Conclave 2022 was also held as part of NERC 2022. It brought together the Biotech Entrepreneurs, industry leaders, researchers, academicians, Government Representatives, policymakers, innovators and investors together on one platform to explore the possibilities of Biotechnology in North East India and to discuss the new opportunities in the transition.

Officers from the Indian Army also actively participated in the Conclave. A talk on “Atmanirbhar Bharat—Indian Army Initiatives towards Self Reliance” was delivered by Lt. Gen. D. S. Rana AVSM, YSM, SM General Officer Commanding, Gajraj Corps on 21st May 2022. The talk was aligned with the vision of the apex leadership of the Government of India and initiatives undertaken by the Indian Armed Forces with a focus on the integration of civil-military establishment in the field of self-reliance. He also elucidated that institutions such as IIT Guwahati which has many running research projects and elaborate student exchange and joint collaboration setup with a large number of Countries have the wherewithal to take up defence-related R&D and also facilitate delivery with Industry Partners. He also invited IIT Guwahati to participate in EAST TECH Symposium planned at Kolkata in July 2022. This led to the signing of an MoU between Indian Army Eastern Command and IIT Guwahati on 7th July 2022 during East Tech 2022. This would further impetus to Indigenisation and Raksha Atmanirbharta.



Royal Society of Chemistry, Global battery experiment was performed by more than 1300 students in three sessions starting from 20th May to 22nd May at IIT Guwahati. Along with the Global Battery Experiment, Creating Skilful Educators (Teacher training programme) was also conducted in parallel sessions. Students had arrived from various schools across Assam and other North-Eastern states.



The Guwahati Declaration was launched at the valedictory ceremony of the conclave by Shri Lok Ranjan, Secretary, Ministry of Development of North Eastern Region (DoNER), in the presence of Shri Kailash Karthik, Deputy Commissioner, Kamrup. The Declaration is intended to create a set of guidelines, through which individual as well as a collective responsibility to promote and encourage innovation at the grass-root level and strive to stimulate and execute indigenization and entrepreneurship, can be taken up.



Science, education, research and innovation are the four pillars on which the development, as well as the work culture of a nation, rests. This was well articulated by the promising number of Exhibitors being seen participating from all across the NE states in the NERC 2022. All the NITs, CFTIs and CFIs were allocated two stalls each, where the delegates showcased the working models of their inventions. Distinctive pavilions were arranged for IIT, NIT, CFIs and CFTIs. Excellent response was obtained from the Start-Ups all across the NE states. Federation of Industry Commerce of North Eastern Region (FINER) had partnered with NERC-2022 as an Industry Partner and they showcased 50 start-ups as a part of the Exhibition under the FINER Pavilion. Other significant organizations that came forward to showcase their allied R&D start-ups were the Oil and Natural Gas (Oil and Natural Gas Pavilion), Indian Army (Defense Pavilion) and NE-Railway (NE-Railway Pavilion).



Multifarious research work on topics of societal relevance was presented by researchers from different organizations/institutes. The presentations were conducted in oral and poster presentation modes. The thematic areas for these presentations were part of some of the Sustainable Development Goals (SDGs) such as SDG-3: Good health and wellbeing; SDG-7: Affordable and Clean Energy; SDG-9: Industry, Innovation and Infrastructure; SDG-11: Sustainable cities and communities and SDG-12: Responsible consumption and production. Some of the papers highlighted environmental sustainability, efficiency and management issues, which are important to be presented in the case of North East regions. Two awards were given under each technical category for these presentations. Overall the technical sessions were a grand success due to the active cooperation from editors, chairpersons of all the sessions and student volunteers of IITG.



The government of India has taken various steps to encourage women in the field of science and technology. In this line, the IIT Guwahati Woman Researcher Award was approved to recognize the contribution of women Faculty members of IIT Guwahati fraternity. This prestigious award was conferred to Dr. Latha Rangan who is a Senior Professor in the Department of Biosciences and Bioengineering, Indian Institute of Technology Guwahati, India. Prof. Rangan has played a key role in Plant Biotechnology and Sustainable development and especially in the areas of energy security, food security and medicinal crops.

The Conclave paved the way for creating mass awareness of Research and Innovation for developing a sustainable society. There was knowledge exchange and dissemination that led to the establishment of Centres of Excellence in Translational Collaborative Research and Innovation. This mega event led to the bridging of the gap between Industry-Academia and Creating Hand holding Pathways for setting up long-term collaboration for R&D innovations towards the goal of establishing sustainable NE India. The Conclave brought together over 8000 participants including Hon'ble Ministers, Official Bureaucrats, Eminent Professors, Scientists, Renowned Industrialist, School Children/Teachers and Others delegates. This revolutionized the R&D road map of all the NE states through various dissemination of policies which will benefit the sustainable development of all NE states in near future.

It is an honour and a moment of extreme pride for getting the NERC proceedings published in the prestigious Springer volumes. We would like to thank and acknowledge the globally active publisher Springer for helping us being able to publish the articles on 15 broad areas. We would also like to thank all the authors for their contribution to the grand success of NERC 2022 and wish them great success in all of their future endeavours.



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From the Desk of Chairman of Technical Committee of NERC 2022

North-East Research Conclave 2022 was successfully organized during May 20–22, 2022 with the participation of thousands of delegates. A total of 879 oral and poster papers were presented in the conference on 16 different tracks. The theme of the Conclave was Sustainable Science and Technology, which is very pertinent in the modern era of globalization. Science and technology has to address economic, environmental and social problems of the world. Technology and sustainability are not incompatible. In fact, technology can achieve the goal of sustainability, which also includes preserving our rich cultural heritage. Concurrently with North-East Research Conclave (NERC), Assam Biotech Conclave 2022 was also organized on May 21–22, 2022. These mega events were organized at Indian Institute of Technology Guwahati (IITG) in physical mode after two years of pandemic period. Along with IITG, Science, Technology and Climate Change Department and Department of Education, Government of Assam were also organizers of these events under the patronage of Shri Dharmendra Pradhan Ji, Honorable Minister of Education and Minister of Skill Development and Entrepreneurship in the Government of India, and Shri Himanta Biswa Sarma Ji, Honorable Chief Minister of Assam.

It is a matter of great pleasure that Springer Nature is publishing the select papers from the conclave in 15 volumes. These are Advanced Functional Materials, Low Cost Manufacturing Technologies, Agro and Food Processing Technologies, Artificial Intelligence and Data Science based R&D interventions, Conservation of Biodiversity in the North Eastern States of India, Disaster Management, Healthcare Research and Related Technologies, Innovative Design for Societal Needs, Policies for Research and Innovation, Research and Innovation for Sustainable Development Goals, Sustainable Environment, Sustainable Energy Generation and Storage, Sustainable Transportation and Urban Development, Teaching and Learning Technologies, Technologies for Rural Development. These volumes are useful archival and reference materials for policy-makers, researchers and students.

As the Chairman of Technical Committee, I am thankful to all Editors of all volumes, reviewers and student volunteers who have put tireless efforts to review, select and edit the papers of respective divisions, overcoming the time-constraint.

Support provided by Convener, Prof. Vimal Katiyar, Dean R&D, IITG, and Co-Conveners Professor Subhendu Sekhar Bag, Associate Dean R&D, IITG and Shri Kailash Karthik N, IAS is commendable. It is difficult to express words of gratitude for the Director, IITG, Prof. T. G. Sitharam who has been motivating and guiding all the teams of NERC 2022 and ABC 2022.

Uday S. Dixit
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Contents

Agro Science and Technology

Assessment of Biological Activities of Various Phytochemicals Isolated from Passion Fruit Bagasse	3
Sukumar Purohit, Emiko Yanase, Lingaraj Sahoo, and Vaibhav V. Goud	

Indigenous Soybean Cultivars of North-East India: Source of Protein and Product Development for Climate-Smart Foods	17
Neha Jha, Siddhartha Singha, and Munmi Borah	

Agro Process Engineering

A Review of Bioactive Compounds and Maillard Reaction-Based Products Generated During the Thermal Treatment of Garlic	35
Ajay Kumar Shakya and Chandan Das	

Optimality of Process Parameters During Refractance Window Drying of Ginger	57
Pankaj Parmar, Preetisagar Talukdar, Srimonti Dutta, and Ramagopal V. S. Uppaluri	

Techno-economic Efficacy of Refractance Window Dried <i>Curcuma Longa</i>	81
Preetisagar Talukdar, Imdadul Hoque Mondal, and Ramagopal V. S. Uppaluri	

3E (Energy, Exergy and Economic) Analyses and Kinetic Studies on Microwave Drying of Star Fruit	99
Sayan Mandal, Koushik Jena, and V. Prabu	

Recovery of Bioactive Constituents from Unripe Papaya Peel and Pulp Using Ultrasound Assisted Extraction: Optimization of Process Parameters	119
Khalid Mehmood Wani and Ramagopal V. S. Uppaluri	

Extraction of Phenolics from Yellow Passion Fruit Rind Using Supercritical Carbon Dioxide Extraction	141
Kakali Borah, Rupesh Kumar, and Vaibhav V. Goud	
Nutritional Efficacy Based Vegetables Selection for the Development of Ready to Cook Soup Mix Formulations	157
Kumudhini Akasapu and Ramagopal V. S. Uppaluri	
Food Product Design and Development	
Sensory and Functional Qualities of Fruit Leather Prepared from Guava (<i>Psidium Guajava</i>), Papaya (<i>Carica papaya</i> L.) and Mirika Tenga (<i>Parameria polyneura</i>)	195
Ginisha Kalsi, Luna Dutta Baruah, and Moloya Gogoi	
Formulation and Characterization of Atta/Multigrain Atta-Aizong Rice-<i>Sechium edule</i> Flour Mix Baked Chips	209
Srimonti Dutta, Pankaj Kalita, and Ramagopal V. S. Uppaluri	
Development and Sensory Evaluation of Cookies from Nutrient Rich Composite Flour	225
Kasturi Pusty, Mriganka Shekhar Borah, Ajita Tiwari, and Sheriful Alam	
Formulation and Characterization of Squash Enriched Cookies	241
Paushali Mukherjee, Rushikesh Rajendra Kudave, and Ramagopal V. S. Uppaluri	

About the Editors

Prof. Ramagopal V. S. Uppaluri holds B.Tech. (Chemical Engineering) from Andhra University, Visakhapatnam; M.Tech. (Chemical Engineering) from IIT Kanpur and Ph.D. (Process Integration) from the University of Manchester, England. After a brief tenure of postdoctoral fellowship at Robert Gordon University, Scotland, he joined IIT Guwahati and eventually became one of the youngest Professor and Professor (Higher Academic Grade) of IIT Guwahati. Till date, Prof. Uppaluri has guided 15 Ph.D. students at IIT Guwahati. Presently, he is supervising 20 Ph.D. students in diverse fields of rural food processing, polymer science and technology, solar energy applications, machine learning, bio-fertilizers, food product design and development, etc. He has published 116 international journal publications in diversified areas of chemical engineering and science. He has been awarded/filed 6 Indian patents in the field of enhanced oil recovery, noble metal composite membrane fabrication and nano-clustered palladium catalyst fabrication. He also completed several funded projects from DBT, CSIR, DST, DRDL, OIL and IOCL. His extra-curricular interests are in the field of Vedanta & Science and Consciousness Studies.

Dr. Chandan Das has received his Bachelors and Masters Degree in Chemical Engineering from University of Calcutta and Ph.D. in Chemical Engineering from IIT Kharagpur, India and currently is a Professor in the Chemical Engineering Department at IIT Guwahati. He has eighteen years of research and fourteen years of teaching experience. Prof. Das works on (i) Membrane Based Separation Technology; (ii) Food Engineering; (iii) Biomedical, (iv) Material Science; (v) Corrosion; (vi) Wastewater Treatment and (vii) Simulation and Modeling. He has guided, so far, 32 M. Tech. students and has been guiding 17 scholars for their doctoral degree. Dr. Das is the recipient of “Dr. A.V. Rama Rao Foundation’s Best Ph.D. Thesis and Research Award in Chemical Engineering/Technology” for the year 2010 from Indian Institute of Chemical Engineers (IIChE). Dr. Das has authored about 187 technical publications in peer reviewed journals (102) and proceedings (85). He has authored four books and eight book chapters. One of these is entitled “Treatment of Tannery Effluent by Membrane Separation Technology” in Nova Science Publishers, USA. He has three patents in his credit. Being associated with various research works

in the area of solid and liquid wastewater treatment, such as treatment of tannery wastewater using membrane separation technology, as well as removal of pollutants using micellar-enhanced ultrafiltration, Dr. Das has gained expertise in membrane separation technology for removing various pollutants from contaminated water and wastewater. Dr. Das has completed 7 sponsored project and 7 consultancy projects worth above INR 351 Lakhs (\$5,39,426) and two more are undergoing. Dr. Das has been admitted as prestigious FELLOW of Royal Society of Chemistry (FRSC), UK and also elected as Life FELLOW of IChE. He has authored three books.

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Agro Science and Technology

Assessment of Biological Activities of Various Phytochemicals Isolated from Passion Fruit Bagasse



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Abstract Non-selective dumping and burning of agricultural waste have caused severe health hazards and environmental pollution. However, these waste materials can be utilized sustainably as soil fertilizer, renewable energy feedstock, and valorization for essential nutrients and phytochemicals. Being an agrarian country, India produces a significant amount of agricultural waste, further contributing to a severe environmental hazard. Therefore, there is a dire need to address this serious issue for the sustainable utilization of agricultural waste. In the present study, passion fruit bagasse (seed) was processed to extract valuable phytochemicals following several bioorganic chemistry techniques and then characterized for their identification and biological activities (in vitro antioxidant activity and antibacterial activity). ^1H NMR, ^{13}C NMR, HPLC, and mass spectrometry confirmed three vital phytochemicals of which two are stilbenoids and the other one is dihydroxy benzoic acid. These isolated and identified compounds were further subjected to in vitro antioxidant activity using DPPH assay, where the stilbenoid compounds showed superior results than the dihydroxy benzoic acid. However, these isolated compounds showed significantly higher antioxidant activity than other tested standard compounds like ascorbic acid, ferulic

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acid, and quercetin. This study indicated that futile waste passion fruit bagasse could be bio-processed for isolating value-added phytochemicals. Further, because of the crucial biological activity possessed by the isolated phytochemicals, their application in the food, health, pharmaceutical, and cosmetic sectors would be very promising.

Keywords Waste valorization · Passion fruit · Preparative HPLC · Antioxidant activity · Antidiabetic activity

1 Introduction

Generally, the human antioxidant system balances antioxidant and reacting oxygen species like hydroxyl and superoxide inside the body. However, an unhealthy lifestyle and exposure to UV radiation accelerate reactive oxygen species (ROS) formation inside the body. Hence, the body's defense system could not maintain balance. Free radical generation in cells causes oxidative stress that induces many life-threatening diseases such as cancer, diabetes, and senescence. These free radicals attack DNA, proteins, and lipids, promoting narcosis and cell death. To encounter these issues, external antioxidants in natural extracts or oxidative quencher medications are in high demand nowadays. These natural extracts are vitamins (ascorbic acid, α tocopherol), polyphenols (phenolic acid, stilbene, flavonoids, etc.), anthocyanins, carotenoids, etc. Polyphenols are well known for their anti-aging, antioxidant, antidiabetic, and anticancer properties. Previously, many plant extracts such as *Anchusa italica* [1], *Clinacanthus nutans* [2], and *Avicennia marina* [3] have also been analyzed for these activities.

Diabetes, more precisely type 2 diabetes, is of great health concern worldwide because of the concerning statistics [4]. When the pancreases cannot provide sufficient insulin to the body, carbohydrate metabolism gets delayed, resulting in an increase in sugar levels in the blood. Furthermore, reacting oxygen species also cause diabetes by accelerating oxidative stress in the body. Non-enzymatic glycosylation [5] is one of the possible mechanisms for oxidative stress-related diabetes. However, natural antioxidants isolated from different fruit, vegetables, and waste by-products (peel, seed, pomace, bark, leaves, root, etc.) can help reduce oxidative stress-related diabetes in the body.

Passiflora edulis var. *flavicarpa* (yellow passion fruit) is a not much-explored fruit in India. It is mostly consumed for its delicious juice, and the rest peel and seeds are discarded as waste or used for cattle feed [6]. The application of juice and rind has been considered by many research groups for food product application. Different varieties of passion fruit juice, rind, and their application in the field of food have been mentioned in Table 1. Passion fruit (PF) seed is one of the major waste by-products discarded directly by the beverage industries after juice extraction. They do not process the waste by-products for waste management which eventually increase environmental pollution. So far, for waste valorization, different sustainable approaches have been explored. Therefore, different methods for waste handling

Table 1 A summary of the applications of passion fruit juice and rind in food product development

Species name	Plant part used	Application	References
<i>P. edulis</i>	Juice	Cardiac tonic	[9]
<i>P. quadrangularis</i>	Juice	Antibacterial	[10]
<i>P. edulis flavicarpa</i>	Juice	Aromatic profile	[11]
<i>P. edulis</i>	Juice	Functional meat product	[12]
<i>P. edulis flavicarpa</i>	Peel	Kefir drink	[13]
<i>P. edulis</i>	Peel and seed	Liquid yogurt	[14]
<i>P. edulis</i> f. <i>flavicarpa</i>	Peel	Aromatic spirit	[15]
<i>P. edulis</i>	Peel	Additive for spam like meat	[12]
<i>P. edulis</i>	Peel	Property enhancement of burger	[16]

have been highly studied over the last decade. Reuse and recycling of agro-waste for the development of value-added products are often addressed first in the waste management hierarchy. The reuse of agro-waste to produce commercial products is growing day by day and is often called a bio-refinery process. Products developed from bio-refinery are classified as bio-fuels, bio-fertilizers, bio-chemicals, food additives, pigments, etc. [7]. For example, vegetable and fruit waste by-products are considered to isolate various pharmaceutically active ingredients to mitigate severe life-threatening diseases such as diabetes and oxidative stress.

In one of our recent studies [8], many polyphenols including gallic acid, caffeic acid, ferulic acid, p-coumaric acid, quercetin, myricetin, etc. have been identified from the peel and seed of yellow passion fruit. Those phytochemicals displayed profound antioxidant and antibacterial properties. However, many unidentified HPLC peaks encouraged us to further phytochemical mining of other prevalent novel compounds in the seed of PF. Therefore, in this study, systematic isolation approaches including fractionation, column chromatography, and preparative HPLC methods were followed to achieve maximum yield of Piceatannol (PT) and Protocatechuic acid (PCA) were targeted using various bioorganic tools followed by the assessment of their in vitro antioxidant and antidiabetic properties.

2 Materials and Methods

2.1 Sample and Chemicals

Yellow passion fruits (YPF) were collected from a local market in Manipur, India. Yellow passion fruit seeds (YPFS) were separated from the juice sacs and further dried and properly stored. All the chemicals used for extraction and fraction were of analytical grade and were procured from Nacalai Tesque Co. (Kyoto, Japan). Diaion HP20SS used for reverse phase column chromatography was purchased from Mitsubishi Chemical Co., Tokyo, Japan. Antioxidant standards were purchased from Sigma Aldrich. The α -amylase and α -glucosidase enzymes were purchased from Sigma Aldrich.

2.2 Extraction and Fractionation of Yellow Passion Fruit Seeds

Initially, the seed sample was extracted with n-hexane to remove the oleaginous substances. The hexane-defatted seed sample (37.5 g) was subjected to Soxhlet extraction using a methanol:water ratio of 80:20 for 48 h. Later, the hydro-alcoholic extract was concentrated using rotary evaporation. Finally, the dried extract was stored in a 4 °C refrigerator until future use. Fractionation of the extract was carried out using 6.5 g extract and with different solvents such as hexane, dichloromethane (DCM), ethyl acetate (EtOAc), and butanol (BuOH). Two volumes of each solvent were added to the separating flask for the complete extraction of various phytochemicals in respective solvents. Afterwards, the fractions were dried using a vacuum evaporator and stored at 4 °C. The EtOAc fraction was further treated with an equal volume of 1 M NaOH followed by an equal amount of 1 M HCl to achieve maximum phenolic acid in the said fraction. The polyphenol-rich HCl-treated EtOAc fraction was further evaporated, and the dried fraction was stored in a 4 °C refrigerator for later use.

2.3 Separation and Isolation of Purified Compounds

Major peaks from the EtOAc and BuOH fractions were identified using a gradient HPLC (Jasco 4000 series HPLC unit, PDA detector, and 5C₁₈-MS-II Cosmosil Packed column at 35 °C). A volume of 5 μ L of a sample (10 mg/ml stock) was injected with a solvent system of 5–40% acetonitrile and 0.5% formic acid for 0–30 min. Major peaks were identified at 254 and 330 nm. Further, the major peaks containing fractions were subjected to reverse phase column chromatography (RPCC) using HP20SS ion exchange to separate PT and PCA. The column was eluted sequentially

from 20% up to 100% with methanol and different fractions were collected. All the fractions collected from the RPCC were analyzed through analytical HPLC, and the solvent system was optimized for further isolation through preparative HPLC. Further, the purified compounds (major peaks selected from the analytical HPLC) were isolated using a preparative HPLC (Jasco 875UV preparative HPLC coupled with UV detector) and Prep ODS Inertsil column (10 mm ID, 250 mm, GL Sciences Inc., Tokyo, Japan) using a column temperature of 35 °C with the optimized condition of 30% methanol with 0.5% formic acid at 254 nm. Required fractions of the samples (PT and PCA) were collected batch-wise. Finally, all eluent systems were mixed together and evaporated to obtain the purified samples. Finally, the purified compounds were stored in ambered color vials and kept at -20 °C in a refrigerator.

2.4 Characterization of Purified Compounds

The purified compounds such as PT and PCA were dissolved separately in CD₃OD and analyzed through ¹H and ¹³C-NMR (JEOL ECA 500 spectrometer, JEOL, Tokyo, Japan). The molecular mass of the purified compounds was obtained from a JMS-T100TD mass spectrometer (JEOL). The purified compounds were dissolved in a methanolic solution before carrying out mass spectrometry. The data were collected in ESI (electro-spin ionization) negative mode.

2.5 In Vitro Biological Activities of Piccatannol and Protocatechuic Acid

In vitro antioxidant activity of the purified PT and PCA was performed using DPPH and ABTS methods mentioned by Purohit et al. [8]. In brief, 3 mL of 2–10 µg/mL sample (dissolved in methanol) was mixed with 1 mL of DPPH (0.1 mM DPPH in methanol) following an incubation period of 30 min, and the absorbance was recorded at 517 nm. For, ABTS analysis, a sample of the same concentration was dissolved in 1 mL methanol and mixed with 3 mL of ABTS (diluted with water) following 20 min incubation, and the absorbance was recorded at 765 nm. The free radical inhibitory concentration of PT and PCA was further compared with six alternate standards such as gallic acid, caffeic acid, ferulic acid, ascorbic acid, p-coumaric acid, and quercetin.

The in vitro antidiabetic activity of purified PT and PCA was estimated by α-amylase and α-glucosidase activities. These were determined by following the methodologies outlined in the relevant prior art article [17]. In brief, for α-amylase activity, 1–5 µg/mL of sample (50 µL) was treated with 50 µL of α-amylase (0.5 mg/mL in 0.1 M PBS, pH 6.8) for 15 min at 37 °C. Afterward, 1% starch solution (50 µL) was added to the mixture and left for another 20 min at 37 °C. Then, 100 µL of DNS solution was added followed by boiling for 15 min. Then, the

samples were diluted with water (1 ml) and the absorbance was recorded at 540 nm. Acarbose was used as the standard. The inhibition percentage was estimated using the following Eq. 1:

$$\%Inhibition = \frac{(Abs_{Cont.} - Abs_{Cont.BG}) - (Abs_{Samp.} - Abs_{Samp.BG})}{(Abs_{Cont.} - Abs_{Cont.BG})} \times 100 \quad (1)$$

where Abs cont. refers to the absorbance of the control group, BG represents absorbance of background, and Abs_{samp} is the absorbance of the sample/standard compound.

The α -glucosidase activity of purified PT and PCA was also determined using a prior article [17]. In short, 1–5 μ g/mL of the sample or 100–500 ng/mL of the standard were mixed separately with α -glucosidase enzyme (50 μ L) and incubated for 15 min at 37 °C. After that, 1 mM of 4-Nitrophenyl- β -D-glucopyranoside (50 μ L) was added to the solution following an incubation period of 20 min at 37 °C. Then, 100 μ L of Na₂CO₃ was added to the mixture, and the absorbance was recorded at 405 nm. The inhibition percentage of α -glucosidase by PT and PCA was calculated using Eq. 1.

2.6 Statistical Analysis

Results reported for antioxidant and antidiabetic activities are presented as mean \pm SD of two independent experiments in duplicates.

3 Results and Discussion

3.1 Extraction and Purification of Piceatannol and Protocatechuic Acid

The fruit sample, seed, and purified compounds with their structures are presented in Fig. 1. The purified compounds were derived from hydro-alcoholic extraction of the defatted seed sample followed by fractionation, column chromatography, and preparative HPLC.

From the hydro-alcoholic extraction of YPFS, 7.2 g (19.2%) extract was recovered. Further, 6.5 g extract was subjected to fractionation using various solvents mentioned in the materials and method section. After the fractionation process, the EtOAc and BuOH fractions yielded maximum extract quantities, i.e. 1.54 and 2.68 g, respectively. To identify the tentative PT and PCA, both EtOAc and BuOH fractions were subjected to gradient HPLC. Their respective chromatograms are presented in

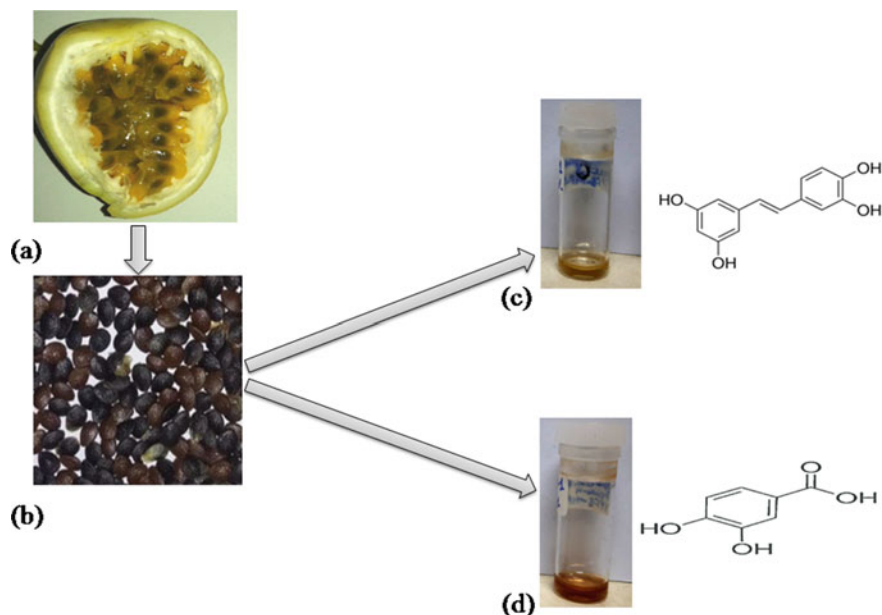


Fig. 1 Transverse section of yellow passion fruit (a), cleaned seeds from the juice sacs of passion fruit (b), purified Piceatannol along with its molecular structure (c), and purified Protocatechuic acid (d) along with its molecular structure

Fig. 2a, b. Further, the presumed PT was isolated using a preparative HPLC (Fig. 3a and the chromatograms for purified PT are presented in Fig. 3b. The intensity of presumed PCA was found to be very low in HPLC analysis (Fig. 3c). Hence, the compound was identified using a polyphenol standard protocatechuic acid (Fig. 3d), and the chromatogram of the purified PCA is presented in Fig. 3e.

3.2 Identification of Piceatannol and Protocatechuic Acid

The NMR and mass spectra of PT are presented in Fig. 4. The negative mode ESI-MS revealed the pseudo molecular ion peak at m/z 243.08 at $[M-H]^-$. The chemical shift (δ), coupling constant (J), and peak signals (brt, brd, Dd, s, and d) of 1H NMR (500 MHz, CD_3OD) are presented. These are δ : 6.15 (1H, brt, J : 2.2 Hz), 6.43 (2H, brd, J : 2.3 Hz), 6.72 (1H, brd, J : 2.5 Hz), 6.74 (1H, s), 6.75 (1H, s), 6.82 (1H, Dd, J : 1.9 Hz), 6.83 (1H, Dd, J : 2.2 Hz), 6.87 (1H, s), 6.90 (1H, s), and 6.97 (2H, d, J : 2.2 Hz). The chemical shift in ^{13}C NMR (CD_3OD) is as follows: δ : 40.3, 49.5, 102.5, 105.7, 113.7, 116.3, 120.1, 126.9, 129.6, 130.9, 141.2, 146.42, and 159.5. The result from this experiment was in accord with the literature data [18] which suggested that the purified compound is a stilbene compound termed piceatannol ($C_{14}H_{12}O_4$).

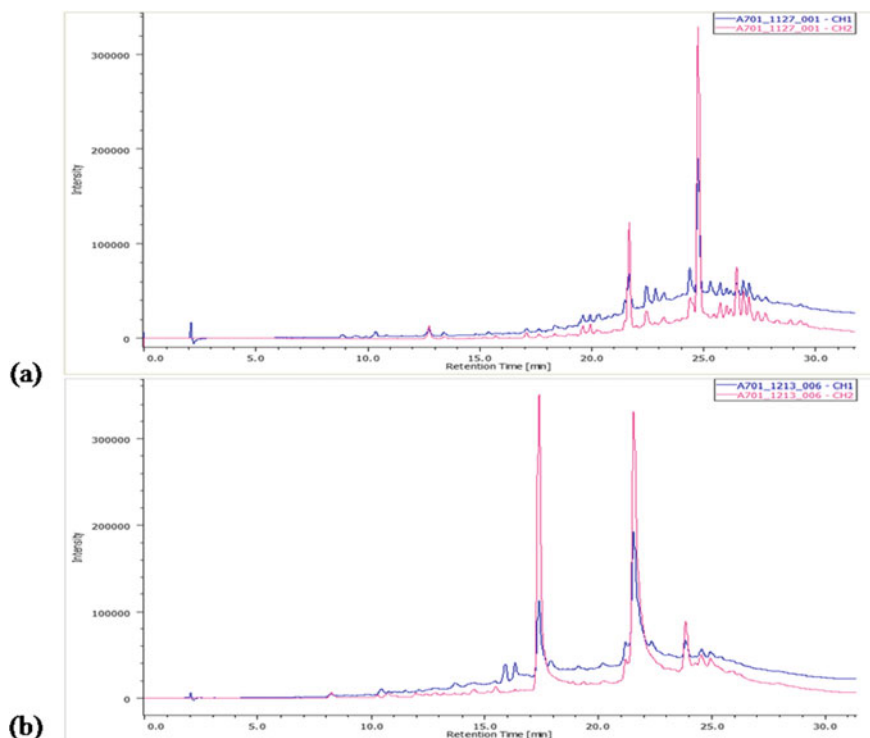


Fig. 2 Gradient HPLC of EtOAc fraction (a) and BuOH fraction (b) of YPFS extract

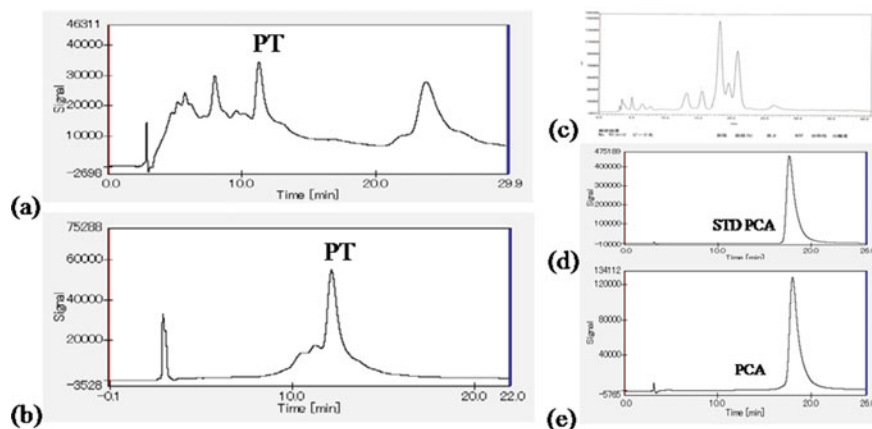


Fig. 3 HPLC chromatograms of isolated PT (a), purified PT (b), isolated PCA (c), commercial PCA (d) and purified PCA (e) from Scirpus B

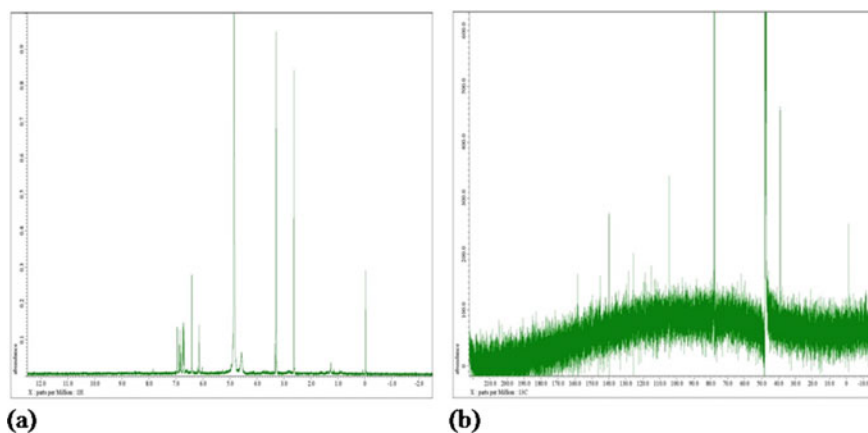


Fig. 4 ^1H NMR (a) and ^{13}C NMR (b) of purified PT

From the ^1H NMR, compound 2 was identified as Protocatechuic acid, as presented in Fig. 5. The negative mode ESI-MS of this compound showed the pseudo ion peak at m/z 153.02 at $[\text{M}-\text{H}]^-$. The chemical shift (δ), coupling constant (J), and peak signals (brt, brd, Dd, s, and d) of ^1H -NMR (600 MHz, CD_3OD) are δ : 6.93 (1H, d, J : 8.0 Hz), 7.39 (1H, dd, J : 8 Hz, J : 1.5 Hz), and 7.42 (1H, d, J : 1.5 Hz). The amount of this compound was significantly less; hence the compound was identified using HPLC analysis by comparing the retention time with the standard compound. From the proton NMR and mass spectroscopy, the compound was assumed to be a dihydroxy benzoic acid and further, it was confirmed as the Protocatechuic acid ($\text{C}_7\text{H}_6\text{O}_4$) from the HPLC analysis. The ^1H NMR data are in agreement with the available data in a relevant prior article [19].

3.3 Biological Activity of Piceatannol and Protocatechuic Acid

The in vitro antioxidant activity of PT and PCA was measured by DPPH and ABTS methods, and their results are presented as IC_{50} value in Table 2. Further, inhibitory activity was compared with various standard compounds. Both PT and PCA exhibited profound antioxidant activity against the tested commercial antioxidant antioxidants. PT showed superior antioxidant activity against ferulic acid (9.80 ± 0.01), caffeic acid (5.77 ± 0.04), ascorbic acid (3.95), and quercetin (7.85 ± 0.01) in the DPPH assay. A similar trend was also noticed in the ABTS assay. Moreover, PCA showed significantly high antioxidant activity compared to all standard antioxidants as well as PT. Both PT and PCA underperformed the in vitro antioxidant activity in comparison to gallic acid (0.89 ± 0.01 , DPPH assay). Both DPPH and ABTS analyses are based on the principle of electron transfer or hydrogen ion transfer [6].

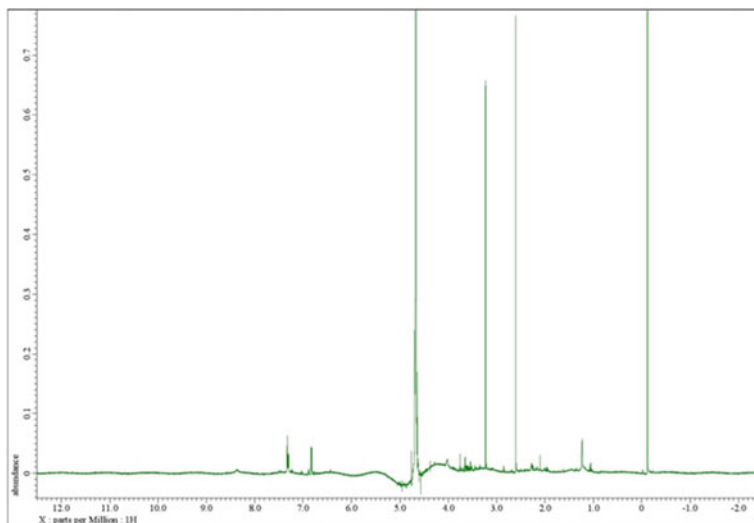


Fig. 5 ^1H NMR of purified PCA

The antioxidant molecules donate electrons or hydrogen ions to the free radicals and thereby quench and reduce them. Various reports suggest hydroxyl and methoxy groups have a major tendency for donating electrons [20], and therefore the profound antioxidant activity of PT [21] and PCA [22] can be argued. The present study indicates that passion fruit-derived PT and PCA are high in antioxidant activity than many commercial antioxidant product systems. Therefore, they can be considered for commercial application. This step could add to the valorization of the waste passion fruit seed.

The parametric data are presented in terms of the IC_{50} value after taking the mean of two individual experiments (in duplicate). All values ($\mu\text{g/ml}$) of antidiabetic and antioxidant experiments are presented as mean \pm SD. NA refers to not applicable in the representation. Similarly, the abbreviations are PT—Piceatannol; PCA—Protocatechuic acid; GA—Gallic acid; FA—Ferulic acid; CA—Caffeic acid; p-CA—p-Coumaric acid; AA—Ascorbic acid; and Q—Quercetin.

The PT and PCA isolated and identified from YPFS were verified for their in-vitro antidiabetic activity and in terms of two different polysaccharide digestive enzymes (α -amylase and α -glucosidase). The results are presented as IC_{50} values in Table 2. The PCA exhibited better antidiabetic activity ($2.12 \pm 0.02 \mu\text{g/ml}$ in α -amylase and 1.89 ± 0.02 in α -glucosidase activity) compared with PT. The antidiabetic activity of PCA in the present study is in accordance with the reported data [23, 24]. PCA is a well-known phenolic acid found in many plant species [23, 24] with profound antioxidant, antidiabetic, and anticancer activities. Moreover, delaying glucose absorption in the body to manage the postprandial glycemic level is one of the approaches being followed for type 2 diabetes [25]. The α -amylase and α -glucosidase digestive enzymes are major digestive enzymes that take part in carbohydrate digestion

Table 2 In vitro antioxidant and antidiabetic activities of Scirpusin B

Activity	PT	PCA	Acarbose	GA	FA	CA	p-CA	AA	Q
<i>Antioxidant</i>									
DPPH	3.16 ± 0.01	1.72 ± 0.03	NA	0.89 ± 0.01	9.80 ± 0.01	5.77 ± 0.04	10.19 ± 0.01	3.95	7.85 ± 0.01
ABTS	3.02 ± 0.01	1.81 ± 0.01	NA	0.79 ± 0.01	6.02 ± 0.01	3.98 ± 0.03	6.36 ± 0.04	6.22 ± 0.04	6.40 ± 0.01
<i>Antidiabetic</i>									
α-amylase	42 ± 0.07	2.12 ± 0.2	0.65 ± 0.01	NA	NA	NA	NA	NA	NA
α-glycosidase	2.17 ± 0.04	1.89 ± 0.02	0.79 ± 0.03	NA	NA	NA	NA	NA	NA

and thereby help in instantly increasing blood glucose levels. Nowadays, natural extract-driven inhibition of these enzymes for delaying carbohydrate digestion is mostly considered as an indirect antidiabetic approach. Stilbenes (resveratrol and its analogues) have been proven to have antidiabetic activity in the literature [26] due to the presence of maximum hydroxyl groups that assist in increasing the bioavailability of the compound [27]. PT is a stilbene molecule majorly found in different *Prunus* species and passion fruit seeds [21]. PT has also proven to be an oxidative stress quencher for many cancer diseases. Such an outcome exhibits more scope for the related evaluation of these compounds in terms of preclinical and clinical studies.

4 Conclusions

In summary, this study reported the isolation of PT and PCA from YPFS by employing various bioorganic chemistry steps, and furthering their antioxidant and antidiabetic activity. Both compounds were found to be effective antioxidant and antidiabetic molecules. PT and PCA were also found to show superior antioxidant activity against different standard polyphenols including caffeic acid, ascorbic, quercetin, etc. This study also reported that PT and PCA inhibited both α -amylase and α -glucosidase digestive enzymes. However, more evidence in terms of preclinical and clinical studies is required. Further, the study also depicted the valorization of waste passion fruit seeds for the isolation of bioactive phytochemicals such as PT and PCA. Such a research directive can open a prospectus for therapeutic product development from waste.

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Conflict of Interest All the authors declare no conflict of interest among each other.

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Indigenous Soybean Cultivars of North-East India: Source of Protein and Product Development for Climate-Smart Foods



Neha Jha , Siddhartha Singha , and Munmi Borah 

Abstract Food and nutritional security is a major challenge that needs to be addressed quickly. Increasing population is impending threat to the adequate supply of nutrition to all. With various steps such as development in agriculture practices introducing higher yielding varieties and integrated farming approaches, some of the needs can be addressed. However additional approaches are required to handle this problem. Soy-based food in particular can prove to be a promising food system in comparison with other crops as it is nutritionally dense with high amount of protein and phytochemicals with medicinal properties. In this article, some of the nutritional properties like total protein content and physical properties, namely bulk density, true density, geometric parameters, sphericity, and external morphology of soybean seed of high yielding soy variety, will be compared with those of local soybean cultivars of North-East India (NEI). Largest sized seeds were of Ukhrul bold (7.88 mm) and smallest size was of Ukhrul small variety (4.96 mm). Sphericity was maximum for Tawang seeds (0.882) and minimum for Ukhrul small from Manipur (0.726). The 100 kernel mass ranged from a maximum of 32.05 g for Kameng seeds to a minimum of 5.78 g for Ukhrul small. Bulk density was highest for Ukhrul small from Manipur followed by Nagaland soybean seeds with a value of 0.651 g/mm³ and 0.641 g/mm³ while lowest was for Ukhrul bold from Manipur, i.e. 0.524 g/mm³. True density of seeds of Ukhrul small was maximum, i.e. 1.292 g/mm³ and minimum for Ukhrul bold from Manipur, i.e. 1.004 g/mm³. Bulk porosity was minimum for Nagaland seeds (0.384) while maximum for Ukhrul medium from Manipur (0.537). Preliminary study on total protein content by the Kjeldahl method shows a wide range of variation for all the cultivars. Protein content of soybean seeds from Nagaland were 45% which is better than that of commercial variety JS 335 (38% protein content). On the other hand, Tawang variety seeds had the lowest protein content of 23%. There lies a huge potential in exploring these local cultivars to know their nutritional

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aspects, digestibility, and processing for traditional usage. These soybean varieties can further be scientifically developed into a wide range of products. This depends on their nutritional and physical properties to meet the ever-increasing demand of food in general and fulfill the protein requirements of the local people. Local cultivars of soybean can be introduced as climate smart foods in several states of NEI as these are suited to weather conditions of this region. This will not only provide nutritional security and fulfill protein needs but also be friendly on an economic basis for the unprivileged sections of the society.

Keywords Soybean · Physical properties · True density · Nutritional · Food security

1 Introduction

Food security, nutritional security, and rapid increase in disease burden are major challenges being witnessed by human civilization. The current population of the world has already exceeded 7.71 billion people, and it is going to touch 9.8 billion by 2050. This population growth has made the supply of adequate food and nutrition to one and all a Herculean task. This is especially more relevant for a country like India. India's population is increasing at a very fast rate and is expected to rise to 1.6 billion by 2030. Malnutrition is a major issue for all countries, and the problem becomes graver for developing and underdeveloped nations. It is becoming difficult to provide adequate nutrition to the ever-increasing population. Along with malnutrition, a surge in non-communicable diseases (NCDs) especially lifestyle diseases like obesity, hormonal imbalances, diabetes mellitus, hypertension, osteoporosis, etc. emerge as a global problem. According to the ICMR India State-Level Disease Burden Study report "India: Health of the Nation's States", there is an increase in the estimated mortality due to Non-Communicable Diseases (NCDs) from 37.09% in 1990 to 61.8% in 2016.

Often, the tri-crisis (food and nutritional security and increment in NCDs) are interlinked. Therefore, tackling these problems needs a variety of steps and not a single measure. Identification and promotion of nutritional foods and nutraceuticals are probably a vital step to increase dietary quality of different target groups as well as prevention of diseases. Soy-based food in particular can prove to be a promising food system in this regard in comparison to other crops. This is due to their nutritionally dense state with high amounts of protein and phytochemicals with medicinal properties (Fig. 1). Soybean can help in combatting not only Protein Energy malnutrition and associated micronutrient deficiency diseases but also hormone-related disorders and other non-communicable diseases. Soybean is unique due to its high-quality protein with minimum saturated fat. Apart from being a legume which is exceptionally a rich source of nutrients, soybean has a number of health-beneficial phytochemicals such as isoflavones, flavonoids, phenolic acids, saponins, sphingolipids, and phytosterols along with soy protein [4, 6]. Soybean also contains heat liable anti-nutritional

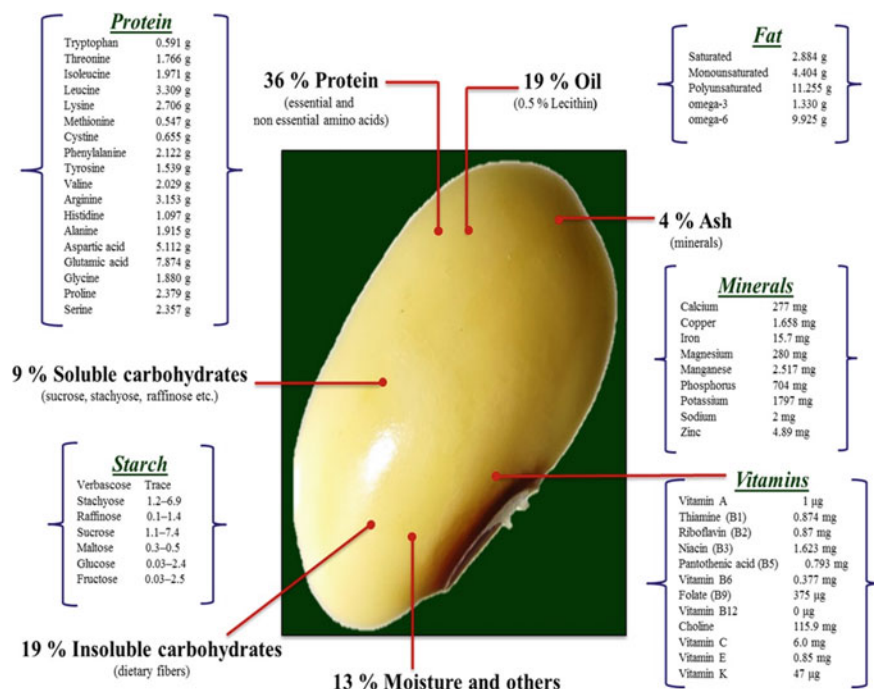


Fig. 1 Soybean composition (Source United Soybean Board, www.nopa.org)

factors. The content of anti-nutritional factors depends upon the cultivar of soybean. These anti-nutrients are reduced by proper processing treatments such as soaking, heat treatment, and fermentation.

Per-capita soy protein consumption is less than 1 g (g) per day in most European and North American countries (data from the UN Food and Agriculture Organization, 2003). The Japanese, on the other hand, consume an average 8.7 g of soy protein per day; Koreans, 6.2–9.6 g; Indonesians, 7.4 g; and the Chinese, 3.4 g. India is in fifth position in producing soybean [1], but its consumption among native people is very low in our country. As per the recommended daily consumption of 50 g of lentils and 25 g of soy foods per day, Indians consume just half the amount of lentils and very low portions of soy foods (NSSO). Through the appropriate usage of soybean processing technology, many developed soy products can find emphasized recognition as a nutrient/protein-dense plant food in India. Soy isoflavones may reduce the risk of osteoporosis as they have weak estrogenic effects and their similarity with ipriflavone; a synthetic isoflavone helps to increase bone mineral density in women post menopause. Soy isoflavones also decrease the risk of diseases like osteoporosis, breast cancer, heart attack, and kidney stone formation.

Soybean is mainly utilized for its protein and oils for human consumption, as animal feed, and also for various industrial applications (Fig. 2). A large proportion of soybean is used to produce soymeal for animal feed after the extraction of soybean

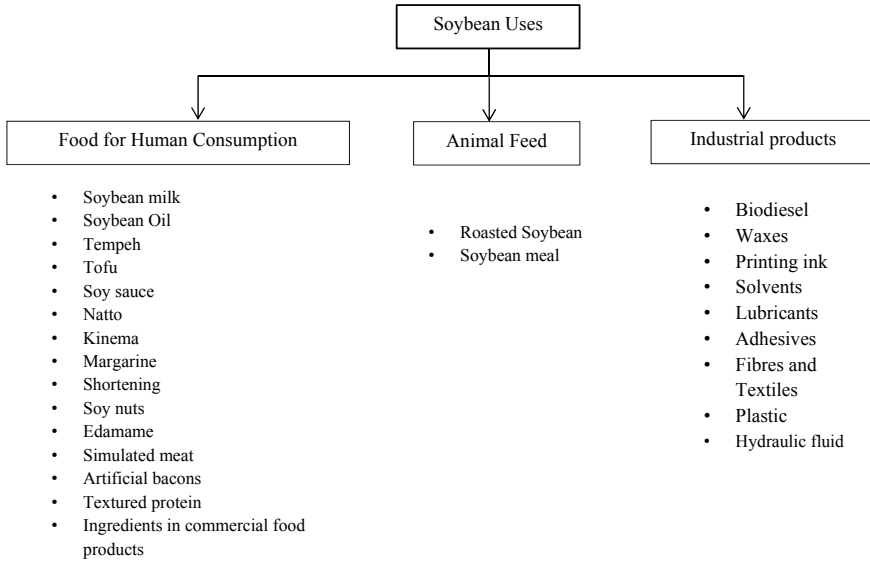


Fig. 2 Soybean uses (Source Statistics Canada, Census of Agriculture, 2007)

oil. The process for the production of soymeal, isolates, and concentrates is given in Fig. 3.

Apart from basic nutrition, soybean offers health benefits in the daily diet and helps in the management of disorders like diabetes mellitus, obesity, hyperlipidemia, etc. Soybean offers unique advantages for health as it is rich in micronutrients, particularly minerals and vitamins as well as nutraceuticals.

Uses of soybean are confined to the production of traditional fermented foods, soymilk a constituent in production of weaning foods, geriatric foods and snack foods, in composite flours, textured proteins, and soymeal for animal feed. Milling, popping, flaking, extrusion cooking, fermentation, etc. are various investigated processing technologies for the successful outcome of soybean and soybean-based foods including ready-to-eat or ready-to-use products. Thereby, these products ensure their utilization as a major food. The extrusion cooking process is a high-temperature and short residence time process. In this process, the moist and soft food material is fed to the extruder to achieve processed products through combinations of temperature, pressure, and residence time. Processing of soybean into various ready-to-eat or ready-to-use products is hindered by various reasons such as non-availability of suitable equipment needed for soybean processing and hence high cost of imported equipment, large variations among various soybean cultivars, high range of structural variations, and non-uniformity within various soybean cultivars. Many of these pose technical problems in the development of suitable milling equipment. Also, the production of soybean is highly fragmented.

Small seeded flat varieties with high protein content and larger surface area are opted for natto, kinema, hawaijjar, axone, tungrymbai, perayaan, be kang, and

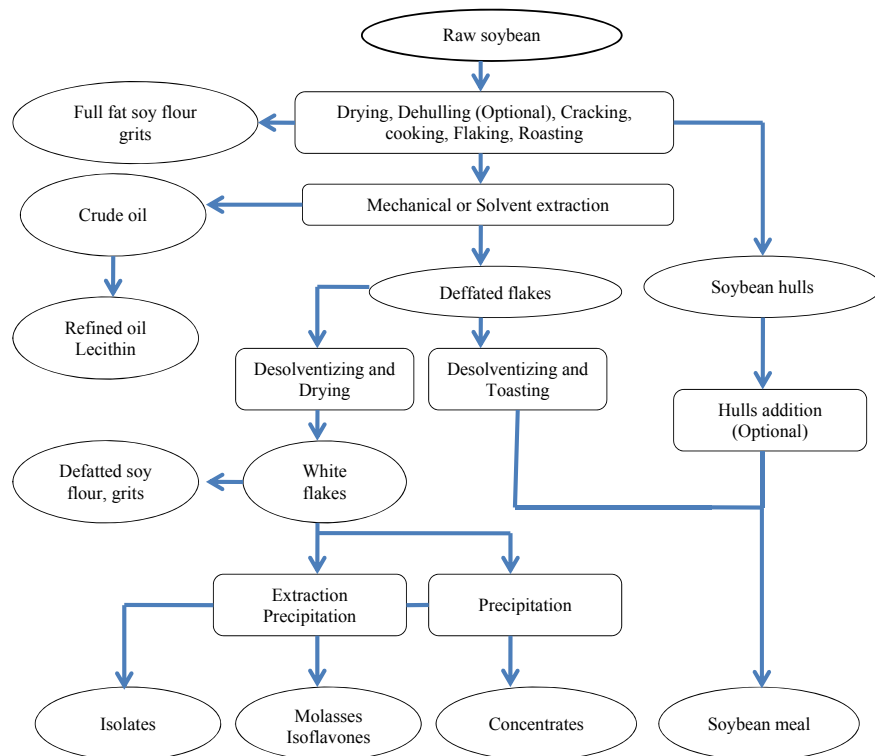


Fig. 3 Process for soybean meal production (Source www.feedipedia.org)

soybean sprouts. However large soybean seeds are preferred for miso preparation. Defatted soy meal is used to prepare soy protein isolate that serves as a primary ingredient in nutritional supplements, meat systems, weaning formulas, soups, etc.

Soy oil is extracted from varieties possessing higher fat content. Soybean varieties with higher fat content are susceptible to oxidative damage. Hence, their storage and processing should be done to minimize oxidative damage. Soybean seeds with soft seed coats are more susceptible to infestation. Hence, there should be arrangements to avoid post-harvest infestation. Some studies reveal the effect of environmental conditions on proximate content and lipoxigenases [3, 12, 15]. However, not many investigations addressed the effect of the physical characteristics of soybean seeds on the proximate and processing parameters. Physical parameters affect the processing properties as different varieties may be suitable for different products. Thus, the equipment design and processing operations need to be customized as per the physical parameters of the soya seed systems.

Also, soybean contains a large proportion of lipids which cause oxidative rancidity. This as an effect reduces the shelf life and reflects poor storage quality and eventually enhances nutritional loss after processing. Further, hesitation in accepting soybean-based products as a staple food in large classes of society. At times, the taste could

be the reason to cause reluctance in soybean consumption. These are a few big challenges in the advancement of soybean utilization. All these reasons render the highly nutritional soybean crop to remain unexplored. For the emergence of new horizons in the development of soybean and soybean-based products, it is necessary to develop the processing techniques from the trivial to the modern one. To make high-quality soybean products available and affordable, it is necessary to do the optimization and standardization of various processing steps and processing conditions. Further, research is needed for the development of high-yield and high protein-containing soybean varieties. Agricultural policies need to be amended for a reduction in the final product costs. This can be addressed through extensive soybean production and a reduction in the cost of equipment and processing.

Soybean is a good substitute for the fulfillment of protein and other nutritional requirements. Especially local soybean cultivars of North-East India can prove to be the best source to meet the daily recommended allowance for protein for the indigenous people and for rural poor families. As the local varieties are studied, they require low agricultural input, and need no chemical fertilizers, pesticides, and manures. This is due to them being rain-fed and to the local climatic conditions. On the other hand, the high-yielding commercial varieties need adequate irrigation and a high amount of chemical fertilizers and pesticides. Also, for such cases, other logistic operational costs are also involved in transportation, storage, preservation, retail and distribution, and wastage. The terrain of North-East India (NEI) is hilly and uneven. Hence, the cost of transportation increases tremendously for both raw materials and finished products. Also, the cost of transporting pesticides and fertilizers from different parts of the country to various regions of North-East India, etc. is high and the increased price has to be paid by the consumers during their purchase. In NEI, the land belongs to the community. Hence, the cultivation of local varieties can be done easily. This will make the physical accessibility of soybean easy as from availability perspective it will be grown within the reach of community in their farms and accessible from economic perspective as well. Local cultivars of soybean can be introduced as climate-smart foods in several states of NEI as these are acclimatized to the weather conditions of the region. This will not only provide nutritional security and fulfill protein needs but also serve as an economy friendly option for the unprivileged sections of the society. These local cultivars of soybean in North-East India can further be explored to visualize their potential as a raw material for the development of food products with better protein content. Proper scientific interventions to understand the physical properties and nutritional parameters for the development of suitable products from various soybean cultivars can also help in generating employment and awareness among the local people. Better utilization of local cultivars of soybean can be achieved through good know-how of their physical properties. This will also assist in designing equipment and suitable processing techniques for various cultivars.

In this article, we will compare a few nutritional properties such as total protein content, and physical properties, namely bulk density, true density, geometric parameters, sphericity, and soybean seed external morphology of high-yielding soy variety with the local soybean cultivars of North-East India (NEI). Six local, unexplored soy



Fig. 4 Soybean varieties from North-East India; 1—JS 335 soybean, 2—Kameng soybean, 3—Ukhrul bold soybean, 4—Ukhrul medium soybean, 5—Ukhrul small soybean, 6—Nagaland soybean, and 7—Tawang soybean

cultivars (2–7) grown in NEI and one commercial variety, i.e. JS 335(1) (Assam Agriculture University), were considered for the investigation (Fig. 4). Protein content, physical parameters, external seed morphology, and product suitability of these local varieties were investigated.

2 Methodology

The present study on “indigenous soybean cultivars of North-East India: source of protein and product development for climate-smart foods” was undertaken at the Indian Institute of Technology, Guwahati, Assam. The six local varieties and one commercial variety of soybean seed samples were procured from Assam Agricultural University and Krishi Vigyan Kendra Jorhat, Assam. These grains contained 11% moisture. Random samples of 1000 g were taken for further testing procedures. Manual sorting was done to remove faulty and infested seeds. Thereby, sound and healthy seeds were selected. All samples were stored at 4 °C in a refrigerator till further analysis. The physical properties were evaluated as per Mohsenin [9] and Indian Standard Institution [2], Methods of Analysis for food grains [2].

2.1 Physical Properties

Seed size: The size of seeds was defined in terms of geometric mean diameter (GMD). It is the cube root of the product of three principal axes, i.e. length, width, and thickness of the seed. These three axes were measured with the help of a micrometer screw gauge with a least count of 0.005 mm. The mean value of hundred observations of healthy and sound seeds was conducted for randomly selected samples to evaluate the seed size using the expression:

$$\text{Size}(De) = (abc)^{1/3} \quad (1)$$

where De is geometric mean diameter, and

a, b, and c are length of seed, width of seed, and thickness of seed.

Seed volume: Seed volume of soybean seeds was determined by displacement method using toluene. Soybean seed sample weighing 5 g was immersed in 20 mL of toluene in a 50 mL graduated measuring cylinder and in triplicates. The volume displaced by the seeds was observed. The true volume of seeds was divided by the number of seeds to find the volume of one seed.

Surface area: The surface area was determined using the expression:

$$As = 3.14(De)^2, \quad (2)$$

where De = seed geometric diameter (mm) and

As is surface area in mm².

Surface-to-volume ratio: The surface-to-volume ratio was determined using the expression:

$$\text{Surface to Volume ratio (RSV)} = \frac{\text{Surface area (As)}}{\text{Seed volume (V)}}, \quad (3)$$

where

RSV Surface to volume ratio;

As Surface area (mm²);

V Seed volume (mm³).

Sphericity: The Sphericity of soybean seeds was determined with the expression:

$$\text{Sphericity} = \frac{\text{Geometric mean diameter}}{\text{Major diameter}}$$

$$\phi = \frac{(abc)^{1/3}}{a}, \quad (4)$$

where a, b, and c are length, width, and thickness of the soybean seed.

Hundred Kernel Mass: The method given in IS: 4333 (Part IV) [2] was used to estimate hundred kernel mass. One hundred randomly selected sound and healthy seeds of each variety of soybeans at a moisture level of 11% were collected and weighed with an electronic weighing balance (Metler Toledo balance) having a least count of 0.001 g.

Bulk density: The bulk density of seeds was determined as a ratio of their mass to bulk volume. For determining the bulk density of soybean seeds, a standard graduated measuring cylinder of 500 cc was filled up with grain and the contents were weighed. Thereafter, the mass was expressed as an average. The expression for bulk density is

$$\rho_b = \frac{m}{v_b}, \quad (5)$$

where

m mass of seeds, kg;
 v_b bulk volume of seeds, mm^3 ;
 ρ_b bulk density, kg/m^3 .

True density: True density refers to the ratio of mass to the true volume of the sample. True density was measured with the toluene displacement method. In a graduated measuring cylinder (accuracy of 0.1 mL), 20 ml of toluene was added. Soybean seed weighing 5 g was submerged in 20 mL of toluene. True volume of the sample was determined by measuring the volume rise in the toluene volume after adding the sample. This was evaluated using the expression:

$$\rho_t = \frac{m}{v_t}, \quad (6)$$

where

m mass of seeds, kg;
 v_t true volume of seeds, mm^3 ;
 ρ_t true density, kg/m^3 .

Bulk porosity: Bulk porosity is defined as the percentage of void volume for given moisture content for a sample. It is determined as the ratio of the difference in the true density and bulk density to the true density of the test sample. Bulk porosity is expressed in percentage and with the following equation:

$$\varepsilon = \frac{\rho_t - \rho_b}{\rho_t} \times 100, \quad (7)$$

where

ε porosity percent;
 ρ_t true density, kg/m^3 ;
 ρ_b bulk density, kg/m^3 .

2.2 *Moisture Content*

5.0 g of well-mixed sample was taken and put in a pre-weighed aluminum dish. It was dried at 105 ± 1 °C for 5 h to constant weight [10].

2.3 *External Seed Morphology*

Seeds were analyzed with a stereo microscope (magnification of $100 \times$) and in terms of the visualized differences in the external morphology of various soybean cultivars and their infestations.

2.4 *Total Protein Content*

Total protein content is based on the amount of total nitrogen in sample [10]. It is determined using the expression:

Total nitrogen (%) = (volume in ml of standard acid \times N of standard acid) – (volume in ml of standard Sodium hydroxide \times N of standard sodium hydroxide) \times 1.4007/weight of sample (grams).

Protein (%) = Total Nitrogen (%) \times 6.25 (For soybean, correction factor = 5.71).

3 Results and Discussion

Prior to the experiments, all test samples were taken from the refrigerator at 4 °C to equilibrate them with the room temperature for 12 h. Thereafter, they were used to determine the physical properties.

3.1 *Principal Dimensions, Seed Size, and Seed Volume*

The results obtained for seed volume, seed size, and principal dimensions for seven cultivars of soybean of NEI are presented in Table 1. Soybean seeds of Kameng possessed the highest volume of 0.322 mm^3 . On the contrary, the seeds of Ukhrl small from Manipur had the lowest volume of 0.065 mm^3 . The largest seeds were the Ukhrl bold variety from Manipur (7.88 mm) followed by the Kameng variety (7.385 mm). The smallest was the Ukhrl small variety from Manipur (4.96 mm).

Table 1 Principal dimension, seed size, and seed volume of soy cultivars of North-East India

Soybean cultivars	Length, a (mm)	Width, b (mm)	Thickness, c (mm)	Seed size (abc) ^{1/3} (mm)	Seed volume (mm ³)
JS 335	6.96 ± 0.484	5.84 ± 0.366	4.98 ± 0.429	5.937 ± 0.058	0.142 ± 0.008
Kameng	8.37 ± 0.978	6.92 ± 0.959	5.58 ± 0.865	7.385 ± 0.456	0.322 ± 0.006
Ukhrul bold	11.2 ± 0.906	7.74 ± 0.640	5.65 ± 0.813	7.880 ± 0.066	0.186 ± 0.002
Ukhrul medium	7.26 ± 0.412	5.94 ± 0.317	4.97 ± 0.485	5.894 ± 0.182	0.128 ± 0.002
Ukhrul small	6.49 ± 0.726	5.03 ± 0.723	3.27 ± 0.579	4.962 ± 0.146	0.065 ± 0.001
Nagaland	6.55 ± 0.784	5.21 ± 0.517	4.4 ± 0.440	5.258 ± 0.069	0.146 ± 0.005
Tawang	7.41 ± 0.601	6.62 ± 0.442	5.77 ± 0.435	6.646 ± 0.084	0.281 ± 0.009

Data is given in Average ± Standard deviation

3.2 Bulk Density, True Density, Bulk Porosity, and Hundred Kernel Mass

The evaluated bulk density, true density, bulk porosity, and hundred kernel mass for seven varieties of soybean from NEI are given in Table 2. The bulk density was highest for Ukhrul small variety from Manipur (0.651 g/mm³) followed by Nagaland variety soybean seeds (0.641 g/mm³). The lowest was for the Ukhrul bold variety from Manipur (0.524 g/mm³). True density of Ukhrul small variety was maximum (1.292 g/mm³). It was minimum for Ukhrul bold variety from Manipur (1.004 g/mm³). Bulk porosity was minimum for Nagaland variety seeds (0.384). On the contrary, it was maximum for Ukhrul medium variety from Manipur (0.537). 100 kernel mass ranged from 32.05 g for Kameng seeds followed by 21.32 g for Ukhrul bold from Manipur. For the case, the lowest value of 5.78 g was for the Ukhrul small variety from Manipur. Incidentally, the hundred kernel mass of commercial variety JS 335 was 13.15 g.

3.3 Surface Area, Surface-To-Volume Ratio, and Sphericity

The data obtained for surface area, surface-to-volume ratio, and sphericity for seven varieties of soybean from NEI is given in Table 3. The surface area was maximum for Ukhrul bold variety (24.743 mm²) and was minimum for Ukhrul Small variety (15.582 mm²). The surface-to-volume ratio was highest for Ukhrul small (245.008 mm⁻¹) and was lowest for Tawang variety seeds (73.452 mm⁻¹). The sphericity altered from 0.742 for the Ukhrul bold variety from Manipur to 0.857 for the JS 335 commercial variety. The sphericity was maximum for Tawang variety seeds (0.882) and was minimum for Ukhrul small variety from Manipur (0.726).

Table 2 Bulk density, true density, and hundred Kernel mass of soy cultivars of North-East India

Soybean cultivars	Bulk density (g/mm ³)	True density (g/mm ³)	Bulk porosity %	100 Kernel mass (g)
JS 335	0.577 ± 0.020	1.056 ± 0.058	0.452 ± 0.045	13.15 ± 0.066
Kameng	0.542 ± 0.039	1.060 ± 0.007	0.489 ± 0.039	32.05 ± 0.771
Ukhrul bold	0.524 ± 0.032	1.004 ± 0.006	0.478 ± 0.030	21.32 ± 0.092
Ukhrul medium	0.571 ± 0.019	1.234 ± 0.033	0.537 ± 0.006	14.44 ± 0.205
Ukhrul small	0.651 ± 0.008	1.292 ± 0.027	0.496 ± 0.007	5.78 ± 0.078
Nagaland	0.641 ± 0.006	1.041 ± 0.023	0.384 ± 0.015	10.24 ± 0.063
Tawang	0.568 ± 0.008	1.051 ± 0.031	0.459 ± 0.017	17.35 ± 0.101

Data is given in Average ± Standard deviation

Table 3 Surface area, surface-to-volume ratio, and sphericity of soy cultivars of North-East India

Sample	Surface area (mm ²)	Surface-to-volume ratio (mm ⁻¹)	Sphericity (abc) ^{1/3} /a
JS335	18.644 ± 0.183	127.267 ± 0.450	0.857 ± 0.017
Kameng	23.187 ± 1.431	73.845 ± 0.676	0.805 ± 0.023
Ukhrul bold	24.743 ± 0.206	132.731 ± 0.828	0.742 ± 0.035
Ukhrul medium	18.508 ± 0.571	145.197 ± 2.305	0.829 ± 0.030
Ukhrul small	15.582 ± 0.457	245.008 ± 1.897	0.726 ± 0.014
Nagaland	16.509 ± 0.215	110.551 ± 0.120	0.824 ± 0.012
Tawang	20.868 ± 0.264	73.452 ± 0.243	0.882 ± 0.006

Data is given in Average ± Standard deviation

3.4 External Seed Morphology

Soybean seeds were examined under a stereo microscope (100 X) to visualize the differences in external morphology and seed coat and infestation rates. The microscopic photos are presented in Figs. 5 and 6. In due course of the storage of the seeds for 30 days at ambient conditions, it was observed that the larger sized seed varieties (Kameng, Ukhrul bold, and Tawang) were more prone to infestation (shown in Fig. 5) in comparison to smaller seed varieties (Ukhrul small) and medium-sized seed varieties (Ukhrul medium, Nagaland, and JS 335). This could be due to the higher content of protein and fat. This could be due to the soft seed coat that damages them easily through mechanical effects and thereby facilitates an easy attack with the insects and pests due to the pertinent surface damage. Soybean seeds of Ukhrul medium and Ukhrul bold cultivars were dark colored with purple patches. This may suggest that they may have a higher polyphenol content. On the other hand, the JS 335 and Kameng variety seeds were uniform and smooth.

Soybean seeds of Ukhrul medium and Ukhrul bold cultivars were dark colored with purple patches. This suggests that they may have higher polyphenol content.

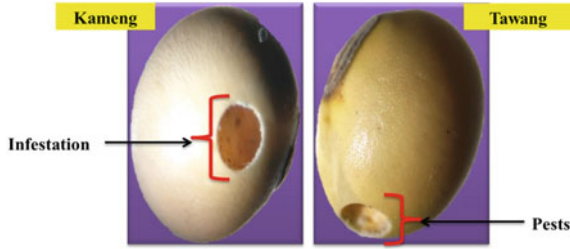


Fig. 5 Infestation of soybean seeds of Kameng and Tawang varieties

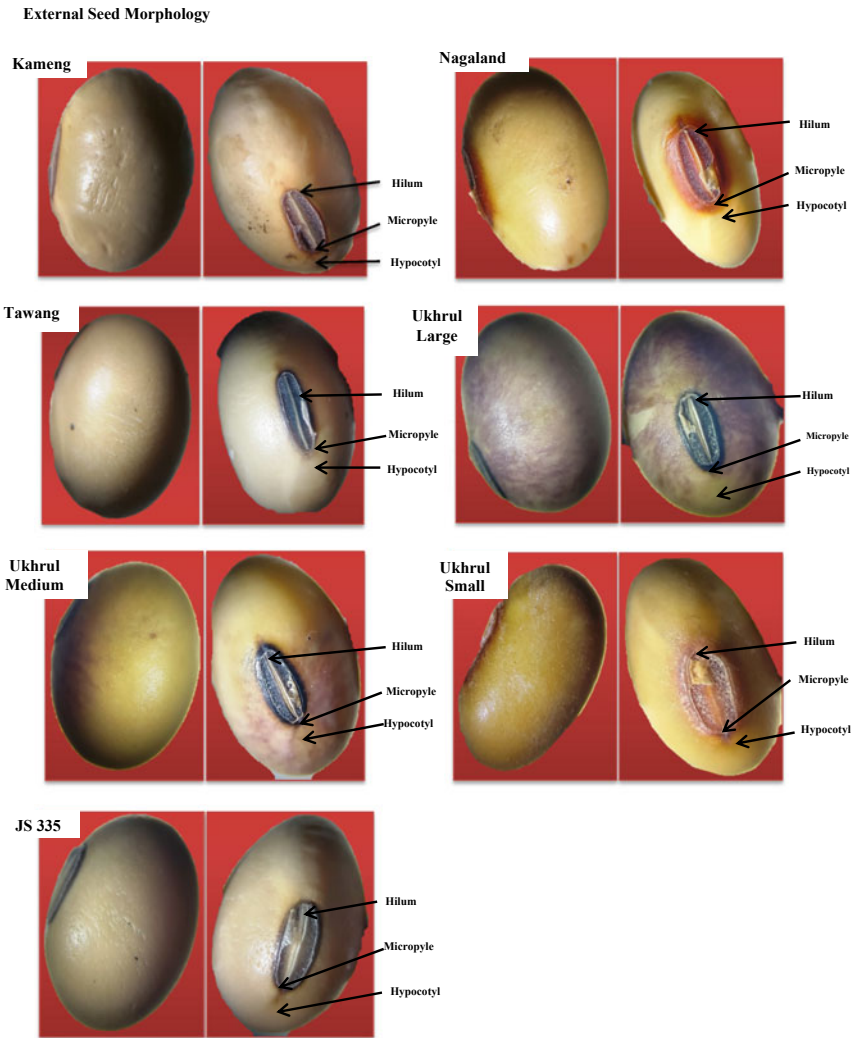


Fig. 6 Mature soybean seeds of various cultivars of NorthEast India

However, seeds of JS 335 and Kameng varieties were uniform and smooth and light colored. Seeds of Ukhru small were rough, brown colored, small, and flattened in shape while that of the Nagaland variety were bright colored and have a smooth surface. The seeds of Tawang variety were light colored and had a smooth surface. Tawang seeds were comparatively bigger than the medium-sized varieties, namely JS 335, Nagaland, and Ukhru medium soybean seeds. The external morphology of various cultivars of mature soybean seeds has been depicted in Fig. 6.

3.5 Total Protein Content

The Recommended Daily Allowance (RDA) of protein is described in terms of a kilogram of body weight. On average, 0.8 g of protein is to be taken per kg weight of a person. For example, for an adult weighing 50 kg, 40 g of protein per day is required.

Table 4 shows the protein content per 100 g of common protein-rich foods and their respective cost in India. From the table, it can be analyzed that the cheapest source of protein is eggs followed by soybean. Thus, soybean is the cheapest source of protein for vegetarians. Soybean has good biological value among the vegan protein sources. Soybean contains 35–45% crude protein. Soy protein has been concluded to be the bean with the highest quality protein (FDA, WHO guidelines) among those protein sources classified for protein quality in children and adults. The score of soy protein isolate is 1. It indicates that the quality of soy protein is almost equal to that of protein in meat and milk. Hence, soybean is used to make soy protein isolates and concentrates.

The Kjeldahl method-based total protein content did confirm that a wide variation exists in the parameter for the chosen cultivars of soybean seeds. The total protein content of seven soybean cultivars of NEI is given in Fig. 7. Protein content of soybean cultivar from Nagaland was 44.9%. This is even higher than that of

Table 4 Protein content, cost, and biological value (BV) of alternate protein sources

Food	Protein/100 g	Cost/Kg (India)	Cost of protein/40 g	BV
Chicken	32.8 g	Rs 220/-	Rs 268.29/-	80
Cheese	26 g	Rs 370/-	Rs 569.245/-	84
Fish (all types)	19.2 g	Rs 250/-	Rs 520/-	76.0
Eggs	13 g (2 eggs)	Rs 80–100/dozen	Rs 45/-	93.7
Yogurt	4.1 g	Rs 120/-	Rs 1170.73/-	68
Milk	3.3 g	Rs 60/-	Rs 727.27/-	90
Soybean	12.5 g	Rs 80/-	Rs 256/-	72.8
Lentils	9.1 g	Rs 160/-	Rs 703.29/-	–
Beans	6.6 g	Rs 100/-	Rs 606.06/-	58

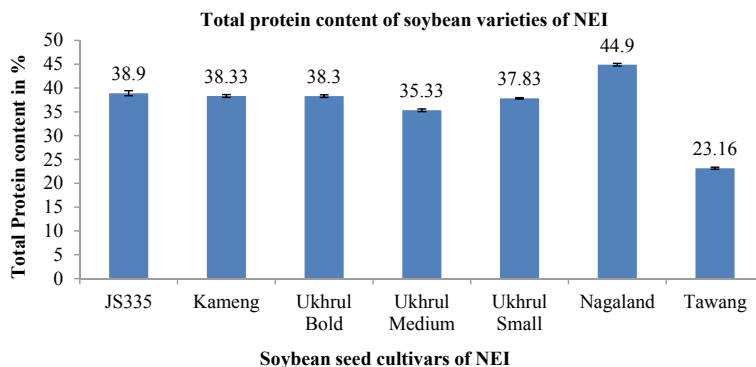


Fig. 7 Total protein content of local soybean seed varieties of NEI

commercial variety, namely JS 335 (38.9% protein content). Tawang variety seeds had the lowest protein content of 23.16%. On the other hand, seed varieties such as Kameng, Ukhrul bold, and Ukhrul small had a protein content of 38.3%, 38.3%, and 37.83%, respectively. Protein content of Ukhrul medium was 35.33%. JS 335 is the deployed commercial variety for cultivation in various regions of India. However, the protein content of the local cultivar of Nagaland has higher protein content in comparison to the mentioned commercial variety.

4 Conclusions

A wider variation has been apparent for various cultivars of soybean seeds in terms of their physical properties, external seed morphology, and total protein content. These wider variations can be further explored for their suitability in developing a number of products. Due to variations in geometric parameters, their packing volume, storage characteristics, milling, and extraction properties will also differ considerably. This provides us a plethora of opportunities for exploring these seeds as various products to fulfill the nutritional and protein requirement of local people at lower prices. The potential of local varieties remains unexplored for food and nutritional security. There lies a huge potential in exploring these local cultivars to know their nutritional aspects, digestibility, and processing for traditional usage as they may be better suited for boiling, germination, fermentation, etc. in comparison to the commercial varieties. Local cultivars of soybean can be introduced as climate-smart foods in several states of NEI as these are acclimatized to the weather conditions of the region. This will simultaneously provide nutritional security, fulfill protein needs, and can also prove to be economic for the unprivileged sections of society. A scientifically developed process technology of processed soy products with indigenous cultivars will not only meet the daily protein requirement of indigenous people but also expand its consumer

base in India as well as create micro industries and associated entrepreneurship opportunities.

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Agro Process Engineering

A Review of Bioactive Compounds and Maillard Reaction-Based Products Generated During the Thermal Treatment of Garlic



Ajay Kumar Shakya  and Chandan Das 

Abstract The biological properties (antioxidant and antimicrobial activity) of garlic during the transformation of raw garlic to black garlic using thermal treatment are discussed in this article, along with current research on bioactive compounds (organosulfur compounds, total phenolics, total flavonoids, etc.) and Maillard reaction-based products of garlic. Present-day treatments for garlic use a variety of technologies. These technologies employ various guiding principles to treat garlic and turn it into black garlic. High temperatures are used in thermal treatment technologies to treat garlic. The purpose of this review is to provide details on the effects of thermal processing on Maillard reaction-based products, such as hydroxymethyl furfural, Melanoidin, and polyphenols, and their capacity to preserve food. Black garlic is produced using various technologies, but thermal treatments are becoming more and more common because they are less expensive than other technologies. The study of the effects of thermal treatment techniques on Maillard reaction-based products like hydroxymethyl furfural, Melanoidin, sugars, polyphenols, and so forth, as well as interactions between garlic components and between nutrients and microbes, requires deeper investigations to fill knowledge gaps. The potential of thermal technologies for enhancing the bioactive compounds and Maillard reaction-based garlic products has been highlighted in this review. These compounds have the ability to inactivate spores and enliven microbe cells in garlic.

Keywords Raw garlic · Black garlic · Bioactive compounds · Maillard reaction · Thermal treatment · HMF · Melanoidin

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Nomenclature

FG	Fresh garlic
MRPs	Maillard reaction products
BG	Black garlic
GSAC	γ -Glutamyl-S-allyl-cysteine
SAC	S-allyl-cysteine
5-HMF	5-Hydroxymethyl furfural
TPC	Total phenolic content
DPPH	2,2-Diphenyl-1-picrylhydrazyl
OSC	Organosulfur compounds
FOS	Fructo-oligosaccharide
ABG	Aged black garlic
FRG	Fresh raw garlic
GC-O-MS	Gas chromatography olfactometry mass spectrometry
LCMS	Liquid chromatography mass spectroscopy
AGE	Aged garlic extract
NMR	Nuclear magnetic resonance
TFC	Total flavonoid content
ABTS	2,2'-Azino-bis (3-ethylbenzothiazoline-6-sulfonic acid)
SPC	S-(propenyl)-L-cysteine
RH	Relative humidity

1 Introduction

Black garlic (BG) results from the long-term aging of fresh garlic (FG) at a high temperature and humidity. Without using any additional chemicals, fresh garlic is thermally processed for one to three months at 60 to 80 °C under controlled humidity to produce black garlic. Under these circumstances, several chemical processes, including Maillard and enzymatic reactions, transform garlic flavor and change its color from white to black [60]. The treatment darkens garlic cloves, imparts a sweet taste, and converts clove texture from chewy to jelly-like [90]. A treated form of garlic called “black garlic” which exhibits more powerful antioxidant properties than fresh garlic due to its increased polyphenol and flavonoid content has become a research subject [33]. The chemical constitution of FG alters significantly with its variety [19], growing location [46], harvesting condition [53, 78], and plant growth [23, 26]. A broad category of characteristic components has been mentioned in terms of lipids [82, 91], carbohydrates [15], proteins [16], organic sulfur compounds [59], polyphenols [12], 5-hydroxymethyl furfural [43], garlic entophytes [72], melanoidins [28], etc. MRPs are a complex group of substances, generated during the non-enzymatic Maillard reaction initiated when reducing sugars and when amino acids or proteins are combined. Additionally, there have been reports that the flavors in food, known

as volatile MRPs, have antioxidant properties [66]. The three primary stages of the MR are the early, intermediate, and final. Each stage is influenced by various variables, including the type and concentration of the reactants, time, temperature, water activity, and pH [38]. Aroma compounds, intermediates that absorb ultraviolet light, and high-molecular-weight melanoidins are just a few Maillard reaction products (MRPs) produced by the Maillard reaction [39]. When an amino group and a reducing sugar carbonyl react together, melanoidins are formed. Melanoidins impart a distinct flavor and for this reason, they are well-liked by consumers. However, they also give food a distinctive brown appearance. There are several foods that have been thermally treated, such as black garlic, bread, pastries, and coffee. All these contain melanoidins [68, 94]. Further, the 6-carbon molecule 5-hydroxymethyl furfural (5-HMF) has aldehydes and alcohol functional groups on its furan ring. It can be produced at high temperatures as an intermediate during the Maillard reaction or by caramelizing sugars [37, 54]. This review provides studies on the thermal treatment impact on the bioactive compounds, Maillard reaction products, and antioxidant activity of black garlic. Additionally, we suggest potential future uses of thermally treated garlic as a functional food product.

2 Factors Influencing Garlic Quality

Several factors influence the quality of garlic in a critical way. Research efforts carried out to date delineate these multiple factors on the quality of garlic. Figure 1 shows the factors that influence the quality of garlic. A few of these are discussed in the subsequent sub-sections.

Technology-based treatments are crucial to the quality preservation of garlic. These technologies work on various principles and treat garlic in various ways. Among various treatment technologies, a few are widely used. These are depicted in Fig. 2.

2.1 *Influence of the Garlic Plant Growth*

A comprehensive study of garlic shows that bioactive compounds have been influenced mainly by various variables. Garlic plants are crucial for several bioactive compounds. Several works have confirmed variations in bioactive compounds during plant growth. Studies reveal that the metabolite concentration in the garlic plant tissues alters and changes in different growth stages. The concentration of metabolites varies significantly between different tissues of the garlic plant. The young garlic plant is a portion of excellent nutrient-dense food. Liu et al. (2020) studied changes in garlic metabolites collected every week, i.e., from April 18 to May 30 (a total of 7 weeks) in 2019. They had concluded that when the leaves started to fade away, major nutrients began to transfer from other tissues to the cloves.

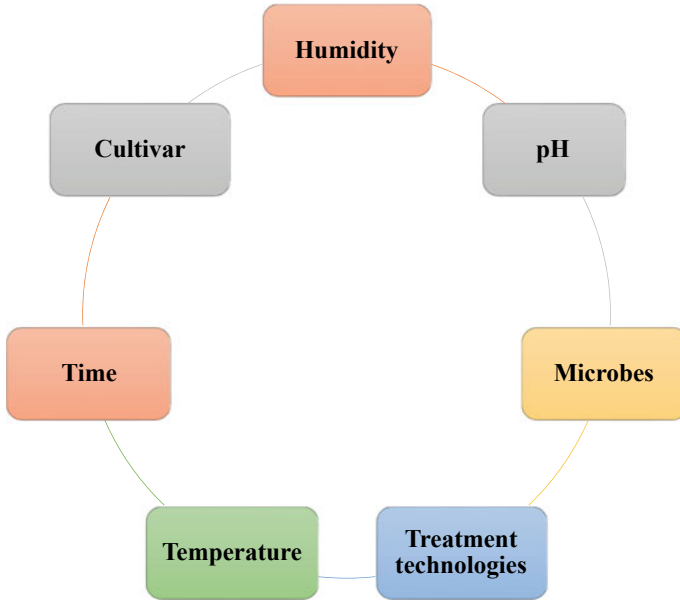
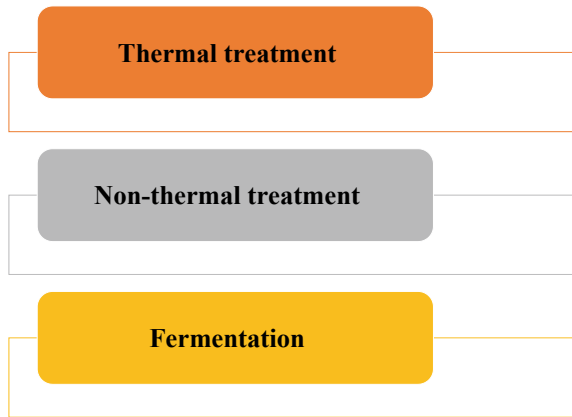


Fig. 1 Variables influencing quality of black garlic

Fig. 2 Widely used garlic treatment technologies



Delaying in harvest may be beneficial to enhance the nutritional content of garlic bulbs. The metabolomics data showed that the quantities of metabolites in five tissues varied considerably throughout different development stages. Nucleotides and alkaloids enhanced due to aging for about two weeks but not the clove. Organosulfur compounds, amino acids, and dipeptides all have mostly increased in the clove [47]. Garlic quality and nutritional content vary significantly and depend upon the time of harvest. The orthogonal partial least squares model might be employed to evaluate the superiority of the garlic bulb and validate the optimal reaping period throughout

its growth. During the development of the garlic bulb, 91 metabolites were identified [48].

While growing garlic bulbs, metabolomics research revealed various quality alterations in garlic bulbs. The presence of amino acids and organosulfur content has been found to have an active metabolism. The majority of metabolites in the “ γ -glutamyl” category were nitrogen and sulfur compounds. These enhanced considerably throughout the development of garlic. S-(propenyl)-L-cysteine (SPC), diallyl disulfide, ajoene, allicin, GSPC, SAC, and GSAC were detected among the 50 compounds researched. Chang et al. [13] discovered many metabolites in glycerophospholipid metabolism. Once the garlic got aged, the constitution of these metabolites grew substantially. The shikimate route is one of the secondary metabolic pathways in plants and microorganisms. It is a remarkable seven-step transformation for the generation of three primary aromatic amino acids, namely phenylalanine, tyrosine, and tryptophan [13].

2.2 Influence of the Location

The studies show that garlic compounds do undergo a transformation with different environmental conditions. Thus, location also has an important role. The garlic from other places has different compounds in it. Thereby, such studies indicate that compounds present in the garlic do alter with the location. The phenolic content of garlic skin samples has been substantially more significant than garlic cloves. It was also higher in Australian-produced varieties than in commercially cultivated garlic from other countries. The organosulfur compounds were more effective in cloves than in skin samples. It was higher in Australian-produced cultivars than in the examined commercial sample [69]. Victor et al. (2011) used garlic cultivars cultivated in four distinct parts of Andalusia, Spain. Researchers observed changes in total polyphenols and phenolic acids. This is due to the cultivation of various garlic varieties, in different places [9].

2.3 Influence of the Varieties

Different Italian garlic extracts were in vitro tested for their antibacterial and antiproliferative effects. The results revealed that the extracts had stronger antimicrobial effects against *E. coli*, *S. aureus*, and *P. aeruginosa* for the against *B. cereus*. Figure 3 shows the compound alterations in Italian garlic varieties [19].

Medina et al. [56] have noticed variations in the process and final product for three different types of garlic. The authors investigated the impact of storing periods on fresh garlic from two different agricultural seasons, 2013 and 2014. Acidity values steadily increased during the production process in all varieties adopted in the conducted study. The reduced sugar content of the var. *California* garlic

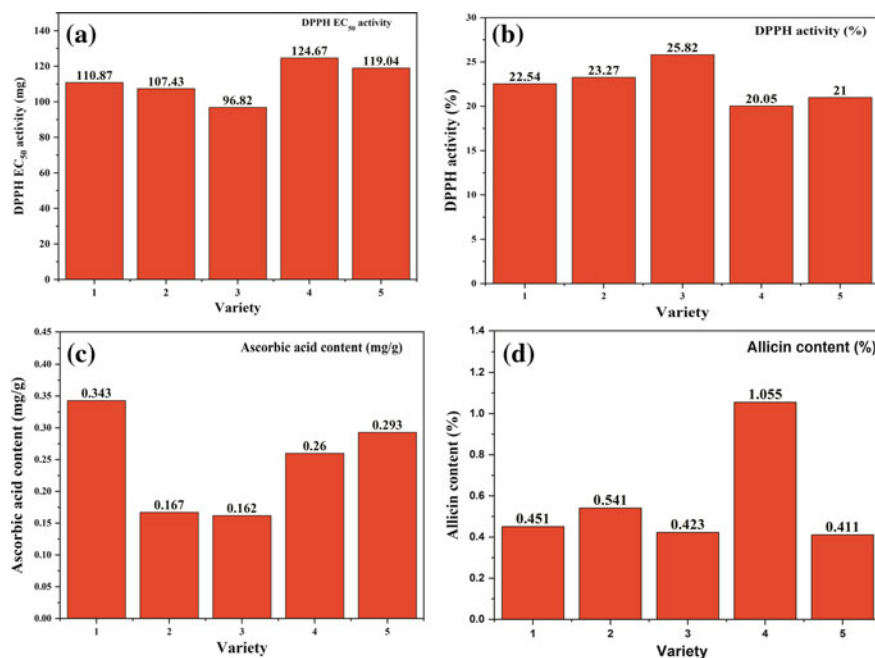


Fig. 3 Compound profile of Italian garlic varieties: 1—Schiaciato, 2—Bianco, 3—Torella, 4—Salomone, and 5—Ufita

harvested in 2013 was much more significant among all adopted varieties. Although there were substantial variations across the varieties, the pH findings were more consistent between types and harvests in 2013 and 2014. Among tests conducted with garlic from the 2013 crop and the absolute control sample from the 2014 crop, the antioxidant capacity outcomes were considerably more remarkable in the variety from *Chinese Spring* [56]. Significantly altered components in *Laba* garlic included metabolites and phenolic components. The distinctive flavor of *Laba* garlic strongly originated from the decreased organosulfur concentration and enhanced non-organosulfur content. Amino acids, sugar alcohols, organic acids, and sugars are among the key metabolites that give *Laba* garlic its sweet and sour flavor. Most phenolic components increased in concentration with time. These findings were most likely due to the bioconversion of the acetic acid of the conjugated phenolic compounds into their free forms. Compared to fresh garlic, the *Laba* garlic on day 12 has the organosulfur compound concentration reduced by 5.7%. However, non-organosulfur compound concentrations have increased by 12.8%. On day 12, the 15 phenolic compounds achieved their maximum concentrations [44].

2.4 Humidity Effect

It is generally recognized that processing humidity influences the rate of chemical processes, enzyme activity, and physical transformation in food. Humidity is crucial in the breakdown of macromolecular structures and non-enzymatic browning processes that occur at high temperatures. The humidity in the garlic treatment affected the browning and texture, the remaining moisture content, and the evaporation rate in the black garlic. Higher humidity reduced organic acid levels while increasing reducing sugars, polyphenols, and 5-HMF content [73]. According to Wang et al. [86], 85% RH and 75 °C may be ideal for producing a BG with enhanced antioxidant properties and more enticing sensory properties (Wang 2017). Prachayawarakorn et al. [71] revealed that temperature and relative humidity significantly affect the color change in garlic. The maximum browning rate occurs at 70% relative humidity [71].

2.5 Effect of Interactions Among Garlic Components

Garlic contains various chemicals. Some of these are functioning and responsive to environmental fluctuations. Interactions between functioning components such as lipids, polyphenols, water-soluble sugars, organic sulfur compounds (OSC), amino acids, vitamins, and specific mineral ions, and enzymes occur during the formation time of BG, and these result in a significant alteration of sensory properties and pharmacological substance profiles in the BG. The Maillard reaction is an excellent instance since it can involve many active chemicals as a starting reactant or an intermediary. Maillard et al. [51] illustrated how the internal compounds of BG interact with one another during manufacturing and collection. It is also possible that interaction occurs among the reactants, the intermediates, and the final products. Pyrroles, pyrazines, furans, and thiophene are the main chemicals produced during the Maillard process. These compounds are responsible for fragrance, browning, and antioxidant characteristics [51].

2.6 Effect of Black Garlic Significant Component Interactions with Favorable and Unfavorable Microorganisms

Investigations by researchers have demonstrated that the microbial metabolism processing of FG into BG may be understood by the metabolism of membrane transport, carbohydrates, and amino acids [65]. Fructooligosaccharide (FOS) has been reported to reduce *Salmonella* growth and colonization (if FOS is the only source of

carbon) and to give substantial antibacterial effects [8]. Additionally, FOSs can drastically increase the antimicrobial activities of *Lactobacillus* against *E. faecalis*, *L. monocytogenes*, *S. Enteritidis*, and *E. coli*. [63]. Short chains of fatty acids, including propionic acid, acetic acid, and formic acid, are produced as a result of intestinal microorganisms in the colon fermenting FOS. Foodborne pathogens like *S. aureus*, *S. typhimurium*, *L. monocytogenes*, *E. coli*, and *Shigella* are strongly inhibited by short-chain fatty acids, while favorable bacteria like Bifidobacteria, Lactobacilli, Bactobacilli, and Fusobacterium are incentivized [18, 24]. Melanoidins possess prebiotic action and function as antioxidant nutrition fibers that may substantially avoid digestion, travel through the upper gastrointestinal system, and initiate fermentation by the intestinal microflora in the gut to provide good health benefits such as those imparted by bread Melanoidins [10].

2.7 Pre-treatment Influence Prior to Thermal Treatment

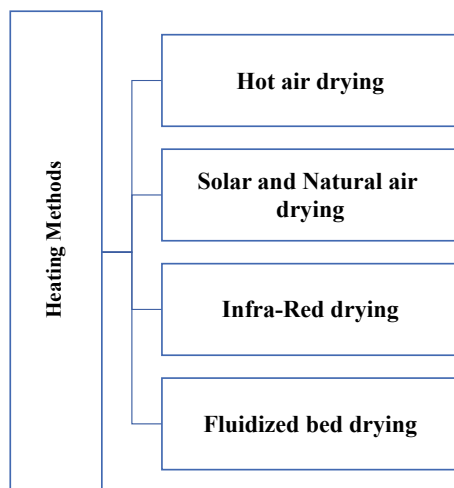
The features of the final BG products have been demonstrated to be altered by pre-treatments into BG performed prior to the thermal transformation of FG. Certain garlic pre-treatments could interrupt or even damage the cell structures of garlic and empower direct contact and interactions between cell components and environmental elements during garlic treatment. The peeled BG cloves exhibit strong antioxidant activity than the unpeeled BG cloves under the same fermentation circumstances [57]. An optimized freezing period (30 h) would substantially speed up the browning rate of the BG, reduce the operation to 22 from 90 days and improve the content of total phenols, sugar reduction, and 5-HMF by 51.88, 58.54, and 25%, and reduce Amino-N content up to 50.97% [42].

2.8 Thermal Treatment

Thermal treatment became the most popular treatment technology to treat garlic. The garlic produced from the thermal treatment at a higher temperature is known as BG. The demand for black garlic has increased due to it possessing many improved properties than the FG. Many heating methods are available to treat the garlic operated under different conditions. A few of these are shown in Fig. 4. A brief account of these is as follows.

Oven: Samples are processed for an extended period at a high temperature in the hot air oven until the weight stabilizes. Oven treatment has a severe disadvantage in the context that it takes so long, even at high temperatures, along with a diminishing quality of the dried product. Compared to oven treatment, using a microwave-hot air drying combination might hasten the drying of food products without losing the attribute of the resulting product.

Fig. 4 Heating methods for garlic treatment



Closed Chamber (i.e., controlled humidity and temperature): A constant temperature and humidity chamber is a climatic chamber that maintains a specific combination of temperature and humidity. Thereby, the facility primarily assesses heat, cold, dry, and humidity resistance.

Natural air drying: The Sun is used as the source of heat in solar drying. The temperature inside the dehydrator is raised with a foil surface. The heating process is accelerated by ventilation. Heat and mass transfer are involved in a complicated process that could affect the quality of the final product. Shrinkage, puffing, and crystallization are a few examples of possible physical modifications during solar heating. An alteration in the color, texture, odor, or other properties of food products may occasionally result from undesirable chemical or biochemical reactions. Radiation, convection, and conduction are all possible methods of providing heat. In many nations, Sun drying is a frequent practice in farming and agriculture, especially when the ambient temperature is 30°C or above. However, because of spoilage driven by unanticipated rainy days, weather conditions frequently prevent sun drying. Additionally, direct sun exposure with high temperatures on samples may result in hardening, a condition in which agricultural products develop a hard shell on the outside and thereby trap moisture within the sample [2, 25].

Infrared heating: Fruits and vegetables can be treated quickly and with less quality loss using microwaves and infrared technology [62]. Osmotically dehydrated blueberries can dry in hot air or microwave in considerably less time and with a similar or higher product quality than freeze-dried products. IR heating has many benefits over hot air oven heating, including better heat transfer rates and heat flux, which leads to a shorter drying time [79]. It was explored as a potentially useful way to improve heating effectiveness and achieve fruits and vegetables of excellent quality [83, 85].

Fluidized bed drying: With a fluidized and semi-fluidized bed, the high moisture content of garlic samples gets exposed to air with temperatures of 50, 60, 70, and 80 °C. The Page model has been the most accurate among the used models for predicting the drying pattern of the layered garlic sheets using fluidized bed dryer systems [79].

3 Influence of Thermal Treatment on the Bioactive Compounds of Garlic

Aged black garlic (ABG) is more prolific in phenols, pyruvate, and flavonoids than in fresh raw garlic (FRG). However, it has lesser allicin content. Using advanced separation and extraction techniques such as GC-O-MS, 52 flavor compounds were discovered in BG. Heterocyclic compounds were the principal producers of BG rather than sulfur-containing compounds. Based on sensory analysis, the flavor profile of black garlic was mostly composed of a roasted odor, sour, and sweet taste [88]. Kodera et al. [35] used the liquid chromatography-mass spectroscopy (LCMS) technique. Also, many unknown peaks in the aged garlic extract (AGE) were found and validated by employing Nuclear Magnetic Resonance (NMR) and MS and as possibilities for sulfur-containing compounds. Model reactions and laboratory studies simulating the aging process were used to investigate the formation mechanism of these chemicals. Researchers were able to isolate and identify three γ -glutamyl tripeptides. Additional chemicals, such as Maillard reaction products, were also identified [35].

SAC: Garlic has several health benefits, including antioxidant, anticancer, antihepatoxic, and neurotrophic properties. These effects are likely to have been initiated by the SAC. γ -glutamyl transpeptidase catalyzes the enzymatic hydrolysis of GSAC to produce SAC, as shown in Fig. 5 [17].

During the production of BG, there is also a significant change in the SAC concentration. Black garlic has SAC concentrations 5–6 times greater than fresh garlic, and typically about 20–30 mg/g [36]. The temperature has less impact on SAC concentration in comparison to the aging period. Surprisingly, despite temperature enhancement, the SAC content reduces with time [4]. Additionally, the SAC concentration was more significant for the garlic treated at a mild temperature. As the temperature increased, both pH and moisture percentage of the garlic reduced. The browning became more intense as the temperature enhanced. This is due to the heat treatment of the garlic that increased antioxidant activity [7]. During the aging process,

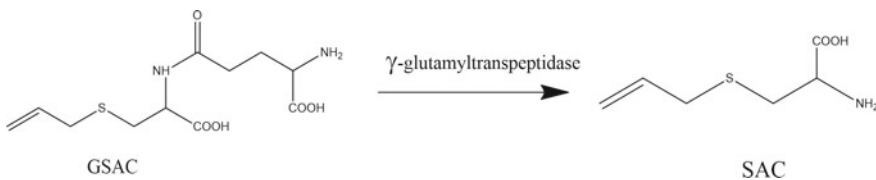


Fig. 5 Chemical reaction for the formation of S-Allyl cysteine

Table 1 Bioactive compound transformations during garlic heat treatment

Compounds	Changes from raw garlic to black garlic	References
SAC	Increased	[1, 4, 6, 74, 77]
Allicin	Decreased	[33]
Alline	Increased	[3]
Diallyl disulfide	Decreased	[20]
Diallyl trisulfide	Decreased	[20]

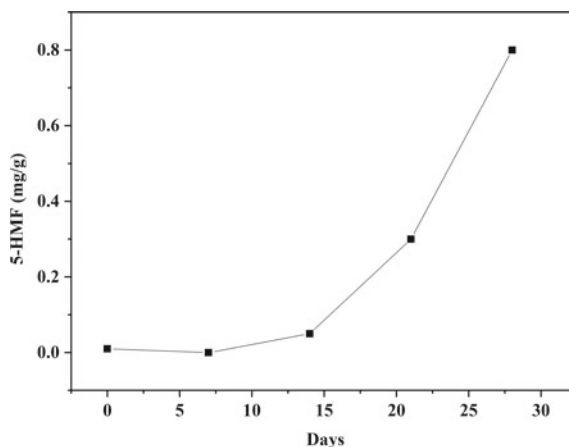
these compounds go through several changes. The study analyzed the variation in distinctive elements in raw garlic extract during aging and numerous chemical and enzymatic reactions involved in their formation. Among the various sulfur and non-sulfur bioactive compounds, a few changes with the heat treatment are mentioned in Table 1.

4 Influence of Thermal Treatment on the Maillard Reaction-Based Products of Garlic

Thermal operation is the most popular processing technique for the reduction of undesirable flavor and aroma of garlic. After processing, phenols and other non-enzymatic browning processes undergo oxidation processes. The Maillard reaction produces an Amadori component through the interaction of a reduced carbonyl sugar group and an amino group [7, 52, 67]. In these reactions, while lipids, amino acids, hydroxymethyl furfural (HMF), and protein are the byproducts, the melanoidins and polyphenol are the primary products. High temperature causes the Maillard reaction to occur more quickly and thereby enables the black garlic to accumulate significantly higher HMF content. With an altered time of garlic heating, Melanoidin, 5-HMF, and phenolic compounds are formed. Black garlic contains more polyphenols than raw garlic. When garlic is subjected to heat treatment, it causes the formation of intermediates and products [65].

HMF: HMF, a Maillard reaction byproduct, is a sign of quality decline in various foods that underwent heat processing [93]. The sources of HMF were fructose and glucose generated by sugar breakdown in the black garlic following a heat treatment [43]. Zhang et al. [92] found that varied processing temperatures had a distinct influence on the quality of BG ($p < 0.05$). The temperature affects a loss in allicin and a faster rise in the levels of total acids, total phenols, and HMF. The Maillard reaction produces HMF as an intermediate product. Among the confirmed temperatures, 70 °C appeared to be the most favorable, and thereby resulted in the most desirable sensory characteristics, larger reducing sugar amount, and appropriate constitution of total acid amount and HMF [92]. Nakagawa et al. [65] investigated total phenolic content and 5-HMF generated by the Maillard process in heated garlic. The authors carried out studies with electron paramagnetic resonance (EPR). The EPR signal intensity

Fig. 6 5-HMF concentration in the BG extract



enhanced with an increase in the period of thermal treatment (days). EPR determines not just the reaction intermediates but also the unpaired electron products. There may be a relationship between the EPR signal intensity and the brown color of garlic, which most likely correlates to melanoidin. [65] revealed that the 5-HMF level of BG does get considerably influenced with time (Fig. 6).

The Maillard process and dehydration of sugars in acidic conditions are the primary sources of generating 5-HMF. The pH, sugar type, temperature, cations, water activity, and other factors influence 5-HMF production.

Polyphenols: Pires et al. [70] found that processed black garlic improved antioxidant activity through heat treatment. Black garlic had high total phenolic components than fresh garlic [70]. Black garlic was prepared using a programmed stepwise heating schedule. Garlic subjected to various thermal processing had a significant quantity of TFC and TPC than fresh garlic. The primary phenolic acids in garlic have been identified as hydroxycinnamic acid derivatives at various processing steps [31]. Najman et al. [64] have researched the thermal treatment of garlic. The authors found that total polyphenols in the BG were more than two times than FG [64]. The total polyphenols content was 13.91 mg GAE/g and 58.33 mg GAE/g on the 0th and 21st day of aging of garlic, respectively [16]. Further, a 12-day thermal treatment increased the quantity of total polyphenols from 0.52 to 11.88 mg/g in black garlic [74]. The conducted research conveyed that the amount of polyphenols in the treated garlic depends on the treatment type, temperature, and aging periods.

Protein, fat, and sugar: Botas et al. [11] studied white and black garlic cloves. Researchers revealed that the most plentiful macronutrients were carbohydrates, followed by proteins in the garlic. BG had the most significant carbohydrate and ash content and outstanding energy contribution. Protein percentage was more effective in white garlic from the Algarve. The fat content was significantly greater in white garlic from Trásos Montes.

Fructose was the most prevalent in BG in the class of free sugars. On the other hand, garlic grown in intensive farming systems had the highest concentration of sucrose.

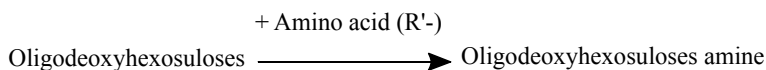


Fig. 7 Melanoidin structure based on carbohydrates

Only BG was found to have xylose. BG had the largest concentration of glucose and fructose. However, garlic from the Algarve had the lowest concentration. The sweetness of ABG is closely correlated with its high free sugar concentration, particularly fructose [41]. Fructose concentration varies significantly, and more quickly than galactose, sucrose, glucose, arabinose, and maltose [77]. Fresh garlic has no sweetness. However, it has alterations in saccharides through thermal processing. On the other hand, BG has polysaccharides with a comparatively high molecular weight post thermal treatment [49]. The thermal process describes the pathway of black garlic production. It may help improve quality control by monitoring variations in the levels of fructan, fructose, pH, and color, as well as variations in the Maillard reaction [90].

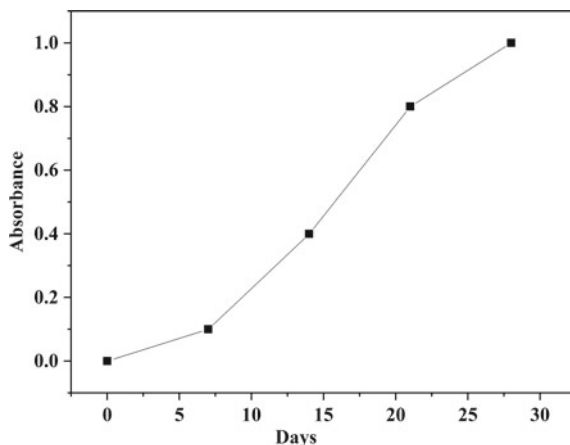
Melanoidins: Melanoidins are produced from the reaction of polysaccharides and amino acids. These compounds are negatively charged and are relatively water-soluble. Melanoidins are substances that form in the last phase of the Maillard reaction. The suggested melanoidin structure is presented in Fig. 7 [84].

Type of food, thermal treating time, temperature [75], solvent [22], pH [32], amino acid, and reducing sugar concentration do significantly influence the complex array of produced Melanoidins [55]. Melanoidins are nitrogenous substances that affect food texture and flavor by influencing the senses [58, 68]. The impact of various heat processing steps on Melanoidin was examined by Kang et al. [28]. Each sample of Melanoidin had increasing intensities in terms of molecular weight. Melanoidin produced in the BG comprised -OH, -CH, amide I, and amide III groups [28]. Melanoidin was isolated from black garlic and divided into several fractions, such as Melanoidins, Melanoidins bounded to low molecular weight compounds, and pure Melanoidin. Melanoidins bound to compounds of low molecular weight generally had higher antioxidant activity than Melanoidins in their pure form [30]. Further, Nakagawa et al. [65] have also discovered that the melanoidin content in garlic increases with time in due course of thermal processing at a particular temperature. This is depicted in Fig. 8 [65].

Acids: Twenty-seven fatty acids were discovered through the deployment of thio-barbituric acid reactive substance assay for the white garlic samples [11]. ABG demonstrates a rise in amino acid concentration which corresponds to the following compounds: glycine, threonine, tyrosine, proline, aspartic acid, alanine, methionine, isoleucine, serine, leucine, phenylalanine, and glutamic acid [41, 77].

Pre-treatment methods such as convection drying or ohmic heating may shorten black garlic production time. Ríos et al. [76] studied the generation of 2-furoylmethyl amino acids with reliable treatment to achieve high-quality products. Among others, furosine quantities were more significant in convective drying (46.6–110.1 mg/100 g protein)-based samples than those achieved with ohmic heating (13.7–42.0 mg/100 g

Fig. 8 The total melanoidin concentration at 400 nm in garlic extracts



protein) [76]. Liu et al. [45] studied model solutions utilizing garlic-neutral polysaccharide hydrolysate and seventeen different amino acids to infer the most significant amino acids that affected the BG color. The model solution with histidine indicated the highest degree of browning [45].

Antioxidant activity: In a study, 90% relative humidity was maintained while fresh garlic was heated at 72, 75, and 78 °C. It was found that the quantity of polyphenols in garlic significantly increased after heat treatment. In addition, after heat treatment, the antioxidant capacity of garlic, as measured by the ABTS radical, increased considerably [81]. The researchers examined the extracts of adenosine, uridine, 5-HMF, carboline alkaloids, and ethyl acetate for their capacity to act as cellular antioxidants. Researchers have observed that all compounds had antioxidant properties. Thus, the more significant a component constitution in the system, the greater the antioxidant activity [50]. Additionally, a high temperature and a prolonged aging period enhance the DPPH radical scavenging capacity [5]. Researchers reported that the garlic aged 21 days as opposed to 35 days has higher antioxidant activities and antioxidant content, such as total polyphenol and total flavonoid content [16]. Various sulfur, non-sulfur, and Maillard reaction-based generated bioactive compounds show higher antioxidant activity. A few bioactive compounds that exhibit higher antioxidant activity have been listed below in Table 2.

5 Conclusions

Several heating methods exist for the thermal treatment of garlic. Therefore, each thermal treatment method has its own benefits and drawbacks. According to the study, thermal-treated food produces byproducts that alter the flavor and appearance of the food. Thermal processing of garlic alters its texture and as well enhances its antimicrobial and antioxidant properties. Black garlic had much higher concentrations of

Table 2 Transformations and alterations in Maillard reaction products during garlic heat treatment

Compounds	Changes from raw to black garlic	References
Amadori & Heyns	Increased	[90]
Polyphenol	Increased	[16, 21]
Water soluble sugar	Increased	[90]
Melanoidin	Increased	[28, 65, 87]
Antioxidant activity	Increased	[29, 81, 86]
[Polyphenols	Increased	[14, 64, 80]
HMF	Increased	[40, 43, 86]

S-allyl-cysteine, reducing sugar, total polyphenol, protein, HMF, and Melanoidin than fresh garlic.

Additionally, it was found that the quantity of bioactive compounds increases with the aging period of the garlic. The shelf life of food is often extended by thermal treatment at a desired temperature that renders the microorganisms to become inactive. This review offers current knowledge on the variations in bioactive compounds and products based on the Maillard reaction in due course of the thermal processing of garlic. Based on the literature review, thermally treated garlic has several advantages over fresh garlic. These include increased bioactive compounds and antioxidant properties. Black garlic had a significantly higher concentration of bioactive substances, reducing sugars, total polyphenol, and protein in comparison to fresh garlic. Therefore, black garlic treated with thermal technology could aid in the mitigation and growth of contaminating microorganisms. Researchers may further enhance garlic with enhanced pharmacological activities to develop medicines for human consumption. Further research can be targeted on the influence of integrated techniques such as thermal-thermal and microwave-thermal combinations on black garlic production. These may reduce microbial activity and enhance garlic shelf life. To confirm the therapeutic effects of treated garlic in humans, researchers should concentrate on conducting more clinical research using thermal-treated garlic.

Author Credit

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Optimality of Process Parameters During Refractance Window Drying of Ginger



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Abstract This article presents an optimization study on the refractance window drying (RWD) of ginger. Here, two optimization methods, namely the trial-and-error approach and Response Surface Methodology-based optimization were followed for the determination of least drying time with respect to optimized nutritional parameters. In the trial-and-error methodology, film thickness (5–14 milli inches or mils), sample thickness (1–2 mm), and temperature (65–95 °C) were varied to observe and evaluate the optimal time, moisture content, antioxidant activity percentage, total phenolic content, and total flavonoid content. Following the trial-and-error methodology, the best results were obtained for ginger slices of thickness 1 mm, film thickness of 10 milli inches (mil), and drying temperature of 95 °C. To design the trials for subsequent model generation and optimization of RWD process factors, RSM's Box-Behnken design (BBD) was utilized. The models developed were then analyzed and optimized for obtaining optimal values considering studied nutritional elements. The influence of drying temperature, time, and air velocity on nutritional parameters such as total antioxidant activity percentage, total phenolic content, and total flavonoid content was studied through the RSM-based optimization procedure. As indicated by the optimized results, the optimal drying temperature, drying time, and air velocity were found to be 95 °C, 135 min, and 0.5 m/s, respectively. The observed and anticipated values did not significantly differ ($p > 0.05$) and confirmed that the

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response surface models portrayed the effects of refractance window drying on the nutritional properties of ginger were fairly accurate.

Keywords Box-Behnken design · Ginger · Optimization · Physicochemical properties · Refractance window drying · Response surface methodology

1 Introduction

Ginger (*Zingiber officinale*) is a flowering medicinal perennial herb from the Zingiberaceae family. This plant is of Asian origin and is widely considered to have originated from Maritime Southeast Asia. Primary nutritional elements of ginger consist of protein, carbohydrates, total dietary fiber, sugars, calcium, magnesium, potassium, phosphorus, sodium, and Vitamin C, which are a few important constituents mentioned in the USDA. Although ginger is primarily valued as a medicinal component, it also finds its application in varied kinds of consumable and non-consumable products, such as medicines, salad dressings, pickles, gravies, meat sausages, tomato ketchup and sauces, bakery items, wine, and even toiletry products. Ginger is a high-importance plant and is extensively cultivated in China, India, Nigeria, Japan, Indonesia, Nepal, and Thailand. India is the leading producer of ginger, with a cultivation area of over 164,000 ha and a yield of 109,024 hg/ha. India accounts for 43.81% of the total ginger production in the world [1].

Approximately 50% of the gross ginger production is used as green ginger, 30% as dry ginger, and the rest goes toward fresh ginger plantations. Although ginger is cultivated in almost all parts of India, the states having significant contributions toward its production are Assam, Orissa, Meghalaya, Gujarat, Arunachal Pradesh, Madhya Pradesh, Kerala, Sikkim, Maharashtra, and West Bengal [2]. In terms of the importance of usability, dried ginger is the most important product after fresh ginger. This is because dried ginger is the raw material for the production of ginger powder, and for oleoresin extraction and ginger oil. Thus, dried ginger rhizomes are the raw materials for making ginger powder, oleoresin extraction, and ginger oil production. Dried ginger is made from completely formed rhizomes that have been dried at maturity when the rhizomes have a fully developed aroma, flavor, and pungency. Dried ginger can also be preserved for a long time. Ginger powder is rarely applied in its pure form and is majorly utilized in the production of ayurvedic medications, extraction of ginger oil, ginger oleoresin, food additive, and pharmaceuticals [2]. The color of ginger rhizomes has been reported to have altered after drying in a tray dryer. For higher quality dried products, the ginger rhizomes should be sliced, blanched, and dried at temperatures below 70 °C. Raising the drying temperature for ginger rhizomes has resulted in darker-colored products [3].

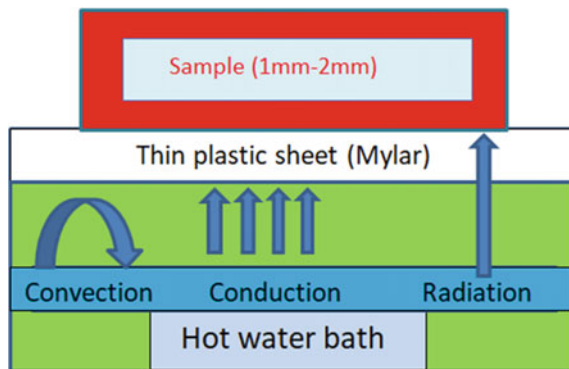
Varied drying processes including vacuum, tray, freeze-drying, oven, solar, and refractance window drying have been used for the development of intermediate and high-valued food products. RWD is a cutting-edge drying method that allows obtaining dried products of exceptional quality having high retention of color, flavor,

vitamins, and phytochemicals. From the perspective of resource consumption, RWD is an energy-efficient drying methodology that provides better drying performance efficiencies.

Because of its effectiveness, low upkeep, and simple drying setup, refractance window drying (RWD) has demonstrated several benefits [4]. RWD typically uses circulating water having a temperature from 95 to 97 °C at ambient air pressure to impart thermal energy to food products kept for drying. A simplified RWD schematic has been presented in Fig. 1. In the process, heat transfer occurs during warm water circulation via an interface (such as plastic) to the dried material. In the majority of cases of RWD-based drying, the actual sample temperature is often less than 70 °C. Even if the temperature of the water is approaching near boiling point, the sample temperature in RWD is limited to a drying temperature lower than 70 °C. The translucent plastic Mylar sheet allows liquid or semi-viscous products such as juices and purees to spread. Due to the thin construction of Mylar film, heat transfer from the water to the sample is not challenging. In a continuous RWD process, the heat is transferred to the sample during conveyor movement due to the water cycling on shallow troughs. During drying, the product no longer remains in direct contact with the heat switch medium. Hence, no cross-contamination occurs. Since water is getting recycled and reused, the system’s thermal efficiency also increases.

Pragati and Preeti [5] conducted research to study the effects of refractance window drying on the alteration in moisture levels of purees and the associated energy efficiency of the process. The authors altered the water temperature from 92 to 95 °C and the drying period from 3 to 5 min. Product temperatures were in-between 70 and 75 °C. It was observed that drying for 5 min at 95 °C produced the best results (a moisture content reduction of 7.5% and an energy efficiency of 77–52%). Nindo et al. [6] conducted a targeted study on refractance window drying of asparagus. The authors varied the water temperature in the range 95–98 °C and the drying period from 3 to 5 min and thereby evaluated how these parameters affected the antioxidant activity, molecular weight distribution, and moisture content of asparagus. Drying at 98 °C for 4 min was determined to be optimal (antioxidant activity of 0.3%, molecular weight distribution of 10 kd, and a moisture content of 4%). Nindo and Tang

Fig. 1 Schematic of refractance window drying process scheme



[4] performed research to determine the effect of drying temperature and duration on the water activity and moisture content of carrots. The authors altered the water temperature from 95 to 97 °C and the drying period from 3.5 to 4 min. The maximum allowed product temperature was 70 °C. The optimal drying time was determined to be 4 min at 97 °C, at which a moisture content of 4% and a water activity of 0.7 was obtained.

Drying process parameters optimization is necessary to maximize the values of heat-sensitive nutritional components such as total antioxidant activity percentage (AA), total flavonoid content (TFC), and total phenolic content (TPC) in the dried food product. Two alternate methodologies can be used to target the drying process-based process parametric optimization. The first is a trial-and-error strategy that involves the evaluation of drying kinetics, moisture variation characteristics, activation energy, and moisture diffusivity. And the second involves employing appropriate statistical methods for experimental design in food processing. Thereby, it involves the response surface approach (RSM) for the identification of process parameters and thereby maximizes nutritional components such as total AA percentage, TPC, and TFC.

The considered drying kinetics analysis approach employs a trial-and-error method to determine parametric optimality, as well as the computation of diffusivity, mass transfer kinetics, and variations in antioxidant activity. In RSM-based optimization, experimental designs using statistics, comparison of variance (ANOVA), optimization, and model fitness were utilized to determine the optimal process parameters. In this optimization methodology, individual response surface plots were utilized to depict how the variation of responses occurred during the drying period and temperature alteration. RSM's numerical optimization tool can be used to improve process parameters while reducing moisture content and enhancing antioxidant activity during the drying process.

According to the available prior art, research has been focused on the RWD of various vegetables and purees. However, no study has examined the impact of RWD on the nutritional and drying properties of ginger. Most research, even those involving other vegetables, solely looked at how drying affected water activity, water activity, and diffusivity. Therefore, the current study focuses on determining RWD parameters for ginger's moisture content, total phenol concentration, antioxidant activity, and total flavonoid content.

There also exists a research gap on the drying kinetics of ginger, as no research has been found with respect to the targeted optimization of RWD parameters using RSM. Thus, the primary objectives of the presented research are to investigate the drying kinetics of ginger using various models and to optimize the process parameters of the considered drying technique with respect to the nutritional properties of ginger using both trial-and-error and RSM approaches.

2 Materials and Methods

2.1 Materials

Raw ginger rhizomes were purchased from a local vegetable market near Indian Institute of Technology Guwahati, Guwahati, Assam, India. Chemicals used were ethanol (99.5% pure, M Tedia, USA), sodium carbonate (99.5% pure, Merck, India), Diphenyl-2, 2-1-Picreylhydrazyl (DPPH) (extra pure), Folin-Ciocalteu reagent (Sisco Research Laboratories Pvt. Ltd, India), and bovine serum albumin (SRL Pvt. Ltd.).

2.2 Sample Preparation

The procured fresh ginger rhizomes were cleaned by washing them under running water for the removal of dirt, mud, and other impurities. The cleaned rhizomes were then cut into slices of 1 and 2 mm thickness with an adjustable slicer.

2.3 Experimental Setup for Refractance Window Drying

The adapted refractance window drying setup consisted of a water bath with a digital controller, a display, and a Mylar film of thickness 1–2 mm. The water bath was filled to the required level, and a waiting time was followed to reach the desired temperature (65–95 °C). As the water temperature reached the desired value, the Mylar film was spread over the water's surface. The weighed sample was then placed over the Mylar film and kept for drying.

2.4 Drying Kinetics

Drying period: The samples' original weights (cut into 1 and 2 mm thick slices) were taken. Then, these samples were placed on the Mylar sheet for drying. The ginger slices were dried at four different temperatures—65, 75, 85, and 95. These samples were weighed at a regular interval of 30 min. However, for the samples which were being dried at 95 °C, after 1 h duration of the drying process, weight measurements were taken at regular intervals of 15 min, as a higher heat and mass transfer rate was observed in comparison to the samples being dried at lower temperatures.

Fitness of Drying Kinetics Models: The drying characteristics of ginger slices dried in an experimental RWD were investigated through the moisture removal rate.

Table 1 A summary of deployed drying kinetics models in the conducted investigations

S. No.	Model name	Model equation
1	Lewis	$MR = \exp(-kt)$
2	Page	$MR = \exp(-kt^n)$
3	Modified page	$MR = \exp[-(kt)^n]$
4	Henderson and Pabis	$MR = a \exp(-kt)$
5	Logarithmic	$MR = a \exp(-kt) + b$
6	Two term	$MR = a \exp(-k_1t) + b \exp(-k_2t)$
7	Two term exponential	$MR = a \exp(-kt) + (1 - a) \exp(-kat)$
8	Approximation of diffusion	$MR = a \exp(-kt) + (1 - a) \exp(-kbt)$
9	Wang and Singh	$MR = a + bt + ct^2$
10	Midilli et al.	$MR = a \exp(-kt^n) + bt$

The moisture removal rate enhanced with drying temperature. For all drying temperatures of 65, 75, 85, and 95 °C, the declining rate of moisture removal from the ginger slices was studied. Several thin-layer drying models were deployed for the study of the drying kinetics of fresh ginger slices. Different models used in this study have been presented in Table 2. The data obtained from the drying of the ginger slices at given temperatures was fit into the presented models, and the respective fitness values with the drying curves were obtained. The significance of these predicted models was determined by conducting a non-linear regression analysis and with the determined R^2 values.

The drying characteristics of the samples were described using various model types using both on-empirical and empirical models (outlined in Table 1). According to the highest R^2 and the lowest chi-square and squared root (RSS) values, the optimum model that illustrates the pertinent behavior of the material during the drying process has been identified.

In order to determine the best fit model for determining the measured drying properties, varied drying model expressions were explored. The drying kinetics curve is well recognized to have an intricate relationship between several factors, including not only drying time, temperature, and sample thickness but also drying technique, initial moisture of material to be dried, and inherent properties of the material. As a result of these complicated interactions, numerous alternative drying models must be considered to reflect measured drying kinetics data. Because of this, empirical analysis can be used to understand the particular system's mass transfer behavior.

Various empirical models (shown in Tables 1 and 2) have been extensively employed to affirm the fitness parameters of dried food products. Resistances to transfer in the external surrounding environment were taken into account during the employment of these models for moisture transport.

These modeling tools rely heavily on the measured experimental data for evaluating relevant drying behavior. Internal resistance to moisture transport is an essential

Table 2 Fitness parameters of alternate drying kinetics models to represent RWD of ginger

Temp (°C)	Sample thickness (mm)	Mylar film (mil) 5 (R ²)	Models	Mylar film 10 (R ²)	Best fit models	Mylar film 14 (R ²)	Models
65	1	0.9827	Singh et al.	0.9996	Page	0.9788	Singh et al.
	2	0.9970		0.9903	Singh et al.	0.9990	Approximation of diffusion
75	1	0.9807		0.9854	Page	0.9654	Singh et al.
	2	0.9849		0.9854	Singh et al.	0.9989	Approximation of diffusion
85	1	0.9936	Approximation of diffusion	0.9998	Page	0.9922	Newton
	2	0.999	Log arithmetic	0.9976	Approximation of diffusion	0.9852	Singh et al.
95	1	0.9905	Singh et al.	0.9949	Log arithmetic	0.9999	Page
	2	0.9930	Approximation of diffusion	0.9993	Approximation of diffusion	0.9835	

assumption in several theoretical models, although this is not completely accurate. Given the presumption that external moisture transport resistance is substantially higher than interior moisture transport resistance, the validity of such an assumption is quite poor (Onwude et al. 2016).

2.5 Determination of Moisture Content

By using the hot-air oven drying technique, the sample’s moisture content was ascertained. A predetermined amount of material was kept at 105 °C for 12 h and then weighed after resting it in a desiccator [7, 8–10]. Moisture content of the ginger slices was calculated using the following equation:

$$\text{Moisture content (\%)} = \frac{w_i - w_f}{w_i} \times 100 \tag{1}$$

where w_i and w_f indicate the initial weight of the sample before drying and the final weight after drying, respectively.

2.6 Determination of Moisture Ratio

Moisture ratio was determined by the method given by Shittu and Raji (2011) and with the expression:

$$\text{Moisture ratio (MR)} = \left(\frac{M_t - M_e}{M_0 - M_e} \right) \quad (2)$$

where M_0 , M_t , and M_e are the initial moisture content (% db), moisture content at time t (% db), and equilibrium moisture content (% db), respectively.

2.7 Activation Energy and Moisture Diffusivity

The drying behavior of agro-produce can be modeled utilizing Fick's diffusion law. For diffusion in an endless planar space, Akpınar [11] presented the following analytical solution for a long drying time:

$$\text{MR} = \frac{M}{M_0} = \frac{8}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{(2n-1)^2} \exp\left(\frac{(2n-1)^2 \pi^2 Dt}{4L^2}\right) \quad (3)$$

where MR stands for moisture ratio, M for moisture content in kg water/kg dw at any moment of time, M_0 for initial moisture in kg water/kg dw, t for drying time in seconds, D for moisture diffusivity in m^2/s , and L for half the thickness of fresh ginger slice in m. Here, n represents the number of terms considered (e.g. 1, 2, and 3). For prolonged drying periods [12], only the first term of Eq. (3) is applicable. Thereby, the following equation is relevant:

$$\text{MR} = \frac{8}{\pi^2} \exp\left(\frac{-\pi^2 Dt}{4L^2}\right) \quad (4)$$

The plot of $\ln(\text{MR})$ versus time derived from Eq. (4) yields a straight line having a negative slope denoted by K_2 and as follows:

$$K_2 = -\frac{\pi^2 D}{4L^2} \quad (5)$$

An equation of the form of the Arrhenius equation has been utilized to compute activation energy [12, 13]:

$$D = D_0 \exp\left(-\frac{E_a}{RT_a}\right) \quad (6)$$

where E_a is the activation energy in kJ/mol, R is the standard gas constant in kJ/mol K, T_a is the actual air temperature in K, and D_0 is the Arrhenius equation's preexponential element in m^2/s .

The slope of the plot of Arrhenius $\ln(D)$ versus $1/T_a$ can be used to calculate the activation energy. From Eq. (6), the plot of the above yields a straight line with the slope K_3 , and is as follows:

$$K_3 = \frac{E_a}{R} \quad (7)$$

2.8 Determination of Antioxidant Activity

The prior art [14] reported the methanol-based extraction technique was used to determine the antioxidant activity. Here, 10 mg of ginger was mixed with 20 mL of 100% methanol, and thereafter a paste of the mixture was prepared with a mortar and pestle. Then, it was sonicated for 30 min before being filtered. 20-mg DPPH reagent was mixed in 100 mL of pure methanol to make the DPPH solution. 1 mL filtrate was combined with 3 mL freshly prepared DPPH solution and was stored for 30 min at room temperature in complete darkness. The control was 3 mL of methanolic DPPH solution comprising 1 mL methanol. The absorbance at 517 nm was assessed with a UV spectrophotometer. The samples' antioxidant values were evaluated using the following expression:

$$A_a = \frac{A_0 - A'}{A_0} \times 100 \quad (8)$$

where A_a , A_0 , and A' denote antioxidant activity, absorbance of the control solution, and absorbance of the sample solution, respectively.

2.9 Determination of Total Phenol Content

Methanol-based extraction technique was followed to assess total phenol concentration, as per DemLa and Verma [14]. To generate a sample solution with a 1 mg/mL concentration, the ginger sample was dissolved in 1 mL of 50% methanol first by means of a vortex mixer (Touch Type), and was subsequently agitated using sonication. A test tube was filled with 3.5 mL distilled water and with 0.25 mL Folin-Ciocalteu reagent (FCR). The sample solution was then incubated for 1 to 8 min at standard temperature. The then-obtained sample solution was then incubated for 2 h after mixing with 0.75 mL of 20% Na_2CO_3 solution. Duplicates of the samples were made. UV-spectrophotometer was utilized to check the absorbance at 765 nm against

a reagent blank, and instead of extract solution, 0.1 mL of 50% methanol–water solution was used to make the blank. The total phenol concentration was calculated in terms of gallic acid equivalent (GAE) and with the calibration curve.

2.10 Determination of Total Flavonoid Content

Methanol-based extraction technique was used to assess total flavonoid concentration, as detailed by DemLa and Verma [14]. In 1 mL of 80% ethanol, 10 mg of ginger sample was weighed and dissolved using a vortex mixer (Touch Type). An addition of 9 mL 80% ethanol was done to the sample solution, followed by sonication-based mixing to prepare a sample solution with a 1 mg/mL concentration. 0.5 mL of this mixture solution was pipetted into a vial containing 1.5 mL methanol, 0.1 mL 10% aluminum chloride, 0.1 mL 1 M potassium acetate aqueous solution, and 2.8 mL of distilled water. The presence of flavonoids was indicated by the yellow coloring of the solution. At room temperature, the prepared samples were incubated for 30 min. Finally, total flavonoid content was evaluated in terms of mg quercetin/g dry weight (mg QE/g dw) by establishing a reference curve for quercetin based on 415 nm absorbance measurements in a UV–Vis spectrophotometer against a reagent blank.

2.11 Drying Procedure

Fresh ginger rhizomes were cut into slices of 1–2 mm thickness and dried in a refractance window drying apparatus at varied temperatures (65–95 °C), air velocities (0.5–1.0 m/s), and for different drying times (135–300 min) and with the Box-Behnken design (BBD) (Table 3). Samples were flipped over after every 30 min during the drying process to ensure uniform heat dispersion. To equilibrate the temperature, the dryer system was turned on for 1 h prior to the drying process.

2.12 Experimental Design

For the rotatable BBD RSM, the air velocity during drying altered from 0.5 to 1.0 m/s, drying temperature altered from 65 to 95 °C, and drying time altered from 135 to 300 min. The temperature and time value limits were obtained through initial trial-and-error investigations. The RSM-based BBD design yielded 15 drying sets of experimental combinations of which 8 were at the center. The response variables have been expressed, and temperature polynomial expression was used as a function of X_1 (drying temperature), X_2 (drying air velocity), and X_3 (drying time), on three response variables Y_1 , Y_2 , and Y_3 , representing total AA percentage, TPC, and TFC, respectively.

Table 3 ANOVA data summary for various responses

Components	Antioxidant activity		Total phenol content		Total flavonoids content	
	F-value	p-value	F-value	p-value	F-value	p-value
Model	65.39 (quadratic)	0.0001 (quadratic)	297.59 (quadratic)	<0.0001 (quadratic)	1268.96 (quadratic)	<0.0001 (quadratic)
A	1.88	<0.0001	1840.02	<0.0001	4544.10	0.0001
B	2.35	0.5795	3.51	0.1201	5.04	0.2127
C	12.90	0.0157	15.95	0.0587	17.08	0.0091
AB	0.84	0.4021	1.10	0.3432	29.96	0.0028
AC	87.81	0.0002	613.11	0.0001	2618.70	0.0001
BC	7.51	0.0408	1.25	0.3148	0.025	0.8796
A ²	20.06	0.0065	60.64	0.0006	613.55	0.0001
B ²	0.19	0.6794	130.52	<0.0001	1976.70	0.0001
C ²	3.16	0.1160	52.46	0.0008	1524.77	0.0001
Lack of fit	1.94	0.3578	2.73	0.2795	1.37	0.4477
R-squared	0.9916		0.9961		0.9996	
Adequate precision	29.927		64.474		121.053	

2.13 Statistical Analysis

The experiments were planned with the Design Expert software (Version 7.0.0) and with a Box-Behnken spherical specification to aid the current study focusing on the independent components' impact on the preferred outcomes. The models were compared, and the model having the optimum fit was chosen according to the combination of the allowed p-value and a high F-value. All responses were submitted to a statistical analysis of variance (ANOVA). A combination of p-values and F-values was used to account for the importance and relevance of the model terms. The model fitness was demonstrated by the lower F-values for the responses' fit. Coefficient of variation, PRESS values, R², and its closeness to adjusted R² and predicted R² values have been used to judge the model's adequacy.

3 Results and Discussion

3.1 Optimality of Time, Temperature, Sample Thickness, and Mylar Film Thickness

The drying kinetics of ginger using the RWD-based drying technique was studied by varying sample thickness, temperature, and Mylar film thickness of 1–2 mm,

65–95 °C, and 5–15 mil, respectively. Average moisture content removal of 92–90% (wb) for 1 mm thick ginger slices and 85–87% (wb) for 2 mm thick ginger slices was observed at all temperatures. For the case of 1 mm samples, the best results were obtained at 95 °C temperature and 1.15 h time, and for 2 mm sample thickness, best results were observed at 95 °C temperature, 2.45 h time, and for a Mylar film thickness of 10 mil. From the moisture ratio versus time graph, it can be observed that the diffusion coefficient for ginger samples of a thickness of 1 mm was higher for all elevated temperatures, and for the samples with a 2 mm thickness, the diffusion coefficient was comparatively lower.

3.2 Activation Energy and Moisture Diffusivity Calculation

Moisture diffusion has been calculated using Eq. (8) and was found to alter from 2.310×10^{-11} to $1.6810 \times 10^{-10} \frac{m^2}{s}$ for ginger samples in the temperature range of 65–95 °C. A maximum moisture diffusivity of $1.68 \times 10^{-10} \frac{m^2}{s}$ and a minimum of $2.30 \times 10^{-11} \frac{m^2}{s}$ were obtained. Aghbashlo et al. [15] reported similar results for fruit barberries. According to relevant literature [16], this parameter ranged from 6.51 to $8.32 \times 10^{-9} \frac{m^2}{s}$ for apricots.

Equation (6) was used to compute the activation energy for each sample thickness (1 and 2 mm) and varied Mylar film thicknesses (5, 10, and 14 mil) (10). In a study conducted by Bablis et al. [17] on barberries, activation energies ranging from 30.8 to 48.47 kJ/mol were observed. According to Aghbashlo et al. [15], activation energies ranging from 110.837 to 130.61 kJ/mol for temperatures from 40 to 70 °C were noted. The value of E_a in this investigation ranged from 26.54 to 28.92 kJ/mol for two distinct sample thicknesses and altered Mylar film thicknesses.

3.3 Antioxidant Activity

Antioxidant activities of 50–70% for 1 mm thick samples and 60–75% for 2 mm thick samples were obtained. The best results of 70–72% for 1 mm thick ginger slices (Fig. 3a) and 73–75% for 2 mm thick ginger slices (Fig. 3b) were obtained at 95 °C and 10 mil Mylar film thickness. Higher antioxidant activity values have been observed in the case of 10 mil thick Mylar film. This is due to the shorter drying time in comparison with 5 mil and 14 mil thick Mylar films. Drying for longer durations might have resulted in the degradation of the heat-sensitive antioxidants (Fig. 2).

The moisture content was low in ginger slices of 1 mm thickness in comparison with the 2 mm thick slices. Shorter drying times are preferred for optimal nutritional characteristics as drying for longer durations often results in the loss or minimization of heat-sensitive nutritional components. At 95 °C, the drying time was less for 5 mil thick Mylar film in comparison with the 10 mil thick Mylar film and the rate of heat

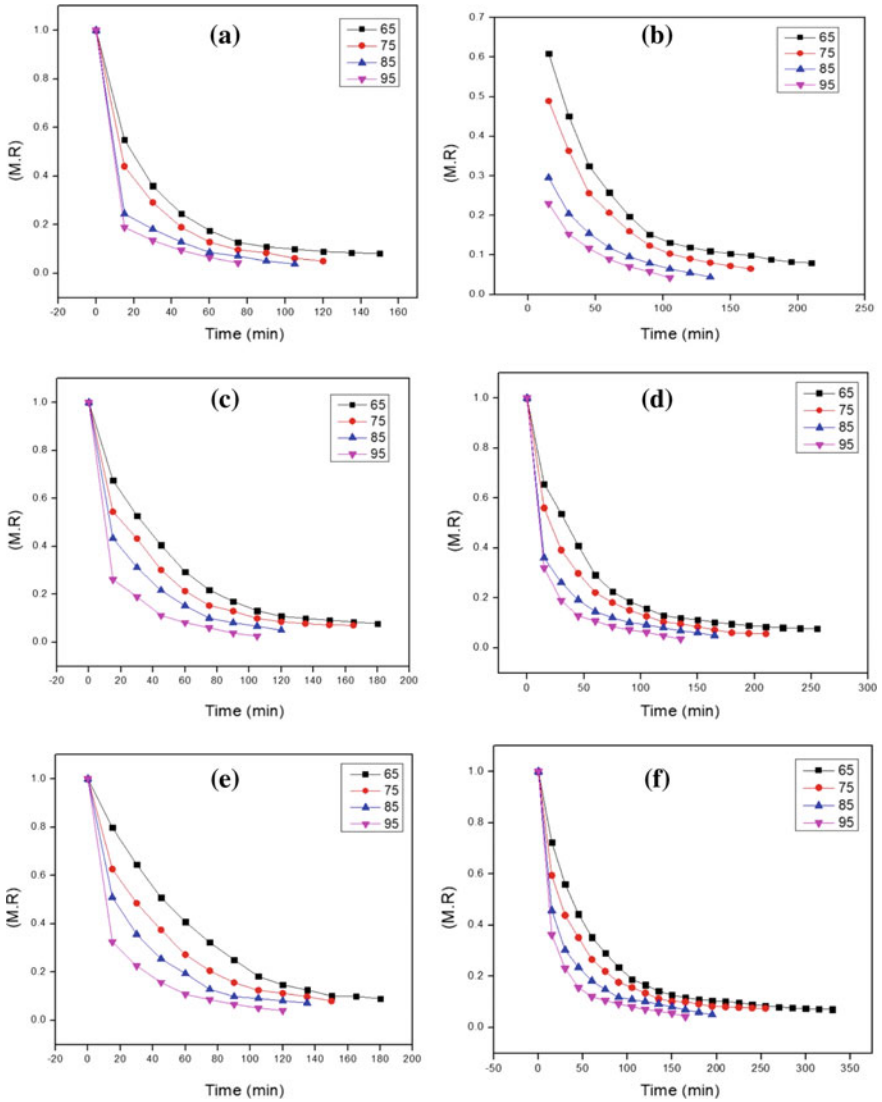


Fig. 2 Plots depicting **a** MR versus time for 1 mm sample thickness on 5 mil Mylar film thickness; **b** MR versus time for 2 mm sample thickness on 5 mil Mylar film thickness; **c** MR versus time for 1 mm sample thickness on 10 mil Mylar film thickness; **d** MR versus time for 2 mm sample thickness on 10 mil Mylar film thickness; **e** MR versus time for 1 mm sample thickness on 14 mil Mylar film thickness; **f** MR versus time for 2 mm sample thickness on 14 mil Mylar film thickness

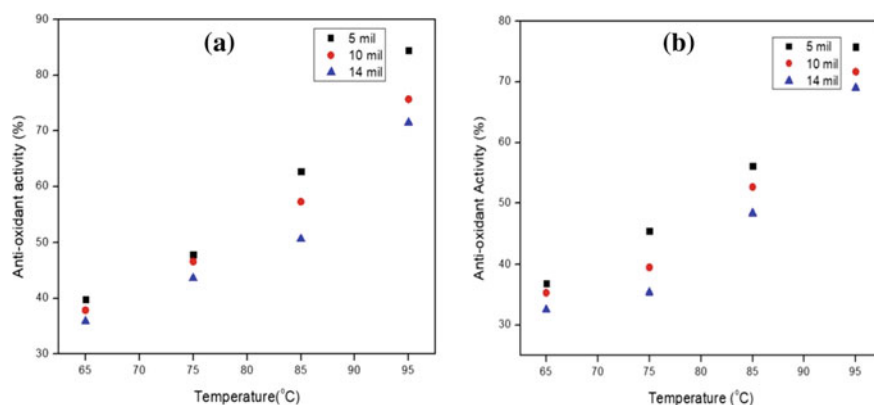


Fig. 3 Variation of antioxidant activity with temperature for **a** 1 mm and **b** 2 mm sample thicknesses

transfer was also analyzed to be faster in 1 mm thick samples due to lesser sample thickness [8–10].

3.4 Total Phenol Content

A total phenol content of 150–200 mg GAE/g dry sample for 1 mm sample thickness and 150–180 mg GAE/g dw for 2 mm sample thickness were obtained. The best results obtained were 230–235 mg GAE/g dw for 1 mm thick slices (Fig. 4a) and 205–210 mg GAE/g dw for 2 mm (Fig. 4b) thick slices at 95 °C temperature and 10 mil Mylar film thickness. This case might be due to the shorter drying time achieved with 10 mil Mylar film in conjunction with 5 mil and 14 mil thick Mylar film thicknesses.

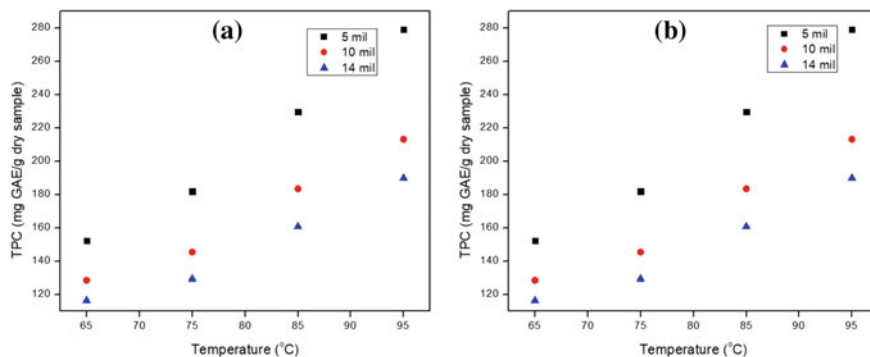


Fig. 4 Variation of TPC with temperature for **a** 1 mm and **b** 2 mm sample thicknesses

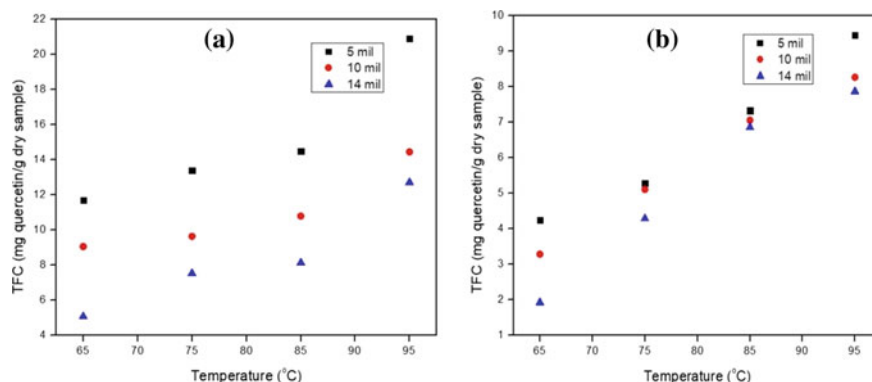


Fig. 5 Variation of TFC with temperature for **a** 1 mm and **b** 2 mm sample thicknesses

3.5 Total Flavonoid Content

A total flavonoid content of 15–18 mg quercetin/g dw for ginger slices of 1 mm sample thickness and 10–12 mg quercetin/g dw for ginger slices of 2 mm sample thickness were obtained. The best results obtained were 18–19 mg quercetin/g dw for 1 mm ginger slices (Fig. 5a) and 9–10 mg quercetin/g dw for 2 mm ginger slices (Fig. 5b) at 95 °C and 10 mil Mylar film thicknesses. Higher total flavonoid content was obtained for the case of 10 mil thick Mylar film due to the shorter drying time achieved with 10 mil Mylar film in conjunction with 5 mil and 14 mil thick Mylar film thicknesses. As flavonoids are heat-sensitive, the total flavonoid content may reduce upon drying for longer durations.

3.6 Fitting the Response Surface Models

Multiple regression analyses were carried out for the evaluation of regression coefficients and statistically significant model terms using response surface methodology. In order to determine an overall optimum range for the response variables, measured experimental data was fit into different mathematical models. Each response (Y_i) was evaluated in relation to temperature (X_1), air velocity (X_2), and time (X_3) using response surface analysis.

Table 3 displays the predicted coefficients of the conducted regression study in response surface models, together with the related R^2 values and the lack of fit test values. At a 5% confidence level, the regression models for the output responses were found to be just significant ($p \leq 0.05$). The final reduced models were found to possess significant fitness ($p \leq 0.05$) for the three response variables studied: total AA percentage, TPC, and TFC, and the R^2 values for all responses altered from 0.991 to 0.999 (Table 3).

The fitness results were thereafter evaluated using ANOVA to determine the ‘goodness of fit.’ In the final reduced model, only terms that were deemed to be significant ($p < 0.05$) were included. The best fit models addressed the significance of quadratic, interactive, and linear process factors in influencing the response variable trends. The sign and value of the coefficients revealed the variables’ impact on the response (Table 3). With relation to the response variables analyzed, the lack of fit, which is used to gauge model fitness, had no significant F value ($p > 0.05$). This indicated the models’ suitability for predicting the response variations.

3.7 Response Surface Methodology Data Summary

Total antioxidant activity percentage, phenolic content, and flavonoid content were measured in ginger samples dried using a varied combination of drying parameters. Second-order polynomial models were fitted for each of the three responses, namely AA, TPC, and TFC, in terms of independent variables such as drying temperature, drying time, and drying air velocity. Thereafter, experiments have been conducted.

Design Expert 7.0 was used to find a solution to the non-linear optimization problem. At a significance threshold of 0.05, ANOVA was used to perform the analysis of variance. A significant model was identified with a $p < 0.05$ and vice versa for an insignificant model.

The generic model equation can be given as

$$\begin{aligned} R_i = & \beta_0 + \beta_1A + \beta_2B + \beta_3C + \beta_{11}A^2 + \beta_{22}B^2 + \beta_{33}C^2 \\ & + \beta_{12}AB + \beta_{23}BC + \beta_{13}AC \end{aligned} \quad (9)$$

where **A**, **B**, and **C** represent temperature, air velocity, and time, respectively, and all the ‘ β ’ terms are coefficients.

Similarly, for RWD, the generic model equation can be written as

$$R_i = \beta_0 + \beta_1A + \beta_2B + \beta_{11}A^2 + \beta_{22}B^2 + \beta_{12}AB \quad (10)$$

where R_i represents responses, which include total AA percentage, TPC, and TFC. **A**, **B**, and **C** represent temperature, air velocity, and time, respectively.

3.8 Model Fitness

The response variables’ mathematical models were represented using quadratic expressions derived from the Box-Behnken Design (BBD).

The R -squared values, p -values, and F -values suggest the combinatorial significance of the model, each independent variable, and quadratic terms associated with

the response variables (Table 3). For each case, the lack of fit was discovered to be insignificant (Table 4).

The following are the quadratic model equations for the responses:

$$\begin{aligned} \text{AA} = & 50.52 + 14.57\text{A} - 0.41\text{B} - 2.46\text{C} + 0.88\text{AB} \\ & - 9.06\text{AC} + 2.65\text{BC} - 4.51\text{A}^2 - 0.44\text{B}^2 + 1.91\text{C}^2 \end{aligned} \quad (11)$$

$$\begin{aligned} \text{TPC} = & 180.83 + 36.66\text{A} - 1.60\text{B} - 2.08\text{C} - 1.26\text{AB} \\ & - 29.92\text{AC} + 1.35\text{BC} - 9.80\text{A}^2 - 14.37\text{B}^2 - 9.11\text{C}^2 \end{aligned} \quad (12)$$

$$\begin{aligned} \text{TFC} = & 9.90 + 2.24\text{A} - 0.048\text{B} - 0.14\text{C} - 0.26\text{AB} - 2.41\text{AC} \\ & - 7.500\text{E} - 0.003\text{BC} + 1.21\text{A}^2 - 2.18\text{B}^2 - 1.91\text{C}^2 \end{aligned} \quad (13)$$

where **A**, **B**, and **C** represent temperature, air velocity, and time, respectively.

Table 4 RSM-based BBD experimental data for refractance window dried ginger

S. No.	Input variables			Output responses		
	Temperature (°C) (A)	Air velocity (m/sec) (B)	Time (min) (C)	Antioxidant activity (%)	Total phenol content (mg GAE/g sample)	Total flavonoid content (mg quercetin/g sample)
1	80.00	0.50	135.00	57.35	160.64	5.91
2	80.00	1.00	300.00	51.93	156.75	5.69
3	80.00	0.75	217.50	52.10	181.45	9.93
4	95.00	0.75	300.00	50.21	164.70	8.89
5	65.00	0.50	217.50	31.68	120.21	6.55
6	95.00	1.00	217.50	61.23	190.58	10.80
7	65.00	0.75	300.00	38.74	151.56	9.16
8	80.00	0.50	300.00	49.12	157.18	5.72
9	80.00	0.75	217.50	49.01	178.91	9.96
10	65.00	0.75	135.00	27.51	99.29	4.69
11	95.00	0.75	135.00	75.23	232.13	14.05
12	65.00	1.00	217.50	30.78	119.47	6.89
13	80.00	0.75	217.50	50.45	182.12	9.8
14	80.00	1.00	135.00	49.56	154.81	5.91
15	95.00	0.50	217.50	58.59	196.38	11.49

3.9 *Optimality of Independent Variables*

Among all independent process variables, a qualitative investigation of the data that was obtained from the RWD-based drying of ginger was summarized. This confirmed the significance of temperature on the nutritional characteristics (Fig. 6). Higher temperatures were observed to better favor drying operations. An increase in temperature aided in the mass transfer of moisture from the ginger samples, and the optimum drying temperature also resulted in the highest values of total phenol content, antioxidant activity, and total flavonoid content. Among the process variables, drying time marginally influenced the measured responses. Since the reduced time of operation significantly contributes to a reduction in electricity consumption, minimization of drying time is a prime advantage of the RWD.

Using RSM-based optimization of drying parameters, the nutritional attributes of dried ginger, such as total AA percentage, TPC, and TFC, were optimized.

According to the summarized prior art in Tables 5 and 6, Thuwapanichayanan et al. [18] dried ginger slices in a cabinet dryer while varying temperatures in the range 60–80 °C at an air velocity of 0.3 m/s. Following this, the optimum antioxidant activity and total phenolic content of 90.59% and 22.73 (mg GAE/g dw), respectively, were obtained at a drying temperature of 70 °C and drying time of 190 min. Ghasemzadeh et al. [19] dried ginger rhizomes using a freeze dryer and obtained maximum values of antioxidant activity, TPC, and TFC as 58.22%, 13.5 mg gallic acid/g dw, and 230 mg GAE/g dw, respectively. Zdravko et al. (2016) vacuum-dried fresh red currants under variable parameters, such as temperatures ranging from 48 °C to 78 °C, pressures ranging from 30 to 330 mbar, and drying times ranging from 8 to 16 h, which were varied independently. BBD was used for the optimization of the drying parameters, through which the optimal process parameters were found to be a temperature of 70.2 °C and a drying time of 8 h. Liyana-Pathirana and Shahidi [20] optimized the extraction of nutritional components such as TPC from whole wheat and wheat bran using RSM. For Whole Grain Wheat, an optimal AA of 54% and TPC of 54.7 TE were obtained at drying parameters 61 °C and 64 min, while for wheat bran, an optimal AA of 49% and TPC of 61.3 TE were obtained at drying parameter values of 64 °C and 60 min.

In the present work, the optimal control variable values obtained for RWD process parameters temperature, time, and air velocity were 95 °C, 142 min, and 0.82 m/s, respectively. Corresponding optimal response variable values obtained were 71.96%, 226.84 mg GAE/g dw, and 13.82 mg quercetin/g dw for antioxidant activity, total phenol content, and total flavonoid content, respectively. The experimentally obtained response variable values for total AA percentage, TPC, and TFC correspond to $70.14 \pm 5\%$, 12.96 ± 2 mg GAE/g dw, and 218.64 ± 12 mg/g dw, respectively. Model fitness is critical, and it can be observed that the experimentally measured values are all in excellent accordance with the RSM-predicted values for the specified range of control variables.

Experimental values obtained at optimal drying parameters as dictated by the model in terms of temperature, air velocity, and time were acceptable for all cases

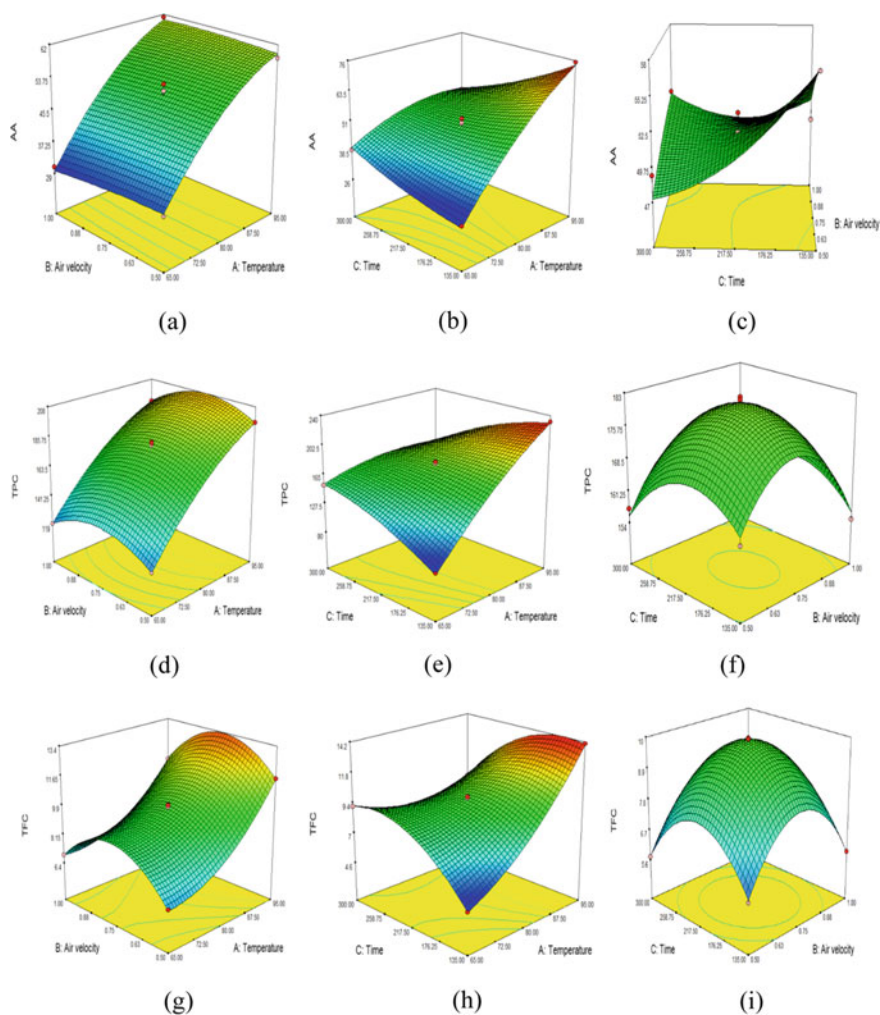


Fig. 6 Response surface plots for antioxidant activity (a–c), total phenol content (d–f), and total flavonoid content (g–i) during RWD of ginger

(standard error values were less than 5%), and hence the validation of the predicted models was concluded.

3.10 Relationship Between the Responses

The generated response surface plots for the independent and dependent variables have been shown in Fig. 6a–i. Except for the time–temperature and time–air velocity

Table 5 Comparison of trial and error-based best findings with those presented in the literature

S. No.	Sample	Drying time	Drying techniques	Temperature (°C)	Antioxidant activity (%)	Total phenol content	Total flavonoid content	Literature
1	Ginger slice	3.17 h	Cabinet dryer	70	90.59	22.73 mg GAE/g dw	–	Thuwapanichayanan et al. [18]
2	Ginger slice	–	Freeze dryer	–	58.22	13.5 mg gallic acid/g dw	4.21 mg quercetin/g dw	Ghasemzadeh et al. [19]
3	Ginger slice	1.15 h	RWD	95	70	230 mg GAE/g dw	18.2 mg quercetin/g dw	This work

Table 6 Comparison of RSM-based best findings with those presented in the literature

S. No.	Sample	Drying techniques	Optimal process parameters	Optimal response variables	Literature
1	Red currants	Vacuum drying; RSM-based optimization	Temp: 70.2 °C Time: 480 min	TPC: 477.34 mg GAE/100 g dw TFC: 306.34 mg CE/100 g dw	Zdravko et al. (2016)
2	Whole grain wheat	Air dried; RSM-based optimization	Temp: 61 °C Time: 64 min	AA: 54% TPC: 54.7 TE	Liyana-Pathirana and Shahidi [20]
	Wheat bran		Temperature: 64 °C Time: 60 min	AA: 49% TPC: 61.3 TE	
3	Ginger slice	RWD drying; RSM-based optimization	Temp: 95 °C Air velocity: 0.82 m/sec Time: 142 min	AA: 75.51% TPC: 232.42 mg GAE/g sample TFC: 14.05 mg QE/g sample	This work

combination, for all the other two variable combination graphs, the response surface plots followed a non-linear pattern. The influence of time was shown to be much higher, with a high F-value of 12.90 in the RWD instance, than the effects of temperature, which was seen to have an F-value of 1.88, and air velocity, with an F-value of 1.81.

Among all obtained responses, the best fit of the quadratic model was observed for total flavonoid content, which displayed the highest F-value of 1268.96 in comparison with the models' fitness for the other nutritional parameters—antioxidant activity and total phenol content (Table 4). Among all three response variables, temperature had the most dominant effect, in comparison with drying time and air velocity. The drying time had the modest effect on all three response variables. The lowest F-values obtained for both nutritional characteristics as well as associated parameters imply that the model and its parameters are both unfit.

The surface plots for antioxidant activity showed that with an increase in temperature and air velocity, antioxidant activity also increased (Fig. 6a–c). In terms of drying time, the surface plots affirmed an initial increase in the profile. Thereby, it can be inferred that an increase in the drying air velocity and temperature led to an increase in the antioxidant activity percentage (Fig. 6a). The temperature altered in the range 65–95 °C and the air velocity altered from 0.5 to 1 m/sec. The air velocity and time had both positive and negative effects on the percentage of antioxidant activity (Fig. 6a–c). The total phenolic content trends were similar to that of the antioxidant activity (Fig. 6d–f). Total flavonoid content trends were almost similar to total phenol content for drying time but not in the cases of temperature and air

velocity (Fig. 6g). For the effect of time on antioxidant activity, the antioxidant activity remained reasonably constant for both periods of increase and reduction in drying times (Fig. 6a, c). The surface plots of total phenolic content (Fig. 6d–f) revealed similar results. The amount of total phenol in the air increased for enhanced combinations of temperature and air velocity. These findings were consistent with the F-values listed in Table 4. Air velocity had no significant effect on the response variables when it came to temperature (Fig. 6e). TFC improved marginally as time and temperature were increased (Fig. 6g–i). For each control variable, the F-values in Table 4 also showed that nutritional features are more dependent on the variation of drying temperature and time than on air velocity.

3.11 Sensitivity of Parameters

The effect of drying process parameters such as temperature, air velocity, and drying time on nutritional characteristics, namely antioxidant activity percentage, total phenolic content, and total flavonoid content, has been discussed in Sects. 3.3–3.5. From the RSM plots of antioxidant activity (Fig. 6a–c), it is apparent that the effects of drying temperature and drying time are more magnified than the impact of drying air velocity. For the cases of total phenol content (Fig. 6d–e) and total flavonoid content (Fig. 6g–i), similar effects can be observed, which indicate greater sensitivity of the said responses on the drying time and temperature.

4 Conclusions

In this research article, the RWD-based drying study was conducted by following two approaches, namely the trial-and-error approach and response surface methodology-based optimization methods, in order to determine the optimal process parameter values. Furthermore, the sensitivity of the response variables to the drying process parameters has also been evaluated. During trial-and-error-based investigations, the temperature, sample thickness, and Mylar film thickness were chosen as process parameters, while antioxidant activity percentage, total phenolic content, and total flavonoid content were considered as the response variables.

The optimal combinations of temperature, sample thickness, and Mylar film thickness were found to be 95 °C, 1 mm, and 10 mil, respectively, in the trial-and-error-based investigations. The corresponding optimal values of antioxidant activity percentage, total phenolic content, and total flavonoid content were determined to be 72%, 230 mg GAE/g dry sample, and 10 mg quercetin/g dry sample, respectively. For the RSM-based optimization, air velocity, temperature, and drying time were chosen as the process parameters for the response variables, namely total antioxidant activity percentage, total phenolic content, and total flavonoid content. Following the RSM optimization, the optimal combinations of temperature, drying time, and air velocity

were found to be 95 °C, 142 min, and 0.82 m/s, respectively. The corresponding best values of antioxidant activity percentage, total phenolic content, and total flavonoid content were observed to be 71.96%, 226.84 mg GAE/g dry sample, and 13.82 mg quercetin/g dry sample, respectively. The RSM model indicated quadratic dependence of total phenol content, antioxidant activity, and total flavonoid content on drying time, drying temperature, and air velocity.

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Techno-economic Efficacy of Refractance Window Dried *Curcuma Longa*



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Abstract In this article, the drying characteristics of slice and paste *Curcuma longa* has been addressed using three alternate drying methods namely refractance window drying (RWD), oven drying and tray drying. A comparative assessment of the alternate drying was targeted in terms of efficacy associated to desired characteristics such as moisture content, antioxidant activity, curcumin content, total phenolic content, and total flavonoid content. Among all cases, slice samples obtained from the RWD at 60 °C process have been analyzed to possess optimal combinations of all evaluated parameters along with reduction in drying time. Finally, a comparative conceptual economic assessment of laboratory scale RWD, tray and oven drying processes has also been targeted in the article. Primarily, the efficacy of RWD has been characterized due to its process simplicity and reduction in drying time to achieve similar nutritional characteristics as those achieved using tray and oven drying processes.

Keywords Refractance window drying · Turmeric · Slice · Paste

Abbreviations

RWD Refractance window drying

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DPPH	2,2-Diphenyl-1-Picrylhydrazyl
AOAC	Association of official analytical chemists
MC	Moisture content
AA	Antioxidant activity
UV-VIS	Ultraviolet-Visible
TPC	Total phenolic content
TFC	Total flavonoid content
CC	Curcumin content
ANOVA	Analysis of variance
EMI	Equal monthly instalments
O	Oven
T	Tray
OS	Oven dried slice
OP	Oven dried paste
TS	Tray dried slice
TP	Tray dried paste
R60S	Refractance window drying at 60 °C for slice
R60P	Refractance window drying at 60 °C for paste
wb	Wet basis
GAE	Gallic acid equivalent

1 Introduction

Advances in applied science and technology coupled with rapid population growth, abundance of farm produce and agro-industrial growth have together enhanced food processing and preservation research. In addition, due to rapid advances in fundamentals associated with biochemistry and nutrition, there is a growing awareness to supplement human food consumption through balanced nutrition of proteins, carbohydrates, vitamins, and minerals. Food processing research primarily targets the retention of nutritional parameters for a marginal increase in processing cost [1].

Preliminary food processing methodology primarily targets shelf life enhancement through the reduction of water activity and moisture content and retention of proteins, vitamins, carbohydrates etc. To do so, some of the popular methods adopted are drying, freezing, refrigeration, pasteurization, canning, pickling, salting, sugaring, vacuum packs, and irradiation. Among these, drying is a simpler process to enhance the shelf life of fruits and vegetables. Compared to fresh vegetables stored under room temperature, dried vegetable products can be stored without much decrease in quality and nutrients for a long time. Other advantages of the drying process are easy product storage, stability of volatile and aromatic compounds, protection against oxidative and enzymatic degradation, weight and volume reduction of the product, availability of the product throughout all seasons, ease of transportation due to the reduction in storage and shipping cost. Further, another promising

feature of the drying process is its ability to produce organic products that are bereft of preservatives and other additives that may detriment and complicate nutritional processes in human beings [2, 3].

The drying process strongly influences mass and heat transfer rates of processed foods and these are complex functions of the composition, structure, shape and size of the samples. Drying methods to process foods include hot-air oven, convective, tray, freeze, spray, vacuum, microwave and fluidized bed drying methods. Among these, due to longer drying period and low drying rates, hot-air oven drying is not highly promising to retain taste, color and nutritional content of the product [4]. On the other hand, tray drying is economical and commonly deployed but suffers from the limitations of product quality. Application of such tray drying is apparent for various leafy and non-leafy vegetables [5]. From processing time perspective, tray drying requires about 2.5–5.5 h of high temperature (70–50 °C) operation, which increases to about 18–24 h for freeze drying at a reduced temperature of 20 °C [6]. From the cost perspective, it is well known that vacuum [7] and freeze [8] drying are significantly expensive methods and are not recommended towards scale-up assisted marginal enhancement in process cost to achieve high volume dried products.

To overcome the limitations of the mentioned drying processes such as high processing time, higher cost, scalability and retention of nutritional parameters, a novel drying method such as refractance window drying (RWD) can be targeted. During RWD, a plastic interface (Mylar film) transfers heat energy from the hot liquid media to a sample placed on the film and thereby all heat transfer modes (radiation, conduction, and convection) are existent. These facilitate intense evaporation of dried sample moisture in a short span of time. In due course of RWD, while the hot water is at 90–95 °C, the sample is effectively dried below a temperature of 60–70 °C. Thus, with all three heat transport modes, the drying time can be reduced substantially to about 45–120 min. Better nutritional parameters could be achieved for the processed foods due to shorter drying and hence effective processing at a significantly lower temperature [6, 8–11]. RWD of saffron petals and its stigma was carried at 25, 60, 70 and 80 °C [12].

Water diffusivity of RWD processed sliced mango samples was determined for 1 and 2 mm thickness [13]. These values have been obtained as 4.40×10^{-10} and 1.56×10^{-10} m²/s respectively which are significantly better than the corresponding values evaluated for hot air dried samples (2.08×10^{-11} and 6.83×10^{-11} m²/s respectively). In another study, the powder of RWD processed haskap berry has been analyzed to retain 93.8% of anthocyanin with respect to original frozen berry samples [14]. Similarly, another prior art [4] indicated that the RWD processed loquat slices at 90 and 80 °C exhibited the highest phenolic content of 328.24 and 323.67 mg malic acid/100 g in comparison with the fresh slices (302.74 mg malic acid/100 g).

On the other hand, limited investigations have been conducted with respect to RWD of vegetable resources and their comparative assessment with other conventional processes such as tray drying. The retention value of phenolic compounds was high and without substantial variation ($p > 0.05$) with respect to the control carrot slice samples of 0.2 and 0.4 mm [15]. 3 mm carrot slice could be effectively dried below 10% db moisture content in 200 min using RWD [16]. For 2 mm tomato slices

[17], the RWD process provided higher resemblances of nutritional characteristics with respect to those evaluated for the fresh sample. The RWD process effectively reduced the moisture content of red onion slices of 1 mm from 7.19 to 0.2 kg water/kg dry solids in a short span of 40 min [18]. Compared to tray drying [19], significant reduction in beta-carotene loss (9.9%) as opposed to 57% for carrot puree samples was achieved with the RWD process. Similarly, another prior art [6] concluded that the RWD reduced the moisture content of pureed asparagus from 21 to 0.04% db in only 4.5 min. On the other hand, a research group [20] confirmed significant microbial reductions of 4.6- to 5.5-log for total counts (aerobic), coliforms, *E. coli*, and *L. innocua*, respectively for pumpkin puree samples using RWD process. In another study [21], the authors considered similar quantities of processed materials and indicated that the RWD based drying of liquid strawberry (strawberry juices) samples involved a reduced time span of 0.77–0.90 h, which was significantly high (5 h) for a conventional hot air dryer operated at 60 °C. Also, a research group [20] indicated drying of pureed pumpkin reduced moisture content from 80 to 5% wb for a reduced time span of 5 min. Further, the quality of the RWD processed product in terms of nutritional and sensory attributes is comparable to that obtained by freeze-drying but with a significant reduction in equipment cost. In summary, RWD can be inferred to enhance reduced loss of desired vegetable product characteristics such as AA, TFC, TPC, curcumin content, carotene content, ascorbic acid, etc. The RWD of *Curcuma longa* (turmeric) have not been targeted. However, few studies targeted oven drying of the turmeric. Among unblanched and blanched turmeric slices that were subjected to hot air drying at 55 °C, the relevant prior art [22] inferred the superior quality of unblanched oven dried samples. Similarly, another article [23] inferred drying process optimality in terms of prior boiling for 1 h followed with drying at 70 °C for 21 h to achieve rhizomes with a maximum curcumin content of 2.97%.

A critical analysis of the above cited literature affirms that mostly fruits such as strawberry, mango, papaya, blueberry, pomegranate, acai; vegetables such as pumpkin, red onion, asparagus, carrot, tomato; spices such as paprika and red peppers; tubers such as potatoes, cassava and yams have been studied using the RWD process. In this regard, it is important to note that both ginger and turmeric have applications in food processing and functional food applications. While their RWD drying characteristics may be similar to that of tubers such as cassava and yams, their nutritional characteristics could be significantly different and thus RWD processed ginger and turmeric may be further effective towards deployment in novel functional food formulations and products. Also, a comparative assessment of the RWD process in terms of its conceptual cost competitiveness with other horticultural produce drying methods has not been addressed till date. Such investigations would be useful to further enhance the applicability of the novel refractance window drying process in the food process industries.

Considering all indicated gaps in the reported prior art, this article devotes to the evaluation of RWD process characteristics of sliced turmeric samples. Both paste and slice samples have been considered along with comparative assessment with oven and tray dried samples. Finally, the cost efficacy of the RWD process in comparison with oven and tray drying methods have been addressed to indicate upon its economic

competence and effectiveness. The following section outlines upon the methodology along with materials.

2 Materials and Methods

2.1 Raw Materials

Turmeric was procured from market complex, Indian Institute of Technology Guwahati, Kamrup, Assam, India and was packed in polythene pouches to prevent contamination while transporting the sample.

2.2 Chemicals

Sodium carbonate, aluminium chloride, potassium acetate, gallic acid and absolute methanol were obtained from Merck India. Folin-Ciocalteu reagent, extrapure quercetin and 2,2-Diphenyl-1-Picrylhydrazyl (DPPH), were bought from SRL Pvt. Ltd., India. Ethanol was bought from MTedia and Curcumin (99%) was bought from Asper in India.

2.3 Sample Preparation

Firstly, running water based washing was conducted for the raw turmeric samples to get rid of dirt and other contaminants. Thereafter, the washed turmeric was thoroughly wiped to eliminate water adhering to the sample and peeled. The turmeric thereafter, was sliced into 1 mm thick slices using a slicer (Model: VDNSI) for drying experiments using oven dryer, tray dryer and RWD system. To prepare the sample paste, the cut samples were ground in a grinder (Philips 500 W grinder) to achieve a thick paste. The sample paste of 1 mm thickness was deployed for drying investigations.

2.4 Oven, Tray and Refractance Window Drying

Laboratory scale hot air oven (Make: REICO) and tray drier (Make: International commercial traders, Kolkata, India) were deployed to dry 1 mm slices and 1 mm thick paste at 60 °C. For RWD, a water bath system (Make: Jain Scientific Glass Work) and a Mylar film (Make: Lamtex Solution) of 0.25 mm thickness were used and the mylar

film was floating on the water bath surface. Thereafter, RWD drying was conducted by placing a layer of slice and paste on the film at 60 °C. The obtained dried samples from various drying equipments were thoroughly grounded to a powder form to determine the nutritional parameters.

2.5 Analysis of Nutritional Parameters

Moisture content: The literature reported approach [24] was utilized to determine the sample moisture content (MC). The procedure involves drying a known quantity of the sample in an oven at 105 °C for 14 h. Thereafter, the sample was managed to keep in a desiccator and weighed. Based on the initial and final weights of the sample, the moisture content of the dried turmeric slice/paste samples can be determined using the expression:

$$\text{MC(\%)} = \frac{W_1 - W_2}{W_1} \times 100 \quad (1)$$

where W_1 and W_2 are weight of known quantity of sample and dried sample respectively.

Antioxidant Activity (AA): The literature reported procedure that highlights upon the 2,2-diphenyl-1-picrylhydrazyl (DPPH) assay method [25, 26] was meticulously followed to quantify the AA of fresh and dried turmeric samples. The procedure is as follows. As a first step, 10 mg sample and 20 mL absolute methanol were mixed and thereafter subjected to ultrasound for 30 min in a sonicator (Make: Elma, Model: Elmasonic S 30 H). Thereby, filter paper (Whatman No. 1) was used to filter the mixture and obtain its extract. The following step involved the addition of 3 mL 0.002% methanolic DPPH solution to 1 mL filtrate. During this step, a control sample was also duly prepared by mixing 1 mL absolute methanol with 3 mL 0.002% methanolic DPPH solution. The third step involved thorough shaking of both samples and subsequent incubation in a dark environment for 30 min. Thereafter, the fourth and final step involved measurement of absorbance at a wavelength of 517 nm and with a UV–Visible spectrophotometer (Make: UV-2800, Shimadzu). Based on the absorbance detected, AA of the turmeric slice sample can be calculated utilizing the following equation:

$$\%AA = \frac{A_1 - A_2}{A_1} \times 100 \quad (2)$$

where A_1 and A_2 are absorbance values of the control and sample, correspondingly.

Total Phenolic Content (TPC): The sample TPC was obtained using Folin-Ciocalteu reagent. The working principle associated to the method refers to a color transformation of the sample system from yellow to blue-black color. The color

change mechanism was associated to the reduction of tungstate-molybdate prevalent in the said reagent with the phenolic compounds of the sample [25]. Typically, TPC are expressed in terms of gallic acid equivalent. Therefore, a calibration chart is prepared by following the standard procedure, which has been outlined as follows. Gallic acid stock solution was first prepared by mixing 0.5 g of dry gallic acid, 10 mL of 50% methanol and water in a 100 mL volumetric flask. Thereafter, upto 500 mg/L gallic acid solutions have been prepared by appropriate dilution of upto 5 mL of the stock solution. Subsequently, 0.1 mL was mixed with 6 mL distilled water and 0.5 mL of the said reagent. After mixing for about 5 min, the mixture was added with 1.5 mL of 20% sodium carbonate solution and was diluted to reach 10 mL volume. Eventually, the standard gallic acid solutions were subjected to absorbance measurements at 765 nm wavelength using UV–Visible spectrophotometer. The absorbance of the fresh and dried sample extracts was as well conducted to determine their TPC characteristics. The extraction procedure involved blending of 0.5 g of the sample with 10 mL of 50% methanol and subsequent filtration using a filter paper. Using absorbance values of the sample extracts and calibration chart, the TPC has been evaluated in terms of mg gallic acid equivalent per gram of the dry sample weight.

Total Flavonoid Content (TFC): Aluminium chloride colorimetric method [27] was adopted to reflect the TFC of various samples. The procedure involved preparing yellow colored processed solutions that indicated the presence of flavonoids.

A standard curve was obtained using quercetin. To do so, firstly, 100 mg quercetin was mixed in 100 mL of 80% ethanol. To prepare quercetin stock solution, 1–5 mL of the solution was taken out using a pipette and diluted with distilled water to obtain standard quercetin solutions of 100–500 mg/L. Thereafter, 0.5 mL of the stock solution was mixed with 1.5 mL of absolute methanol, 0.1 mL of 10% aluminium chloride, 0.1 mL of 1 M potassium acetate aqueous and 2.8 mL of distilled water. The final solution after thorough mixing was placed at 27 °C for 30 min. After this step, the absorbance of the standard solution was measured at a wavelength of 415 nm using a UV–Visible spectrophotometer. Similar procedures were followed for fresh and dried turmeric samples after carrying out the extraction process. This involved a thorough mixing of 1 mL of 80% ethanol with 10 mg of the turmeric sample using a vortex mixer followed by the addition of 9 mL of 80% ethanol. Such solution was sonicated to achieve a sample concentration of 1 mg/mL i.e. 1000 ppm. Thereafter, the solution was filtered using a filter paper. The extract solution eventually was subjected to procedures similar to those mentioned earlier for standard quercetin solutions. Using the measured absorbances of the standard and dried turmeric samples, the TFC of the dried sample was estimated using the calibration curve in terms of mg quercetin/g dry weight.

Curcumin Content (CC): The CC of the fresh and dried samples was determined by adopting the approach summarized in a research article [28]. This involved the preparation of a standard or calibration curve using curcumin. To do so, 10 mg of the curcumin standard was mixed in 10 mL of 95% methanol and was subjected to sonication for 10 min. Following this, 10 mL of the obtained solution was mixed with 90 mL methanol (95%) to achieve a standard solution of 100 mg/L. Thereafter, 0.5–5 mL of standard solutions were taken out using a pipette and mixed with methanol

(95%) to obtain 0.5–5 mg/L standard solutions. The absorbance of these samples was measured at a wavelength of 425 nm with the UV–Visible spectrophotometer.

Similar procedures were followed for fresh and dried turmeric samples after carrying out the extraction process. This involved a thorough mixing of 5 mL of 95% methanol with fresh/dried turmeric (10 mg) sample and sonication for 10 min. Eventually, using a volumetric flask (10 mL) and 95% methanol, 5 mL of the extract volume was made up to 10 mL. Thereafter, the solution was filtered using Whatman filter paper No. 1. 0.4 mL of the filtrate was then added to 5 mL of 95% methanol. The extract solution eventually was subjected to procedures similar to those mentioned earlier for standard curcumin solutions. Using the measured absorbances of the standard and turmeric processed samples, the curcumin content of the fresh/dried sample was determined using a calibration curve in terms of % curcumin content w/w.

2.6 Statistical Analysis

One-way ANOVA methodology has been applied for the evaluation of the variations in the analyzed parameters. Based on the analysis of variance approach, the methodology involved the differences among the mean values and significances within independent variables [29]. IBM SPSS statistics software was deployed to conduct one-way ANOVA analysis and thereby instil confidence in the trends associated with various responses.

2.7 Cost Efficacy

Considering operating, equipment, maintenance, depreciation and manpower costs, the cost efficacy studies targeted the annualized total processing costs associated to sliced and paste turmeric samples processed using oven, tray and refractance window drying processes. The total annualized cost was evaluated as the sum of electricity, equipment, manpower, maintenance and depreciation costs. Cost evaluations have been supplemented with other auxiliary parameters such as power (kW), power tariff, time taken for 1 batch (h), cost of electricity per batch, batch per day, no. of days per year, cost of electricity per year, amount of sample per batch, no. of days per year, amount of sample per year (kg). Using these and other cost parameters, electricity costs associated to drying process per one kg of fresh and dried samples were determined. Similar calculations have been considered for the electrical costs associated to the grinding operation. The equipment costs for RWD, tray drying, oven drying, slicer and grinder have been determined by considering factors such as equipment purchase cost, life span, personal interest loan for year, and equal monthly instalments (EMI) per year. The equipment maintenance costs were evaluated to be about 10% of overall equipment cost while depreciation was calculated to be 10% of the process cost. The manpower costs were evaluated based on the number of persons, salary for

each personnel and overall salary per year for the appointed manpower of a small batch process. Thereby, annual production costs have been determined. The reported cost efficacy in this article could serve as a reference or guideline for the commercial turmeric drying processes on a laboratory or pilot scale systems [30]. Supplementary material presents a brief account of the cost efficacy related calculations and relevant data associated to technical and cost parameters. The methodology followed for the economic evaluation has been as per the procedure outlined in the relevant literature [31]. Accordingly, normalized equipment, maintenance and depreciation costs have been determined for all processes by considering uniform processing of about 1090 kg of turmeric on an annual basis. Accordingly, Guthrie correlation exponent factors have been taken as 0.65 and 0.25 for equipment and manpower costs respectively.

Various economic indices such as annualized processing cost per kg fresh and dry sample and % contribution of various costs (electricity, manpower, equipment, maintenance and depreciation costs) have been considered to gain useful insights into the conceptual processing cost of alternate drying processes (RWD, oven and tray drying). Both slice and paste samples have been considered to gain useful insights.

3 Results and Discussion

3.1 Drying Time

The operating temperature of 60 °C for oven (O) and tray (T) drying method of 1 mm slice and paste have been based on available literature for oven and tray drying of turmeric slice samples. The drying time for oven and tray drying for both 1 mm slice (OS and TS) and 1 mm paste (OP and TP) was obtained from drying kinetic studies. The RWD time at 60 °C for both slice (R60S) and paste (R60P) were estimated from drying kinetics. These variations in temperature and sample thickness have also been ensured through preliminary experiments at the chosen combinations of drying time and temperature to ensure achievement of good combinations of nutritional parameters for all cases. Among oven and tray drying, time taken to dry 1 mm slices and 1 mm thick paste at 60 °C was 8 h and 4.5 h respectively. Similar experiments were conducted to dry 1 mm slices and 1 mm thick paste at 60 °C for 7 h and 3 h respectively in RWD.

3.2 Moisture Content

Table 1 summarizes the moisture content (MC) of slice and paste turmeric samples obtained after oven, tray and RW drying. For fresh turmeric, the moisture content has been evaluated to be significantly high and about 87–88% wb, thus affirming the

Table 1 A summary of moisture content of *Curcuma longa* dried samples obtained with alternate drying methods

Drying methods	Moisture content after drying (% wb)	Standard deviation
Oven slice	3.8	±0.1
Oven paste	2.0	±0.2
Tray slice	2.9	±0.3
Tray paste	1.2	±0.3
RWD 60 °C slice	4.2	±0.2
RWD 60 °C paste	3.7	±0.2

need for a significant reduction in short time duration. It can be seen from the table that the MC varied from 2.9–4.2% wb and 1.2–3.7% wb for 1 mm slice and paste cases respectively for all drying methods.

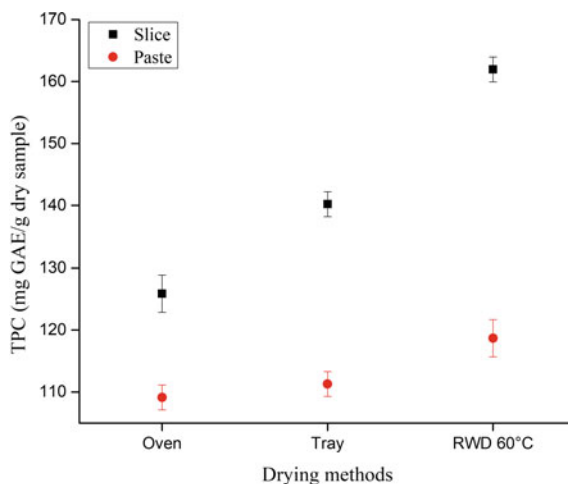
For both slice and paste samples, the lowest (2.9 and 1.2% wb) and highest (4.2 and 3.7% wb) MCs were obtained with tray drying and RWD at 60 °C respectively. Compared to tray and oven drying, a significantly lower drying time was required for RWD to achieve an MC value lower than 8% wb to prevent microbial growth. This is due to effective heat transfer for the RWD case that was facilitated by the Mylar film as a contact area between the slice/paste samples and hot water bath. In other words, besides convection, conduction played a dominant role to effectively reduce MC in the sample. A relevant article [15] reported similar trends for carrot drying. For several cases, MC for paste was significantly low in comparison with the slice samples. This is due to the disruption of the cellular structure of turmeric during paste preparation that releases bound MC for its effective removal.

3.3 Total Phenolic Content

Figure 1 depicts the total phenolic content (TPC) of turmeric slice and paste samples for various cases. The TPC of fresh turmeric is 81.55 mg GAE/g fresh sample. The TPC enhanced to 125.86, 140.21, and 161.95 mg GAE/g dry sample for slice and 109.13, 111.30, and 118.69 mg GAE/g dry sample for paste samples obtained with oven at 60 °C, tray at 60 °C, and RWD at 60 °C respectively. This enhancement in TPC for all dried samples is due to the significant reduction in MC of the dried samples. Highest and lowest TPC was obtained for the slice and paste samples prepared with RWD at 60 °C and oven at 60 °C respectively.

For all cases, TPC in paste was significantly lower than those obtained for slice samples. This is due to breakage of cellular structure that enhances the release of phenolic compounds and subsequent oxidation for the paste case. Among all cases, RWD indicated the highest TPC values and this is due to significantly lower drying time. The significantly faster MC loss during RWD is hypothesized to reduce oxygen partial pressure at the dried sample air interface due to the higher vapour pressure of the evaporated water [19]. Thereby, lower oxidation would impede reduction of

Fig. 1 Total phenolic content (TPC) characteristics of alternate drying processes and turmeric slice and paste samples



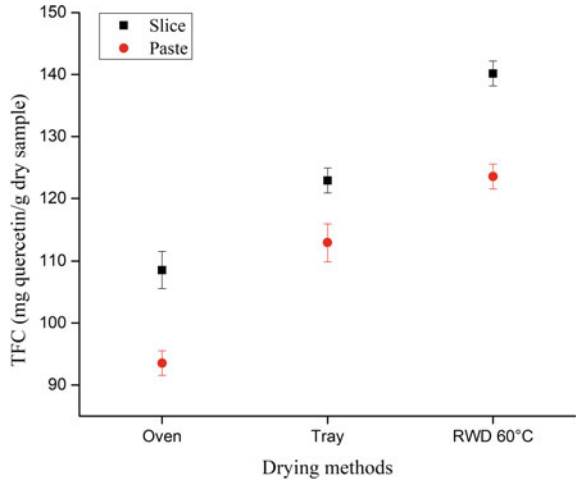
TPC [15]. Further, it can be analysed that RWD at 60 °C is the best to effect higher TPC retention and this is due to faster removal of the MC.

3.4 Total Flavonoids Content

Figure 2 illustrates the total flavonoids content (TFC) of turmeric paste and slice samples obtained after drying with various methods. For fresh sample, the average TFC has been evaluated as 17.41 mg quercetin/g fresh sample. However, for the dried sample, the TFC can be analyzed to increase significantly to 108.54, 122.90, and 140.16 mg quercetin/g dry sample for slice and 93.54, 112.90, and 123.54 mg quercetin/g dry sample for paste samples obtained with oven at 60 °C, tray at 60 °C, and RWD at 60 °C respectively. The enhancement in TFC after drying is due to a significant reduction in moisture content.

For all cases, slice samples possessed higher TFC than corresponding values obtained for paste samples. This is due to better cellular integrity in slice samples that do not favor the oxidation of hidden TFC in the cell structure [15]. The RWD at 60 °C reported highest TFC values for both slice and paste samples. This is due to the fast drying rate in short duration that detracts TFC oxidation and enhances the distribution of flavonoids from the sliced samples [32]. Compared to oven and tray drying, it can be analyzed that significant TFC retention can be achieved through RWD and this conveys its promising performance.

Fig. 2 Total flavonoids content (TFC) of turmeric slice and paste samples and alternate drying methods

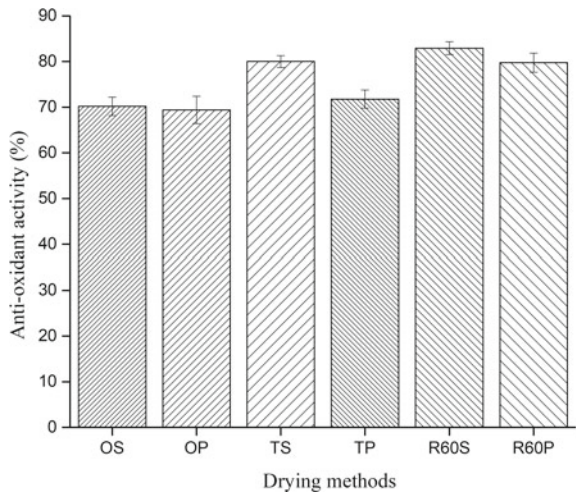


3.5 Antioxidant Activity

Antioxidant activity (AA) is an important parameter and its retention in significant quantities is required for functional food formulation. Figure 3 depicts the variations of AA for various drying methods. As shown, the AA varied as 70.17, 80.00, and 82.93% for slice and 69.38, 71.76, and 79.75% for paste samples obtained with oven at 60 °C, tray at 60 °C, and RWD at 60 °C processes respectively.

For fresh turmeric, the average AA value is 27.27%. Compared to the fresh sample, such an enhancement in AA is due to significant loss of moisture content due to drying. Among all cases, the lowest and highest AA have been obtained for

Fig. 3 Antioxidant activity (AA) of tray, oven and refractance window dried turmeric slice and paste samples



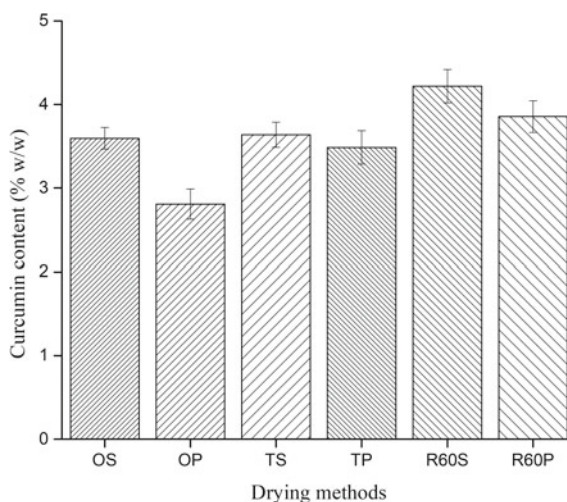
samples obtained with oven at 60 °C, and RWD at 60 °C respectively. Compared to paste samples, slice samples had marginally higher AA values and this is due to cellular disruption during grinding that enhances the surface area and reduces the retention of heat sensitive AA compounds. It is well known that prolonged heating at higher temperature allows oxidation of AA compounds. However, since RWD facilitates relatively lower temperatures and significantly shorter drying times, the RWD provides the highest AA values. These in turn effectively reduces oxidation of phenolic antioxidants [15]. Also, the retention of TFC and TPC enabled higher retention of the AA content of dried turmeric after RWD [27].

3.6 Curcumin Content

With its lipophilic nature, curcumin is a polyphenolic compound that imparts an yellow color to the turmeric. Therefore, its retention is required from a textural perspective as well as polyphenolic properties. For fresh samples, the CC is 0.73% CC w/w. This is due to the high moisture content of the sample. Figure 4 shows significantly higher values of 3.59, 3.63, and 4.21% CC w/w for slice and 2.80, 3.48, and 3.85% CC w/w for paste samples obtained with oven at 60 °C, tray at 60 °C, and RWD at 60 °C respectively. Among all samples, RWD at 60 °C with slices indicated the highest value of CC of 4.21% w/w. Compared to oven drying, tray drying and RWD, RWD was able to retain high CC.

Compared to the quantitative differences in the retention of TPC, TFC and AA in RWD with respect to tray/oven drying, the quantitative difference in the retention of curcumin is significantly low. This is due to the fact that the concentration of curcumin was not significantly influenced with heat treatment [33].

Fig. 4 Curcumin content characteristics of tray, oven and refractance window dried turmeric slice and paste samples



3.7 Statistical Analysis

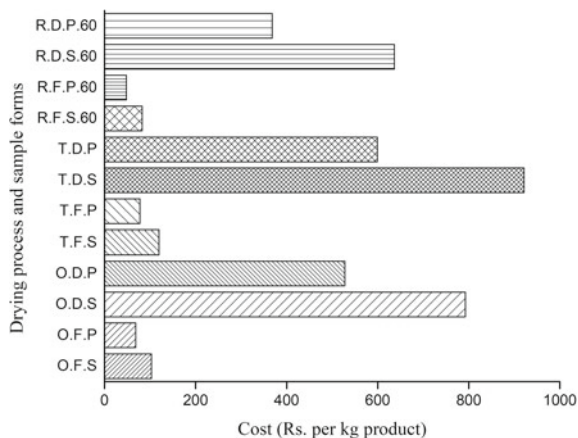
The one-way ANOVA affirms that beyond 95% confidence interval level, no statistical differences can be seen for all variables. F-values obtained for data associated to moisture content, AA, TPC, TFC and CC were 628.35, 132.29, 655.15, 926.53 and 67.49 respectively. To do so, each dried sample was subjected to a minimum of three trails for each data point all cases. Also, it has been analysed that for all samples, high F-values and low p-values (<0.0001) have been obtained. These findings affirm upon the greater significance of the conducted experiments.

3.8 Comparative Economic Competence

Figure 5 depicts the processing cost of drying per kg fresh sample and processing cost of drying per kg dried sample for all drying cases. As illustrated, among all processes, the lowest annualized processing cost was obtained for the RWD process operated at 60 °C. For such system, the processing cost was about Rs. 82.61 and Rs. 47.75 per kg of fresh sample for slice and paste cases respectively and Rs. 636.59 and Rs. 367.97 per kg of the dried sample product for slice and paste cases respectively. Following this, the oven annualized processing costs are significantly higher and these refer to Rs. 102.84 and Rs. 68.48 of fresh sample for slice and paste cases respectively and Rs. 792.47 and Rs. 527.73 per kg of dried sample product for slice and paste cases respectively. For the tray drier, the annualized processing costs are the highest and correspond to Rs. 119.58 and Rs. 77.76 per kg fresh sample for slice and paste cases respectively and Rs. 921.45 and Rs. 599.24 per kg dried sample for slice and paste cases respectively. The obtained costs for all processes are significantly higher in comparison with the actual cost of the turmeric sample (about Rs. 120–200/- per kg for fresh sample). However, it shall be observed here that the costs indicated in this work are conceptual and for laboratory scale systems. With process scale up, the costs can reduce significantly. However, the cost competence is very likely to remain as it is for the RWD process due to lower electricity costs achieved due to lesser processing time and simpler process equipment in comparison with the tray and oven drying processes.

The contribution of various costs in alternate drying process systems has been depicted in the bar diagrams (kindly refer to Fig. S1 in supplement). As affirmed, for the RWD system, the cost contributions of electricity are lower for the best choice of the RWD process (61.43 and 45.54% at 60 °C) in comparison to those obtained for the oven drying (67.67 and 57.16%) and tray drying (72.75 and 62.92%) processes for the slice and paste samples respectively. Corresponding equipment costs are about 3.98 and 4.42% in the RWD process at 60 °C, 4.30 and 4.99% in the oven drying processes and 3.24 and 3.85% in tray drying process for both slice and paste samples respectively. Thus, manpower costs and electricity costs dominate the costs of all processes in comparison with the equipment cost.

Fig. 5 Annualized total cost (Rs. per kg of dried sample) of laboratory scale alternate turmeric drying processes (Notation: R = RWD, T = Tray dryer, O = Oven dryer, D = dried, F = Fresh, S = slice and P = paste)



3.9 Literature Comparison

Table 2 summarizes the best findings of this work with those available in the literature. The available literature does not elaborate upon the slice and paste sample drying using RWD, oven and tray drying processes. The best findings only correspond to those reported in the literature as far as comparative assessment is concerned. Thereby, they clearly indicate that the RWD has the edge due to lower processing time and associated costs for the slice and paste turmeric sample drying to reach the lowest moisture content of 4%. Also, only curcumin content and moisture content estimation were carried out in most of the literature, analytical characterizations like AA, TFC and TPC have not been reported in all critical literature findings. Thus, the findings of this work are novel and are expected to serve as a useful guideline to further the commercial application of RWD process for the drying of horticultural and agro-produces.

4 Conclusions

This article addressed the comparative assessment of tray, oven and RW drying of *Curcuma longa*, and thereby indicated prominent influences. Firstly, RWD proved to be significantly effective to provide better nutritional parameters in a time span of 7 h, as opposed to 8 h for the conventional drying process. Secondly, among all cases, the optimal process and sample combinations are RWD and turmeric slices, which upon RWD at 60 °C provided 4.2% wb, 161.95 mg GAE/g dry sample, 140.16 mg quercetin/g dry sample, 82.93%, 4.21% w/w for moisture content, TPC, TFC, AA and CC respectively for *Curcuma longa*. Thirdly, among paste and slice samples, RWD provided higher retention for both paste and slice in comparison to the oven and tray drying process and was the most cost effective process. Fourthly, further

Table 2 A summary of optimal tray, oven and refractance window drying process-product characteristics

Drying method	Sample constitution	T (°C)	Time (h)	TFC (mg quercetin/g dry sample)	TPC (mg GAE/g dry sample)	AA (%)	CUR (%w/w)	MC (%)	References
RWD	1 mm slice	60	7	140.1	161.9	82.9	4.2	4.2	This work
Tray	1 mm slice	60	8	122.9	140.2	80	3.6	2.9	
RWD	1 mm paste	60	3	123.5	118.6	79.7	3.8	3.7	
Tray	1 mm paste	60	4.5	112.9	111.3	71.7	3.4	1.2	
Oven	Rhizome	70	21	–	–	–	2.97	–	[22]
Oven	Rhizome	55	5	–	–	–	4.6	8	[21]

investigations are required to evaluate the optimality of sample thickness, drying time and temperature in the range of 0.5–2 mm, 0.30–2 h and 60–95 °C respectively. Finally, the conceptual annualized laboratory scale processing cost of the RWD process (operated at 60 °C) was the lowest among RWD, tray and oven drying processes. This corresponds to Rs. 47.75 and Rs. 367.97 per kg of fresh and dried paste sample respectively. Contrary to this, higher processing costs were obtained for tray and oven drying processes. This is due to greater processing time of these systems in comparison with the RWD process system. In summary, promising drying characteristics have been indicated by RWD for the achievement of dried *Curcuma longa* with a better combination of nutrition and other parameters. These findings are expected to be effective to further functional food development with RWD processed *Curcuma longa*.

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3E (Energy, Exergy and Economic) Analyses and Kinetic Studies on Microwave Drying of Star Fruit



Sayan Mandal , Koushik Jena , and V. Prabu 

Abstract Star fruit (*Averrhoa carambola*) is beneficial for humans as it is enriched in natural antioxidants. However, due to its seasonal availability, drying of star fruit is essential for storage purposes. This study is focussed on microwave drying of star fruit such as estimation of kinetic parameters, effective moisture diffusivity, and activation energy and model predictions by standard thin layer models. Further, a 3E analysis (energy, exergy and economic) is performed to examine the feasibility of commercial implementation. The outcomes revealed, that the drying time has remarkably reduced by 11–16 times and the moisture diffusivity has raised by 50–250 times in contrast with convectional drying method. Energy efficiency is found to be 16.74% at 150 W power level, which is further raised to 26% at 525 W power level. The exergy efficiency of the microwave drier is found to be 3.93% at 150 W, which is further raised to 11.99% at 525 W microwave power level. From the economic analysis of microwave drier, it is found that the cost of microwave drying reduced below 1 INR at every microwave power level with enhanced capacity of the plant.

Keyword Activation energy · Effective moisture diffusivity · Energy and exergy analyses · Microwave drying · Star fruit

1 Introduction

Star fruit scientifically known as *Averrhoa carambola*, is mainly found in Southeast Asia, Brazil, Micronesia, the United States, parts of East Asia, etc. [1]. In India, star fruit trees are grown in the southern states and on the west coast, from Kerala to West

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Bengal [2]. The star fruit has a mixed taste of sweet and sour and it can be utilized for making chutney, pastry and curry [3]. Due to the presence of carotenoids, star fruits possess natural antioxidant properties and help to treat various diseases like nausea, mouth ulcers, diarrhoea, toothache, ascites, etc. [4]. Star fruit has the property to alleviate pain from bleeding haemorrhoids [5]. As the star fruit is seasonal in nature, its preservation is highly essential. One of the primordial and classical preservation techniques employed in cultivated processing field is drying.

The microwave method has several benefits including low drying time, enhanced product quality, and improved energy efficiency, in comparison to other processes [6]. As microwave drying considerably decreases the processing period, it is an appealing source for saving thermal energy [7]. The traditional technique used for drying of foodstuffs is convective drying [8]. Food colours, flavour and nutritional alteration, shrinkage, surface textural alteration occur upon convective drying and thereby affect the quality of dried foods [9–11]. Alternatively, these effects can be reduced through a strong microwave-material interaction in microwave-assisted drying process [12].

The outcomes of various pre-treatment methods on the drying of star fruit such as submerging in sugar solution and hot water blanching and drying kinetics and associated product quality have been reported in the literature [13]. Star fruit undergoes shrinkage with significant colour change on drying [13]. Kinetics of star fruit has been reported [14]. Further, blanching of star fruit in microwave and its quality was studied. It was reported that prior to microwave blanching, the ascorbic acid and oxalic acid were found to be 7.4 (mg/g dm) and 28.04 (mg/g dm) respectively. After microwave blanching, the ascorbic acid was found to be altered to 5.8–6.6 (mg/g dm) and oxalic acid reached to the value range of 15.8–25.86 (mg/g dm) [15]. Even though few studies reported upon star fruit drying, microwave drying of star fruit has not been targeted. Hence, the present study is focussed on utilization of microwave heating for the elimination of moisture in the star fruit at different input power level. Feasibility of the operation is analysed in terms of 3E analyses such as energy, exergy also economic assessment has been performed. Further, kinetic parameters are evaluated for the microwave drying process of star fruit using different models reported in the literature. Effective moisture diffusivity and activation energy required for microwave-dried star fruit are assessed.

2 Materials and Methods

2.1 Star Fruit Sample Preparation

Star fruit is collected from the local vendors of Kamrup district, Assam, India. The fruit samples are washed and cut into slices. For each experiment, almost 25 g of fruit samples are used. The average initial moisture content is estimated by a hot air oven operated at 105 °C for 3 h. It is found that the star fruit contains an initial

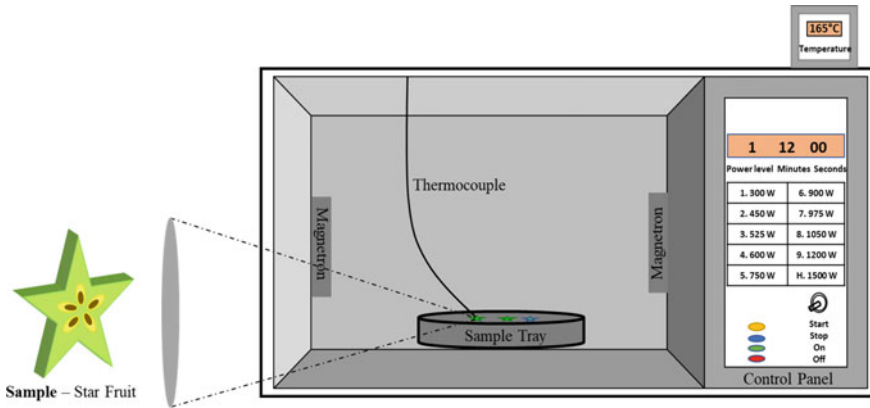


Fig. 1 Schematic view of microwave reactor

moisture of 91.5% on a wet basis (g of water/g of fruit). A slice thickness of 10 mm of the star fruit has been maintained in all experiments.

2.2 Experimental Procedure

A microwave oven (RAGA’S microwave dryer) with a power input capacity ranging from 150 to 1500 W at 915 MHz is used for star fruit drying. The layout of the microwave set-up is presented in Fig. 1. The moisture released from the drying chamber is discharged using a fan installed in the oven. A weighing balance is used externally to measure the loss of moisture by measuring the difference in the mass of fruit samples at different time intervals. The surface temperature of the star fruit is monitored using a K-type thermocouple as shown in Fig. 1. The desired sample is equally distributed in a sample tray and the loss in the moisture content is measured at various power levels of 150, 300, 450, and 525 W.

2.3 Drying Kinetics

Moisture ratio (θ) is defined as the ratio of moisture retained at a specific period to the total moisture present in the fruit with respect to the equilibrium conditions. It is expressed as:

$$\theta = \frac{(m_t - m_e)}{(m_0 - m_e)} \tag{1}$$

m_t = Moisture content of the sample at any moment.

Table 1 Various tested kinetic model equations in the present study

Model name	Model equation	References
Henderson and Pabis	$\theta = ae^{-kt}$	[17]
Newton	$\theta = e^{-kt}$	[18]
Wang and Singh	$\theta = 1 + at + bt^2$	[19]
Page	$\theta = e^{-kt^n}$	[20]
Logarithmic	$\theta = ae^{-kt} + b$	[21]
Midilli	$\theta = ae^{-kt^n} + bt$	[22]

Whereas letter a , b , n and k are constants and “ t ” is the dried time

m_0 = Initial moisture content.

m_e = Equilibrium moisture content.

All moistures are expressed in (g of water/g of dm). Table 1 shows various kinetic models from the literature to estimate the characteristics of the dried fruit [16].

All parameters of these model equations are calculated by nonlinear regression. Lower RMSE (root mean square error), χ^2 (reduced chi-square), and R^2 (coefficient of determination) confirm upon the best-fit model. These have been determined using the expression:

$$\chi^2 = \frac{\sum_{i=1}^N (\theta_{\text{pre},i} - \theta_{\text{exp},i})^2}{N - Z} \quad (2)$$

$$\text{RMSE} = \left(\frac{\sum_{i=1}^N (\theta_{\text{pre},i} - \theta_{\text{exp},i})^2}{N} \right)^{\frac{1}{2}} \quad (3)$$

$$R^2 = 1 - \frac{\sum_{i=1}^N (\theta_{\text{pre},i} - \theta_{\text{exp},i})^2}{\sum_{i=1}^N (\overline{\theta_{\text{pre},i}} - \theta_{\text{exp},i})^2} \quad (4)$$

where θ_{pre} = Predicted moisture ratio, θ_{exp} = Experimental moisture ratio, N = Total number of experimental data points and Z = Total number of parameters of the model.

2.4 Drying Rate, Effective Moisture Diffusivity and Activation Energy

The ratio of removal of moisture from wet solid per unit time is known as the rate of drying. The drying rate can be estimated with the succeeding equation:

$$DR = \frac{m_{t+\Delta t} - m_t}{\Delta t} \quad (5)$$

where m_t and $m_{t+\Delta t}$ are the moisture content of the specimen fruit at any instant “ t ” and “ $t + \Delta t$ ”.

Moisture diffusivity is assessed using Eq. 6, which is applicable for the falling rate period and for the scenario in which moisture content is below the critical limit [23]. This equation is precise to use in the microwave drying of the food samples.

$$\ln(\theta) = \ln\left(\frac{8}{\pi^2}\right) - \left(\frac{\pi^2 D_{eff}}{4L^2}\right)t \quad (6)$$

where L = One-half thickness of the sample, t = Dried time (s) and D_{eff} = Effective moisture diffusivity of the sample.

Activation energy is calculated by using the effective moisture diffusivity and with the succeeding “Eq. 7”. [24]

$$D_{eff} = D_0 \exp\left(-\frac{E_a m}{P}\right) \quad (7)$$

where E_a = Activation energy (W/g), m = mass of sample (g) and P = Microwave power level (Watt) and D_0 = Intercept.

2.5 Energy Analysis

During microwave drying, the fraction of heat used for removing water to the amount of heat provided is known as energy efficiency. It can be written as,

$$\eta_{\text{drying}}(\%) = \frac{m_{wt} \times \lambda_{wf}}{P \times t} \quad (8)$$

where m_{wt} = Mass of water removed (kg), λ_{wf} = latent heat of vaporization of free water (J/kg), P = Input microwave power level (W) and t = Dried time (s).

Specific energy consumed (E_{consumed}) is the energy needed to dry a kg of sample. It can be estimated with the following equation:

$$E_{\text{consumed}}(\text{MJ/kg water}) = \frac{P \times t \times 10^{-6}}{m_{wt}} \quad (9)$$

Specific energy loss (E_{loss}) during drying is calculated using the following expression given by Darvishi et al. [25]:

$$E_{\text{loss}} = (1 - \eta_{\text{drying}}) \times \frac{P_{in} \times t}{m_{wt}} \quad (10)$$

where m_{wt} = Amount of water removed (kg), P_{in} = Input microwave power (W), t = dried time (s) and η_{drying} = energy efficiency.

2.6 Exergy Analyses

Exergy efficiency of the drying process is estimated as follows [26],

$$\eta = \frac{EX_{evap}}{P_{in}} \quad (11)$$

where EX_{evap} = rate of utilisation of exergy for removal of moisture (J/s) and P_{in} = input microwave power level (W). The EX_{evap} is calculated using the expression:

$$EX_{evap} = \left(1 - \frac{T_0}{T_p}\right) \times m_w \lambda_{sample} \quad (12)$$

where λ_{sample} = Latent heat of the sample (J/kg), T_0 = Room temperature (K), T_p = Temperature of product (K) and m_w = Removal rate of water vapour (kg/s).

Exergy loss (expressed in MJ/Kg) can be calculated using the following equation:

$$EX_{loss} = \left(\frac{P_{in} - EX_{evap}}{m_w}\right) \times 10^{-6} \quad (13)$$

2.7 Economic Analyses

Economic analysis is performed for the microwave drying of star fruit by considering all the expenses in the drying operation. The annualized cost of the microwave is estimated with the following equation:

$$C_a = C_{ac} + C_m + C_e - V_a \quad (14)$$

where (C_a) = Annualized cost of the dryer, (C_{ac}) = Annualized capital cost, (C_m) = Annualized maintenance cost, (C_e) = Annualized electricity cost and (V_a) = Annualized salvage value [27].

The payback period is estimated as per the following equation:

$$\text{Payback period(No interest charge)} = \frac{\text{Depreciable fixed capital investment}}{\text{Avg profit/year} + \text{Avg depreciation/year}} \quad (15)$$

The cost of the microwave equipment at different capacities is found by using the cost of the known equipment and as per the following equation [28]:

$$\text{Cost of equipment X} = \text{Cost of equipment Y} * \left(\frac{\text{Capacity of equipment X}}{\text{Capacity of equipment Y}} \right)^{0.6} \quad (16)$$

3 Results and Discussion

Fresh and microwave-dried fruit sample is presented in Fig. 2. Initial moisture content of the fruit is decreased from 91.5% to a final moisture in the range of 19–24%. The fruit sample did shrink significantly due to the evaporation of moisture content.

3.1 Drying Curves

Drying curve of star fruit at several input microwave power levels is presented in Fig. 3. During microwave drying process, heating of fruit sample occurs in volumetric manner. This in turn creates variation in vapour pressure from the centre to the outer surface of the fruit.

It is quite evident that for the microwave drying of star fruit, the moisture ratio is gradually decreasing with a rise in the drying time. From this curve, it can be noticed that in the early phase of drying, there is an increasing amount of input

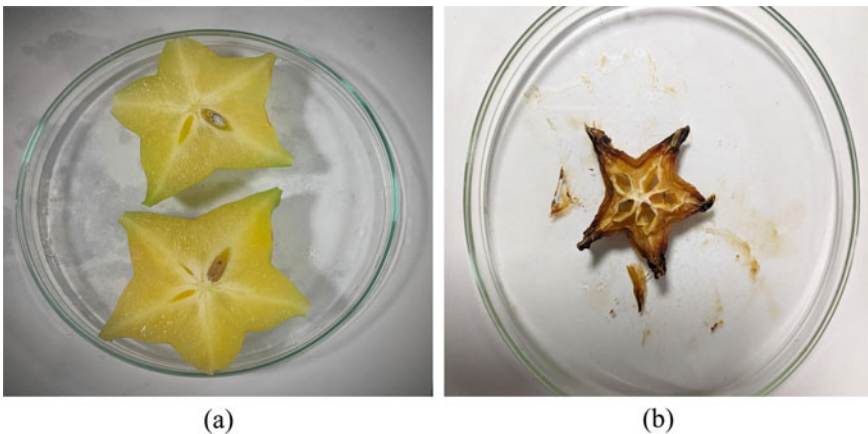
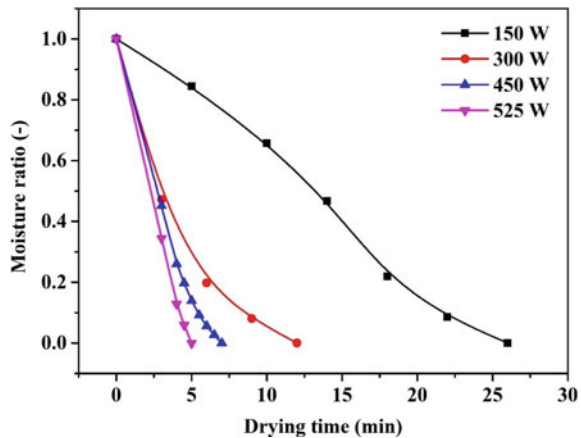


Fig. 2 Snapshot of **a** fresh and **b** microwave dried star fruit sample

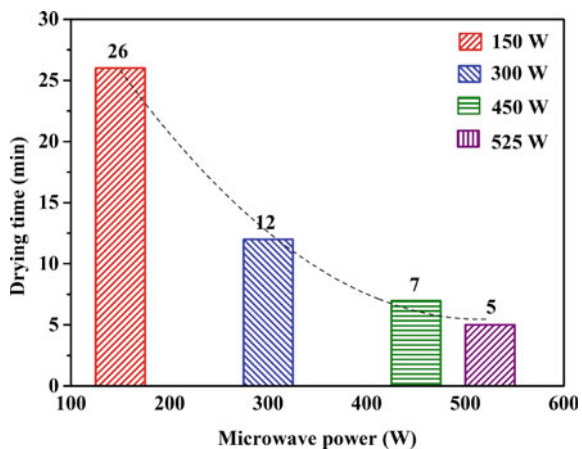
Fig. 3 Drying curve of star fruit at alternate microwave power levels



microwave power absorption due to higher moisture content and consequently, a higher rate of drying is noticed due to soaring moisture diffusion in the fruit. As drying proceeds, due to lesser moisture content, the absorption input microwave power reduced and therefore the drying rate falls. The relationship between drying time and input microwave power can be explained in Fig. 4. Drying time reduces consequently with the input microwave power level. Time needed for removing the moisture from 91.5% to the final moisture of 19–24% is found as 26, 12, 7 and 5 min at 150, 300, 450, and 525 W, respectively. As input microwave power level rises, the removal of moisture is rapid due to the higher heat generation by volumetric heating of the fruit sample. The dried time got reduced by 5 times at 525 W in comparison to that at 150 W.

For conventional hot air oven based drying, the maximum drying time for star fruit was reported as 425 min [13]. In the case of tray drying of the star fruit, the

Fig. 4 Drying time versus microwave power levels



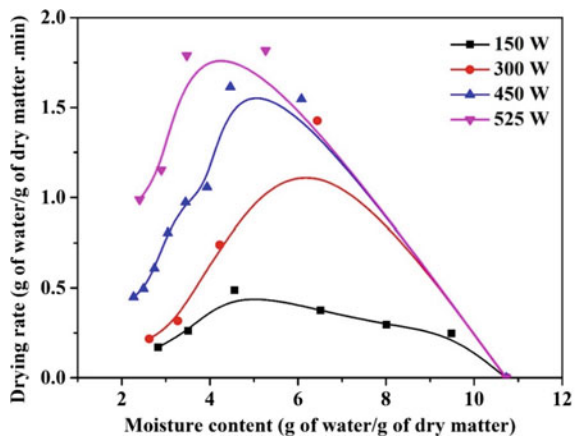
maximum drying time was reported as 300 min at 50 °C and the minimum reported drying time was 150 min at 80 °C [14]. Hence, the drying time of star fruit using a microwave oven is reduced almost by 11–16 times at 150 W microwave power level in contrast to conventional drying methods. Also, it was reported in the literature that at the 120 W input microwave power, the microwave blanching time was found to be in the extent of 300–720 s [15]. Hence, the microwave drying time of star fruits is 5 times higher than their blanching time. The microwave drying time for star fruit is found less, compared to apple pomace. During microwave drying of apple pomace, a longer drying time from 21 to 77 min is reported to remove the moisture content from 80.2 to 5% (db). For pre-treated apple slices, the total drying time was reported in the range of 6.5–23 min [18].

3.2 Drying Rate

The change of drying rate with their moisture content at various input microwave power level is presented in Fig. 5. It is noticed that there exist at least two different drying periods such as constant rate period and falling rate period. Warming up period is invisible due to the rapid absorption of input microwave power by excessive initial moisture content of the fruit. An extended constant rate period can be seen for 150 W power level, whereas this period is reduced in other power levels. An abrupt falling rate drying period is found during the power level higher than 150 W. This shows the progressive rise in the inner resistance of the fruit for the diffusion of moisture. This appears due to the immediate absence of free moisture on the surface of the fruit.

Due to the formation of pressure difference between the centres of the fruit to its surface, a rapid diffusion of moisture takes place at the constant rate period. As soon as the free moisture gets evaporated, there appears a falling rate period. In microwave drying of star fruit, the constant rate period is only visible in lower microwave power

Fig. 5 Drying rate versus moisture content curve for star fruit at various microwave power levels



of 150 W. But in the higher microwave power level, only warming up and falling rate period are visible with decreased drying time. Alike trend can be seen in the microwave drying of soybean, spinach, parsley leaves [16, 29, 30].

3.3 Kinetic Analysis

There are various models which are being used to predict thin layer drying kinetics [31].

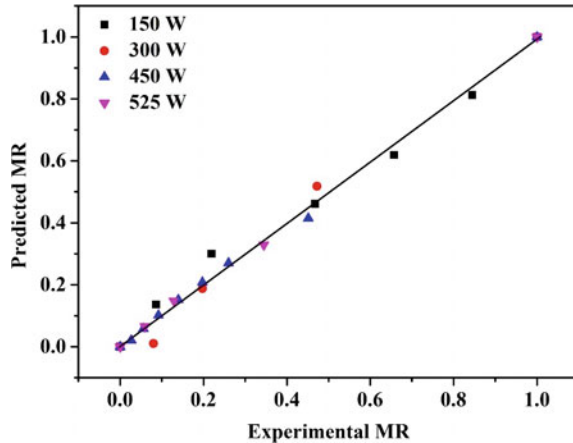
In this study, the model fitness was tested by performing nonlinear regression analysis and with Origin 2021b software. Models used to evaluate kinetic parameters of microwave-based star fruit drying process are proposed by Newton Wang and Singh, and Henderson. Using the kinetic parameters assessed from the above models, the moisture ratio was predicted to validate the fitness of the model. By using ANOVA, statistical parameters like RMSE (root mean square error), R^2 (coefficient of determination) and X^2 (chi-square) were calculated between the experimental and predicted moisture ratio. These values are shown in Table 2. The value of R^2 varies from 0.929 to 0.999, chi-square X^2 value from 0 to 0.008, and RSME from 0 to 0.012. The best-fit model can be found by the highest R^2 value and lowest chi-square (X^2) and root mean square error (RMSE) values. It is clear from the models that the value of drying constant (k) increases with enhanced microwave power and due to volumetric heat generation.

It is found that Wang and Singh model is the best fit for microwave drying of star fruit. Though in many studies, Page model and Midilli model have reported as

Table 2 Drying kinetic parameters and associated statistical parameters of the star fruit microwave drying process

Model	Power	a	b	k	R^2	Chi-square X^2	RMSE
Newton model	150			0.064	0.966	0.003	0.003
	300			0.253	0.989	0.001	0.001
	450			0.327	0.960	0.003	0.003
	525			0.480	0.999	0.000	0.000
Henderson model	150	1.135		0.075	0.929	0.006	0.005
	300	1.099		0.282	0.982	0.002	0.001
	450	1.022		0.374	0.963	0.004	0.003
	525	1.009		0.485	0.965	0.008	0.005
Wang and Singh model	150	-0.037	-0.000		0.987	0.002	0.012
	300	-0.186	0.008		0.992	0.002	0.005
	450	-0.233	0.013		0.998	0.000	0.002
	525	-0.255	0.011		0.999	0.000	0.000

Fig. 6 Plot between predicted and experimental moisture ratio using Wang and Singh model



the best fit, Wang and Singh model also affirmed significantly good fit to the experimental results. Figure 6 shows the fitness of the Wang and Singh model plotted with experimental and predicted moisture ratio at various input microwave power levels. A straight line of the plot showed that the model is quite stable for the microwave drying of star fruit. This model also showed significantly good fit for microwave drying of other foods species such as soybean [29], pepper [17], fresh turmeric fingers and cured turmeric fingers [32].

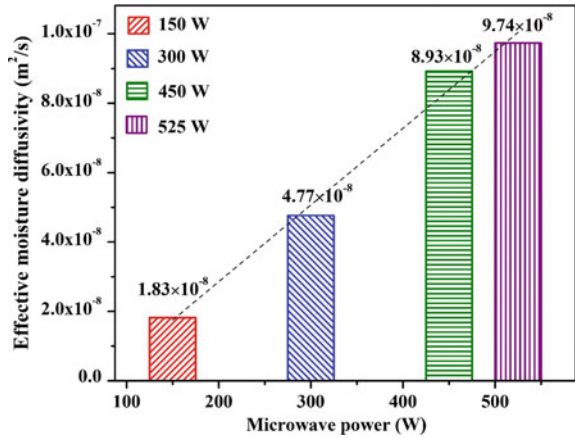
3.4 Effective Moisture Diffusivity

Effective moisture diffusivity is the rate of diffusion of the moisture in the fruit. Figure 7 shows the increase in effective moisture diffusivity with input microwave power level alteration from 150 to 525 W. It is found to alter as 1.83×10^{-8} to $9.74 \times 10^{-8} \text{ m}^2/\text{s}$ at various microwave power levels. The effective moisture diffusivity increased by 5 times at 525 W in comparison to that at 150 W. The higher moisture diffusivity at 525 W significantly reduced the constant rate period in comparison to that at 150 W input microwave power level.

The effective moisture diffusivity of various selected food specimen was found to alter as 10^{-6} to $10^{-11} \text{ m}^2/\text{s}$. The reported mean effective moisture diffusivity for white mulberry was found to alter as 1.06×10^{-8} to $3.45 \times 10^{-8} \text{ m}^2/\text{s}$ for input microwave power alteration from 100 to 500 W [25]. Incidentally, for soybean, the reported effective moisture diffusivity altered from 1.99×10^{-9} to $12.25 \times 10^{-9} \text{ m}^2/\text{s}$ for input microwave power variation from 200 to 600 W [29]. Hence, the outcomes obtained in this study showed a similar trend as in the literature.

In conventional air drying of star fruit, effective moisture diffusivity was reported to be about 3.10×10^{-10} and $3.45 \times 10^{-10} \text{ m}^2/\text{s}$ at 60 and 70 °C respectively for fresh samples [33]. It is quite evident that the moisture diffusivity for microwave drying

Fig. 7 Plot depicting the variation of effective moisture diffusivity (m^2/s) with input microwave power level (W)



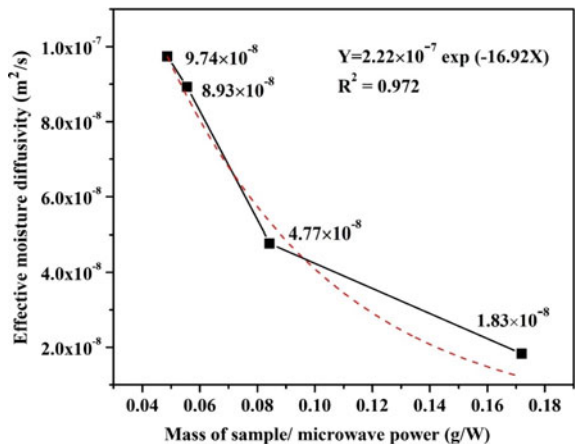
of star fruit is higher by 50 to 250 times than the reported value for the conventional drying process.

3.5 Activation Energy

The lowest amount of energy needed to commence the diffusion of moisture in the fruit is known as activation energy. It is estimated using the Arrhenius plot depicted in Fig. 8. The D_0 is estimated as $2.22 \times 10^{-7} m^2/s$ and the activation energy (E_a) of microwave-dried star fruit is found as 16.92 W/g.

Incidentally activation energy values for the microwave drying of Pandanus leaves (13.6 W/g) and mint leaves (12.3 W/g) were similar [34, 35]. The reported activation

Fig. 8 Plot depicting the variation of effective moisture diffusivity (D_{eff}) with mass of specimen/microwave power (m/p)



energy values for microwave drying of kiwi slices were 17.9, 20.1, 21.4 W/g at alternate sample thickness of 3, 6 and 9 mm respectively [36]. For pepper drying, the activation energy was found to be 14.194 W/g [17].

3.6 Energy Analysis

Energy efficiency (shown in Fig. 9), specific energy consumption (shown in Fig. 10) and specific energy losses (shown in Fig. 11) at various input microwave power levels have been assessed.

Figure 9 shows that the energy efficiency of drying has a proportional relationship with the microwave power level. Energy efficiency varied from 16.74 to 26% for an input microwave power variation from 150 to 525 W. At a low microwave power level, the quantity of water evaporated was comparatively low and drying time was comparatively high. This leads to lower energy efficiency. However, with

Fig. 9 Variation of microwave energy efficiencies (%) with microwave power (W) levels

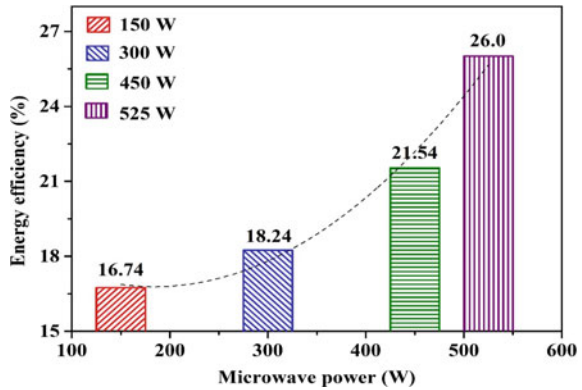


Fig. 10 Variation of specific energy consumption (MJ/kg) of star fruit with input microwave power levels (W)

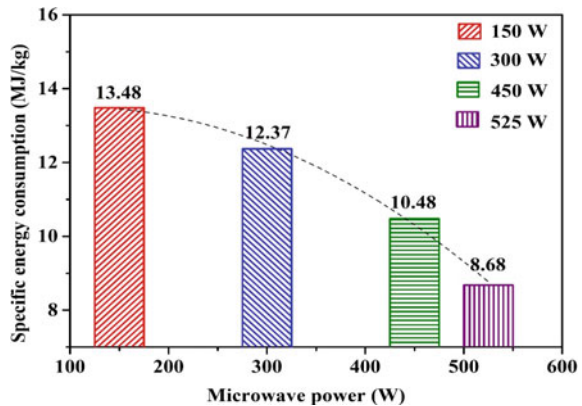
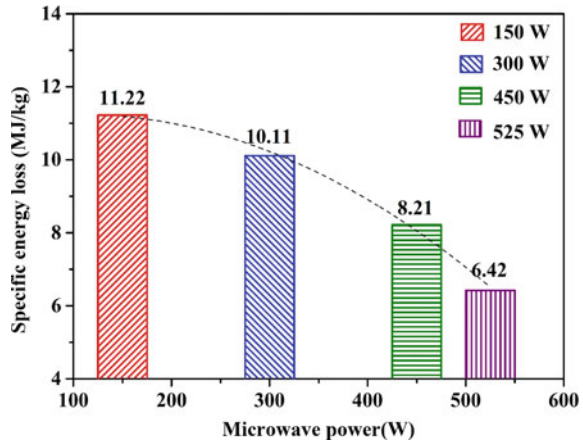


Fig. 11 Plot depicting specific energy loss (MJ/kg) of star fruit at various microwave power levels (W)



increased microwave power, the mass of evaporated water enhanced and leads to energy efficiency. Similarly, for microwave drying of turmeric slices, the energy efficiency values for fresh samples were revealed to be about 10.12 to 24.78%, and for cured samples were about 9.24 to 23.07% for an input microwave power range of 270–900 W [32]. For microwave drying of kiwi slices, the energy efficiency altered from 15.15 to 26.16% for a slice thickness of 9 mm for similar microwave power alteration [36].

As the energy efficiency increased with microwave power, the specific energy consumption (SEC) and specific energy loss were consequently reduced. This can be seen in Figs. 10 and 11. Specific energy consumption (SEC) and specific energy loss reduced from 13.48 to 8.68 MJ/kg and 11.22 to 6.42 MJ/kg, respectively for the microwave drying of star fruit. There is a relative reduction in the specific energy consumption (SEC) of 35.62% at 525 W in comparison to that at 150 W microwave power level. The reported SEC value for microwave drying of apple slices was 6.2 MJ/kg at 360 W [37]. In the case of microwave drying of soybean, the reported SEC and specific energy loss were reduced from 9.12 to 4.91 MJ/kg and 6.04 to 1.67 MJ/kg, respectively, for a surge in microwave power from 200 to 600 W [29].

3.7 Exergy Analyses

The consequence of altered microwave power on exergy efficiency and specific exergy loss can be seen from Figs. 12 and 13 respectively. With an increase in microwave power, the exergy efficiency enhanced and specific exergy loss reduced. Exergy efficiency is 3.93, 6.04, 8.74, and 11.99% at various input microwave power levels of 150, 300, 450, and 525 W, respectively. While comparing with the energy efficiency, the exergy efficiency is found to be lower by 12–14% for all microwave power levels. This is due to entropy generation. Similar trends and results have been

revealed in microwave-dried turmeric slices. In this study, the exergy efficiency for fresh samples in the range of 2.18–12.77% for a surge in input microwave power from 270 to 900 W [32].

The specific exergy loss is found to reduce from 12.95 to 7.64 MJ/kg for a surge in input microwave power level from 150 to 525 W. Although exposure of microwave at high power levels leads to high-temperature regimes in the fruit affirming higher exergy loss, a reverse trend is observed with the reduction in the exergy loss at elevated input microwave power level. This may be due to elevated drying time during the low microwave power input leading to higher entropy generation in the system.

Fig. 12 Exergy efficiencies (%) of star fruit at various levels of microwave power (W)

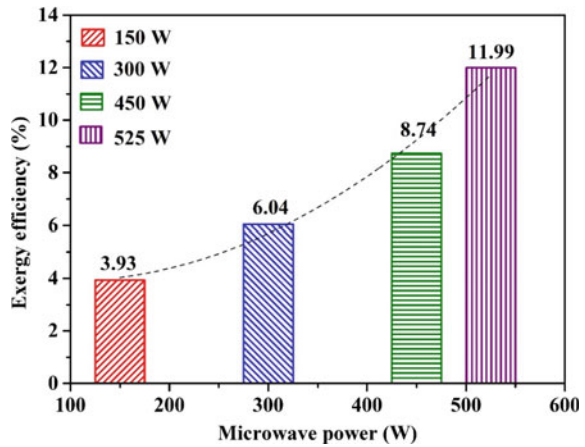
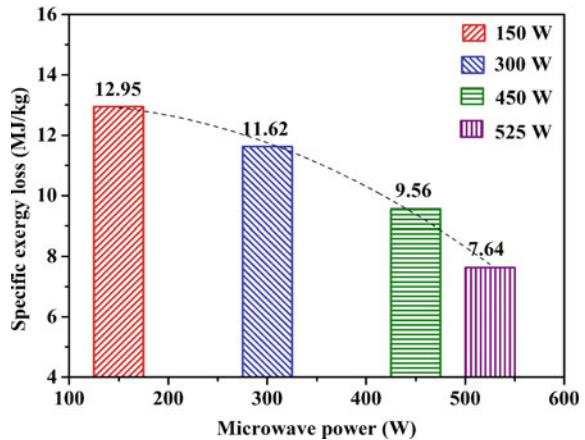


Fig. 13 Specific exergy loss (MJ/kg) of star fruit at various levels of microwave power (W)



3.8 *Fourier Transform Infrared Spectroscopic Analyses*

The functional groups alteration in the star fruit after microwave drying is analysed by FTIR from 4000 to 500 cm^{-1} . FTIR analysis of the fresh star fruit and dried star fruit at various input microwave power levels (150, 300, 450, and 525 W) is shown in Fig. 14. The fresh star fruit indicated a broader peak at 3315 cm^{-1} . This is recognised as O–H stretching due to the inherent moisture. The dried star fruit demonstrated different functional groups in comparison with the raw fruit [38]. The peak around 1640 and 1066 cm^{-1} can be recognised as C=O and C–O stretching respectively. C=O stretching represents ketones, aldehydes, carboxylic acids and C–O represents primary, secondary, and tertiary alcohols [39]. For dried star fruit, the peaks at the wave numbers 2924 and 2850 cm^{-1} can be recognised as C–H and CH_3 . Around the wavenumbers 1460 and 1033 cm^{-1} , the peaks were recognised as C–H and C–O and thereby affirmed on the presence of ketones, aldehydes, carboxylic acids and primary, secondary and tertiary alcohols. Hence, this analyses showed the retention of various acids, primary, secondary and tertiary alcohols in dried sample due to water removal. The functional groups in dried fruits are found to be identical at all microwave power levels. This showed that the fruit sample is able to retain all its chemical constituents at a higher micro wave power level of 525 W and without any deterioration.

3.9 *Economic Analysis*

The economic analysis is executed to calculate the cost of dried fruit by microwave drying and in terms of payback period. Table 3 shows the economic parameters considered in the present study.

The payback period for the microwave drying of star fruit is estimated as 6.13 years and the life period of the equipment as 20 years. It is found similarly in the literature that confirmed payback period of a greenhouse dryer to be about as 3.2 years for a service life period of about 10 years [27].

Figure 15 shows the price of drying per kg of the final product obtained for altered microwave power and plant capacity. It can be seen that the cost of drying reduced with enhanced with capacity and microwave power level. The cost of drying with 0.6 kg plant capacity at 525, 450, 300 and 150 W is found as 4.63, 6.36, 8.64, and 15.27 INR respectively. At 1000 kg capacity, the cost of drying is reduced below 1 INR at 525, 450, 300 and 150 W microwave power levels.

Fig. 14 FTIR analysis of starfruit (A represents fresh starfruit and B, C, D, E represent dried starfruit at 150, 300, 450, and 525 W respectively)

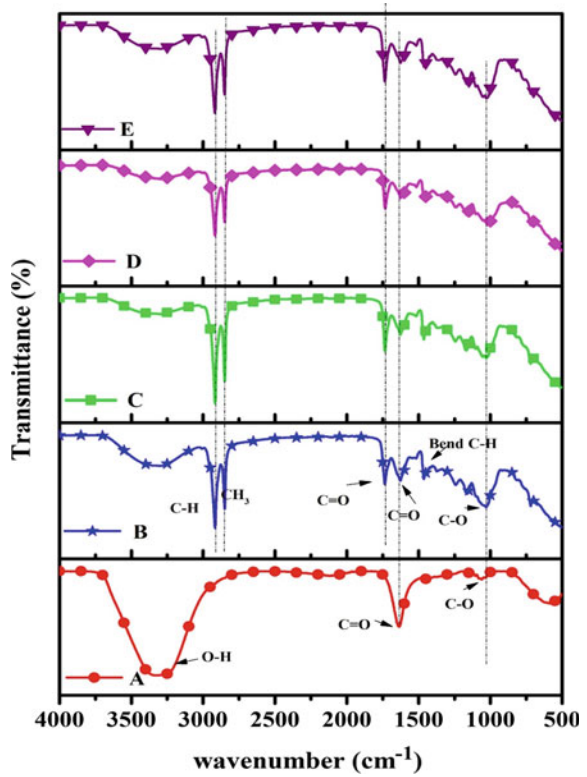


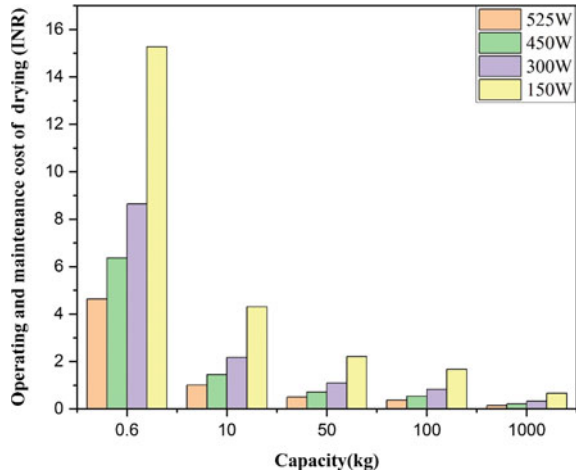
Table 3 Economic parameters for the estimation of cost of the dried products

Economic parameters	Values
Capital cost of microwave dryer	450,000 INR
Annual depreciation	10%
Service life of microwave	20 years
Salvage value	54,709.5 INR
Rate of interest	5%
Rate of inflation	3%
Rate of discount	8%
Electricity charge	7.4 INR/kwh
Number of hours microwave run	7200 h/year

4 Conclusions

This study reveals the drying characteristics of microwave-dried star fruit. Activation energy, effective moisture diffusivity and drying kinetics are estimated. A 3E analysis

Fig. 15 Variation of operating and maintenance cost (in INR) with capacity



(energy, exergy and economic) is performed to assess the feasibility of commercial implementation.

- The drying time under microwave condition at 150 W is reduced significantly by 11–16 times in conjunction with the conventional drying method. FTIR analyses confirmed that the microwave drying up to a certain input microwave power level of 525 W did not deteriorate the chemical constituents of star fruit.
- A predominant falling rate period is observed at elevated input microwave power levels (300–525 W). However, a constant drying rate period is prevailed at low input microwave power levels at 150 W. The microwave drying enhanced the moisture diffusivity by 50–250 times in comparison to the conventional air drying method.
- Wang and Singh model is the best-fit model for the obtained experimental drying rate characteristics of the microwave drying of star fruit.
- With increased input microwave power, the energy efficiency of the microwave drier enhanced from 16 to 26%. Hence, both specific energy consumption, specific energy loss got reduced. The exergy efficiency is obtained as high as 12% at 525 W due to the entropy generation.
- At a higher capacity of 1000 kg drier, the operating cost of drying reduced to reach below 1 INR for all microwave power levels.

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Recovery of Bioactive Constituents from Unripe Papaya Peel and Pulp Using Ultrasound Assisted Extraction: Optimization of Process Parameters



Khalid Mehmood Wani and Ramagopal V. S. Uppaluri

Abstract Papaya (*Carica papaya L.*) contains a wide range of bioactives with beneficial properties for human metabolism. However, solvent selection, processing time, and extraction efficiency are a few issues that need to be considered during such investigations. The conducted study aimed to determine the influence of aqueous ultrasonic-assisted extraction (USAE) process factors on the total flavonoid constitution (TFC), total polyphenolic constitution (TPPC), antioxidant activity (ANOX) and vitamin C (VITAC) from papaya pulp (PPU) and papaya peel (PPE) system along with the hot water extraction (HWE) process. The investigation was carried out utilizing a face-centered design (FCD) response surface approach (RSM). The impact of sonication process factors such as sonication time (SOT) (5–20 min), extraction temperature (EXT) (40–70 °C), and solid to solvent ratio (SLR) (0.2–0.5 mL⁻¹) were all targeted. Based on the results, the bath type ultrasound-assisted system inferred 53.35–145.21 mg GAEg⁻¹ total polyphenolic constitution (TPPC), 35.25–85.83% antioxidant activity (ANOX) and 16.44–139.5 mg (100 g)⁻¹ vitamin C (VITAC) for the PPU case. The investigations affirmed that the sonication process performed quite well in comparison with the HWE. Besides, the study also quantified that the extract of PPU constituted the maximum amount of bioactives in comparison with the PPE system. These results demonstrate that both PPU and PPE systems are promising sources of bioactive constituents and that UAE can be tailored as a doable and environment benign approach for the formulation of products with the larger constitution of the aqueous bioactive extracts and for food and pharmaceutical industries.

Keywords Bioactives · Papaya pulp · Papaya peel · Ultrasound assisted extraction · Hot water extraction · Optimization

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Highlights

- USAE of bioactives from PPU and PPE was investigated.
- PPU extract possessed more bioactives than PPE extract.
- Optimisation study was conducted with FCD-based RSM.
- Effect of USAE factors (sonication time, extraction temperature, and loading ratio) on response variables were targeted.
- Effect of loading ratio was predominant, followed by extraction temperature and sonication time.

1 Introduction

In recent times, there has been renewed thrust in the recovery of bioactives from fruits and vegetables. Bioactive components, being the secondary class of metabolites derived from the plants, are present in minute quantities and have very high nutritional value [1]. These compounds definitely contribute to desired features such as the reduction of the human health ailments and precision towards the non-occurrence of neurodegenerative disorders such as heart disease, inflammation, cancer, arthritis, arteriosclerosis, cognitive dysfunction, and aging process [2]. Thus, growing awareness upon bioactive components in terms of their potential health benefits prompted researchers to identify efficient extraction methods [3]. In this scenario, focussing on the extract products from the plant sources characterised with low cost, abundance and ease of isolation is often targeted through sustainable extraction to meet the demands of the consumers [4].

Papaya (*Carica papaya L.*) is a well-known low-cost fruit in the *Caricaceae* family and is grown all through in the year [5]. Unripe papaya pulp (PPU) has a rich constitution of proteins, carbohydrates, and vitamins. Conversely, as the fruit ripens, the concentration of these constituent's decreases [6]. Papaya is a widely cultivated fruit and is often consumed due to its excellent antioxidant activity. The fruit is used as a traditional medix due to its vitality for notable nutrients such as carotenoids, vitamins and is a good source of iron and calcium [7]. The fruit also contains two biologically important enzymes namely papain and chymopapain which are well known for their utility towards the treatment of ulcers, digestion problems, traditional medicine for various ailments such as wound healing and abortifacient activity [8]. The PPU also contains other bioactive components such as phenols and polyphenols that are well known for their pharmacological significance [9].

Numerous research findings have been reported for the assessment of the biological activity of various components of the papaya fruit. These refer to the leaves, rinds, fruits, peel, shoots, root and seeds of the papaya plant [10]. Papaya peel (PPE) is one of the most common by-products of papaya processing and makes up roughly 12% of the fruit's overall weight [11]. There are several value-added bioactive substances that are found in PPE, including, flavonoids, polyphenols, carbohydrates, fibres, fatty acids, proteins tannins, and minerals [12]. These ingredients can be effectively used as dietary supplements and nutraceutical additives in innovative pharmaceutical and

food products. In the past, PPE has been used as a component in pet food, cosmetics, and some home remedy systems [13]. Recent studies have shown that both PPU and PPE are excellent sources of bioactives that may be recovered and purified to serve as a significant component in a variety of value-added products. Hence, in order to extract these bioactives from PPE and PPU, a simple, quick, and inexpensive method must be explored [9].

So far, many alternative methodologies have been being deployed to recover bioactive components from plant resources. The selection of extraction method is affected by two main factors namely extraction yield and operational cost [14]. In comparison to conventional extraction techniques, ultrasound assisted extraction (USAE) reduces processing time and solvent consumption, improves permeation, increases yield and extraction rate, improves the quality of the extract, allows the usage of more environmental friendly alternative solvents and low energy requirements [15]. Both deployed extraction process and the solvent type critically influence upon the extract's quality and yield [16]. Food-grade solvents such as methanol, acetone water, ethanol, or their combinations are commonly deployed in the production of solvent extracts. Water, on the other hand is regarded as environmentally safe and non-toxic, affordable and widely accessible food grade organic solvent [17]. Hence, the primary goal of the research was to improvise the USAE technique utilising water as the extractant media to obtain the greatest proportion of bioactive components in the PPU and PPE extracts.

Recent studies have demonstrated that acoustic cavitation and mechanical effects of the USAE can substantially increase the recovery of bioactive components from diverse parts of the plant resources [18]. USAE utilizes the principle of cavitation which causes the breakage of the plant cellular structure and thereby enables the liquid solution to seep into the plant material and release its intracellular components [19]. The USAE generates small bubbles in the liquid media (cavities) which form, grow and collapse with the plant material [20]. The collapse of cavitation bubbles results in improved cell disruption and increased mass transfer from the plant material [21]. Several factors impact the extraction yield of bioactive components during USAE. Prominent among these are sonication duration (SOT), solid to solvent ratio (SLR), extraction temperature (EXT), and particle size [22]. As a result, it is crucial to optimise the extraction conditions in order to obtain a greater yield of bioactives.

For the optimization of extraction techniques, various experimental design methods have been utilized. Well-known among these are single-factor trials and response surface methodology (RSM) [23]. Among these, the first one is complex and time consuming and does not facilitate any interaction among the process factors [24]. RSM, on the other hand, is more reliable method due to its consideration of the interactive impact of the diverse process factors (linear, quadratic, and interaction impacts on response variables) and its promising ability to foster a set of optimum conditions through a reduced conduct of the experimental trials [25]. According to the reported literature, very few investigations exist that address the recovery of bioactives from PPU and PPE using bath type ultrasound extraction system and water as a solvent [9, 26].

In summary, it can be concluded that the prior art did not consider towards the bioactive production from PPU and PPE. Precisely, the available prior art has the following lacunae. Firstly, no investigation targeted the bioactive compounds recovery from PPU and PPE using only USAE. Secondly, the impact of process factors of USAE (SLR, SOT and EXT) on bioactives recovery from PPU and PPE extract was not studied. Thirdly, RSM-based optimisation of process factors of USAE was not targeted. Fourthly, water as an extracting media or green solvent was not considered in the prior art to maximize extraction of the bioactive constituents from PPU and PPE. Thus, the objective of this article is to achieve aqueous extract of PPE and PPU by employing the RSM with face centered design (FCD) methodology to evaluate the (1) optimization of process factors such as SOT, EXT, and SLR to achieve maximum constitution of response variables namely the total flavonoid constitution (TFC), total polyphenolic constitution (TPPC), antioxidant activity (ANOX), and vitamin C (VITAC) for PPU and PPE based aqueous extracts and (2) comparison of efficacies of the USAE and hot water extraction (HWE) for bioactives yield from PPU and PPE.

2 Materials and Methods

2.1 Raw Materials and Sample Preparation for Papaya Peel and Pulp Extraction

The raw materials and chemicals deployed to conduct this study have been published elsewhere and detailed description for sample preparation can be acquired from our previous work [15]. Briefly, USAE extraction was subsequently carried out by placing a certain amount of PPU and PPE pieces (5 mm) in 100 mL capacity beaker together with the specified amount of water. These conditions were achieved using the FCD-RSM method (Tables 1 and 2).

2.2 Extraction Methods

Two extraction methods are compared in the present work for the extraction of bioactives from PPU and PPE. These are USAE and HWE. A detailed description on the experimental work for USAE and HWE can be obtained from our previous work [15].

Table 1 Experimental data summary of response variables for USAE of PPU—water system

S. No	Temperature (°C) (A)	Time (min) (B)	Papaya pulp/water ratio (g mL ⁻¹) (C)	Total polyphenols (mg GAE g ⁻¹)	Antioxidant activity (%)	Vitamin C (mg) (100 ⁻¹ g ⁻¹)
1	40.00	5.00	0.20	53.35	35.25	16.44
2	40.00	20.00	0.20	94.76	50.82	56.22
3	55.00	12.50	0.20	80.8	47.26	30
4	70.00	5.00	0.20	89.38	55.19	30.47
5	70.00	20.00	0.20	115.05	67.67	75.23
6	40.00	5.00	0.50	118.5	70.63	76.96
7	40.00	20.00	0.50	140.25	78.13	98.87
8	55.00	12.50	0.50	129.74	74.7	90.5
9	70.00	5.00	0.50	138.2	78.9	106
10	70.00	20.00	0.50	143.92	83.61	134.46
11	40.00	12.50	0.35	114.8	66.13	101
12	55.00	5.00	0.35	118.33	69.66	105
13	55.00	12.50	0.35	124.84	76.12	117.3
14	55.00	12.50	0.35	125.7	75.1	118.7
15	55.00	12.50	0.35	124.52	72.78	113.56
16	55.00	12.50	0.35	124.68	76.12	115.57
17	55.00	12.50	0.35	122.4	73.55	118.5
18	55.00	12.50	0.35	125.9	74.5	117.4
19	55.00	20.00	0.35	145.21	85.83	139.5
20	70.00	12.50	0.35	137.06	81.1	132.6

2.3 Analysis of Extract

The PPU and PPE aqueous extracts were investigated for TPPC, TFC, % ANOX, and VITAC. For TPPC analysis, Folin–Ciocalteu procedure with procedural modifications [27] was used to determine the TPPC constitution of raw PPU and PPE extract. To evaluate the TFC of PPU and PPE, AlCl₃ method was adopted [27]. The ANOX of aqueous PPU and PPE extract was determined by employing the DPPH method [28] with the following equation:

$$ANOX (\%) = \frac{A_C - A_S}{A_C} \times 100 \quad (1)$$

where A_s and A_c are the absorbance's of sample and control at 517 nm.

The ascorbic acid concentration of PPU and PPE extract was determined using the 2,6-Dichlorophenol indophenol titration test [15]. Utilising titration volumes V₁

Table 2 Experimental data summary of response variables for USAE of PPE—water system

S. No	Temperature (°C) (A)	Time (min) (B)	Papaya peel/water ratio (g mL ⁻¹) (C)	Total polyphenols (mg GAE g ⁻¹)	Total flavonoids (mg QE g ⁻¹)	Antioxidant activity (%)
1	55.00	20.00	0.35	119.26	290	80.36
2	40.00	5.00	0.50	117.42	240.89	70.62
3	55.00	12.50	0.35	116.7	270	74.1
4	70.00	5.00	0.20	83.5	135.64	58.6
5	55.00	12.50	0.35	116.4	269.7	73.75
6	40.00	12.50	0.35	109.79	250.87	68.2
7	55.00	12.50	0.35	117.1	272	75.79
8	70.00	5.00	0.50	119.67	260	74.92
9	40.00	5.00	0.20	53.68	70.67	47.67
10	70.00	20.00	0.50	123.7	275.56	75.3
11	70.00	20.00	0.20	102.39	176.08	67.85
12	55.00	12.50	0.20	81.21	117.55	55
13	55.00	5.00	0.35	105.93	253	72.59
14	55.00	12.50	0.50	120.86	252.03	72.84
15	40.00	20.00	0.50	121.79	253.11	74.3
16	55.00	12.50	0.35	115.8	274.4	75.5
17	55.00	12.50	0.35	115.5	270.2	75.8
18	40.00	20.00	0.20	76.77	105.2	55
19	55.00	12.50	0.35	116.45	274	74.5
20	70.00	12.50	0.35	126.6	302	77.7

and V_2 , the VITAC content was calculated using the following equation:

$$VITAC (mg/100g) = \frac{0.5 \times V_2 \times 20 \times 100}{V_1 \times 5 \times W_s} \quad (2)$$

where W_s , V_1 and V_2 respectively, denotes the sample mass, dye volumes pertaining to the standard and sample.

2.4 Experimental Design

A detailed discussion on experimental design for the UASE of bioactives from PPU and PPE can be obtained from our previous work [15]. Using the Design Expert software (Version 13.0, State-Ease Inc., MN, USA), the FCD-based RSM was established. The second-order polynomial model was evaluated for the fitness with the

data constituting three independent process factors and three response variables:

$$Y = \beta_0 + \sum_{i=1}^n \beta_i X_i + \sum_{i=1}^n \beta_{ii} X_i^2 + \sum_{i=1}^n \sum_{j=i+1}^n \beta_{ij} X_i \beta_i X_j \quad (3)$$

where β_0 , β_i , β_{ii} , and β_{ij} ($i \neq j$) and X_i and X_j respectively are the regression coefficients for intercept, linear, quadratic, and interaction terms, and independent variables.

3 Results and Discussion

3.1 Experimental Summary

The USAE process factors for bioactives extraction from PPU and PPE were achieved after conducting a few experimental studies. The SOT, EXT and SLR respectively, varied in the range of 5–20 min, 40–70 °C and 0.2–0.5 g mL⁻¹. The upper levels of process factors for SOT and EXT respectively were selected as 20 min and 70 °C. This has been due to the reason that most bioactive components get degraded for process conditions of higher EXT and long-time duration [15]. However, in this study, it was identified that at higher SLR (0.5 g mL⁻¹), the extent of the yield enhancement narrowed. Likewise, observations were inferred for the recovery of bioactives from the peels of black chokeberry and pomegranate [29]. The TPPC, VITAC, and % ANOX yield for PPU respectively varied significantly during USAE (as shown in Table 1) for a variation in SLR in the range of 0.2–0.5 g mL⁻¹, SOT in the range of 5–20 min, and EXT in the range of 40–70 °C as 53.35–145.21 mg GAEg⁻¹ sample, 16.44–139.5 mg (100 g)⁻¹, and 35.25–85.83%. Likewise, results were found for PPE (Table 2), for a variation in SLR, SOT, and EXT respectively in the range of 0.2–0.5 g mL⁻¹, 5–20 min, and 40–70 °C resulted in yields of TPPC, TFC, and ANOX ranging in the range of 53.68–126.6 mg GAEg⁻¹ sample, 70.67–302 mg QE g⁻¹, and 47.67–80.36%.

For the USAE of PPU, the finest experimental data were obtained as 0.35 g mL⁻¹ SLR, 55 °C EXT and 12.50 min SOT. For the PPE case, the finest experimental data were obtained as 0.35 g mL⁻¹ SLR, 70 °C EXT, and 12.50 min SOT for TPPC and TFC. However, for the ANOX, the finest experimental data corresponds to 55 °C EXT, 12.50 min SOT and 0.35 g mL⁻¹ SLR. Furthermore, the HWE ensured lesser amount of bioactives in comparison to the USAE of PPU and PPE. The HWE was conducted at the optimised condition of USAE. During HWE, the TPPC, VITAC and % ANOX yield for PPU case were obtained respectively as 105 mg GAE g⁻¹, 89.4 mg 100⁻¹ g⁻¹, and 76.50%. Likewise, for PPE, the yield of TPPC, ANOX and TFC respectively have been obtained as 88.5 mg GAE g⁻¹, 71.5% and 145 mg QE g⁻¹.

Model fitting for USAE of PPU: Table 3 gives an account of the analysis of variance (ANOVA) statistics for the TPPC, ANOX, and VITAC for the PPU case. The mathematical equation correlating the response variables with the UAE process variables has been established as follows:

$$\begin{aligned} TPPC = & 124.96 + 10.19A + 12.14B + 23.73C - 3.97AB - 4.12AC \\ & - 4.95BC + 0.55A^2 + 6.39B^2 - 20.11C^2 \end{aligned} \quad (4)$$

$$\begin{aligned} ANOX = & 74.39 + 6.55A + 5.64B + 12.98C - 0.73AB - 2.88AC \\ & - 1.98BC + 0.33A^2 + 3.80B^2 - 12.96C^2 \end{aligned} \quad (5)$$

$$\begin{aligned} VITAC = & 115.57 + 12.93A + 16.94B + 29.84C + 1.44AB + 3.95AC \\ & - 4.27BC + 3.13A^2 + 8.58B^2 - 53.42C^2 \end{aligned} \quad (6)$$

Model fitting for USAE of PPE: Table 4 displays the ANOVA data summary for the response variable for USAE of the PPE system. As stated previously, the empirical data were fitted to the second-order polynomial model. Thus, the following are the finest quadratic models for TPPC, ANOX, and TFC characteristics of the USAE of the PPE extraction system:

Table 3 Analysis of variance data summary for USAE of PPU—water system

Components	Total polyphenols		Antioxidants activity		Vitamin C	
	<i>F</i> value	<i>P</i> value	<i>F</i> value	<i>P</i> value	<i>F</i> value	<i>P</i> value
Model	646.33 (Quadratic)	<0.0001 (Quadratic)	118.96 (Quadratic)	<0.0001 (Quadratic)	373.84 (Quadratic)	<0.0001 (Quadratic)
A	603.79	<0.0001	145.00	<0.0001	226.44	<0.0001
B	856.58	<0.0001	107.59	<0.0001	388.90	<0.0001
C	3270.39	<0.0001	569.07	<0.0001	1206.84	<0.0001
AB	73.29	<0.0001	1.46	0.2547	2.25	0.1644
AC	78.84	<0.0001	22.42	0.0008	16.90	0.0021
BC	113.93	<0.0001	10.60	0.0086	19.78	0.0012
A ²	0.48	0.5038	0.098	0.7602	3.65	0.0853
B ²	65.20	<0.0001	13.45	0.0043	27.42	0.0004
C ²	646.14	<0.0001	156.07	<0.0001	1063.49	<0.0001
Lack of fit	1.21	0.4186	2.20	0.2032	2.86	0.1367
R squared		0.9983		0.9907		0.9970
Adequate precision		99.305		41.384		65.504

Table 4 Analysis of variance data summary for USAE of PPE—water system

Components	Total polyphenols		Total flavonoids		Antioxidant activity	
	<i>F</i> value	<i>P</i> value	<i>F</i> value	<i>P</i> value	<i>F</i> value	<i>P</i> value
Model	1122.30 (Quadratic)	<0.0001 (Quadratic)	1074.91 (Quadratic)	<0.0001 (Quadratic)	111.60 (Quadratic)	<0.0001 (Quadratic)
A	843.28	<0.0001	563.67	<0.0001	101.50	<0.0001
B	586.26	<0.0001	210.77	<0.0001	55.04	<0.0001
C	6122.71	<0.0001	4938.27	<0.0001	479.57	<0.0001
AB	3.72	0.0826	1.15	0.3079	0.16	0.6955
AC	474.77	<0.0001	119.93	<0.0001	29.11	0.0003
BC	203.58	<0.0001	30.04	0.0003	13.36	0.0044
A ²	17.87	0.0018	6.97	0.0247	2.83	0.1233
B ²	48.08	<0.0001	2.332E-003	0.9624	9.89	0.0104
C ²	898.36	<0.0001	2235.95	<0.0001	197.38	<0.0001
Lack of fit	3.03	0.1247	3.25	0.1109	2.59	0.1598
R squared		0.9990		0.9990		0.9901
Adequate precision		122.262		107.201		39.078

$$TPPC = 126.55 + 9.56A + 4.60B + 16.68C + 0.61AB - 4.87AC - 2.06BC - 0.88A^2 - 1.72B^2 - 14.31C^2 \quad (7)$$

$$ANOX = 74.62 + 3.86A + 2.84B + 8.39C - 0.17AB - 2.31AC - 1.56BC - 1.23A^2 + 2.30B^2 - 10.26C^2 \quad (8)$$

$$TFC = 271.67 + 22.85A + 13.97B + 67.64C + 1.16AB - 11.79AC - 5.90BC + 4.85A^2 - 0.089B^2 - 86.80C^2 \quad (9)$$

3.2 Response Surface Characteristics

Impact of USAE process variables on TPPC: The TPPC response surface characteristics (RSP) depicts similar pattern for both PPU and PPE. Figure 1a–c and d–f respectively shows the impact of any two process variables on TPPC for the PPU and PPE extract of USAE. For PPU and PPE, the 3D surface graph clearly shows that the TPPC yield enhances for any two combinations of SOT, EXT, and SLR. The TPPC rose considerably in the range of 53.35–140.25 mg GAEg⁻¹ for PPU and 53.68–121.79 mg GAEg⁻¹ for PPE for an alteration in SOT and SLR respectively in the range of 5–20 min and 0.2–0.5 mL⁻¹ at 40 °C. Also, there was a little increase

in the TPPC yield when SLR was changed in the range of 0.35–0.5 g mL⁻¹ and a small dip in the TPPC yield was observed when SLR reached to 0.5 g mL⁻¹.

Likewise, at 0.35 g mL⁻¹ SLR, initially a steady increase in the TPPC yield for both PPU and PPE cases was observed. However, the corresponding TPPC yield started increasing. The TPPC for PPU and PPE respectively altered in the range of 114.8–145.21 mg GAEg⁻¹ and 109.79–119.26 mg GAEg⁻¹, for the SOT and EXT variations in the range of 12.5–20 min and 40–55 °C respectively.

In addition, for a given SOT of 20 min, the TPPC for PPU and PPE respectively altered in the range of 94.76–145.21 mg GAE g⁻¹ and 76.77–123.7 mg GAE g⁻¹, for an alteration of SLR in the range of 0.2–0.5 g mL⁻¹ and EXT in the range of 40–70 °C. The 3D plot shows that the TPPC yield grows substantially with SLR and EXT and eventually reaches a maximum in the middle region and eventually reduces marginally. Thus, for PPU and PPE respectively, the maximum TPPC yield of 145.21 mg GAE g⁻¹ and 126.6 mg GAE g⁻¹ was obtained at 55 °C, 12.5 min, and 0.35 g mL⁻¹ and 70 °C, 12.5 min, and 0.35 g mL⁻¹. The 3D plot confirmed nonlinear alteration in the TPPC in relation to any two process variables. The TPPC was analysed to be comparatively sensitive as per the hierarchy of SLR > SOT > EXT among all considered degrees of freedom.

Impact of USAE process variables on ANOX: For the ANOX case, the SLR, followed by EXT and SOT respectively, profusely impacted the response profiles for PPU and PPE (Fig. 2a–f). The 3D response plot (RSP) at a given SLR demonstrated that every combination of SOT and EXT fostered an enhanced ANOX value set. The ANOX surface plot depicts similar pattern for both PPU and PPE systems. At the beginning, the ANOX increases with the SOT and SLR and eventually reaches a highest value at 0.35 g mL⁻¹. Afterwards, small decline in the yield of ANOX.

Additionally, the ANOX rose considerably in the range of 35.25–78.13% for PPU and 47.67–75.3% for the PPE case for the respective extraction duration and SLR alterations of 5–20 min and 0.2–0.5 g mL⁻¹, at 40 °C. Conversely, for a fixed 0.2 g mL⁻¹ SLR, ANOX altered in the range of 35.25–50.82% for PPA and 47.67–67.85% for PPE for the SOT and EXT respectively for a variation in the range of 5–20 min and 40–70 °C. These alterations affirmed that a linear increase prevailed initially for the ANOX with SOT and EXT. Eventually, the yield increased and reached saturation in the USAE process.

With changes in SLR and EXT respectively in the range of 0.2–0.5 g mL⁻¹ and 40–70 °C, and for a constant choice of 20 min SOT, the ANOX varied substantially in the range of 35.25 to 83.61% for PPU and 55–75.3% for PPE. At first, the yield increased linearly with a variation in EXT and SLR and eventually reached a maximum value. Thereafter, a small dip in the ANOX yield was evident. The highest combination of USAE factors for the PPU case corresponds to 55 °C EXT, 0.35 g mL⁻¹ SLR and 12.50 min, for which the highest antioxidant activity of 85.83% was obtained. For the PPE case, the finest process factors were 0.35 g mL⁻¹ SLR,

Impact of USAE process variables on TFC: When EXT (70 °C) was held constant, the TFC increased substantially in the range of 135.64–275.56 mg QE g⁻¹ for an alteration in SLR in the range of 0.2–0.5 g mL⁻¹ and SOT in the range of 5–20 min. The RSP (Fig. 3a–c) affirmed linear escalation of the TFC yield during

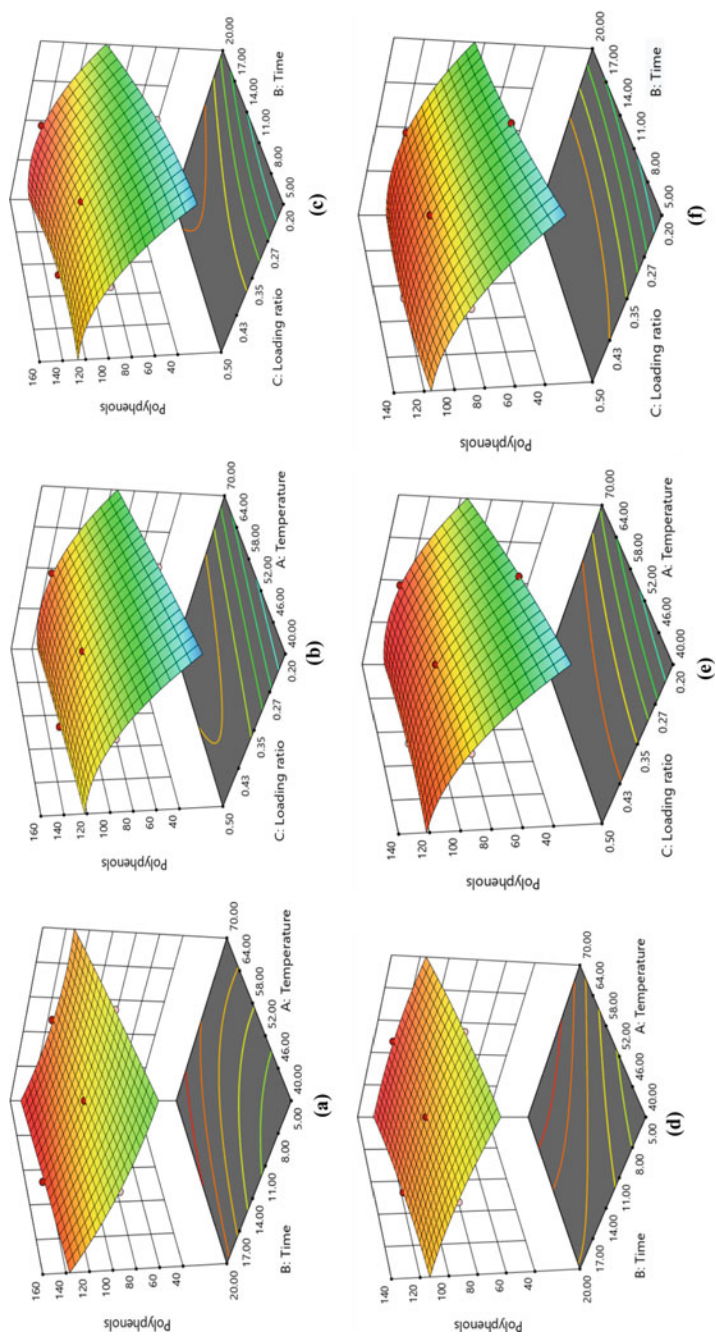


Fig. 1 TPPC response surface plots of PPU (a–c) and PPE (d–f) during USAE

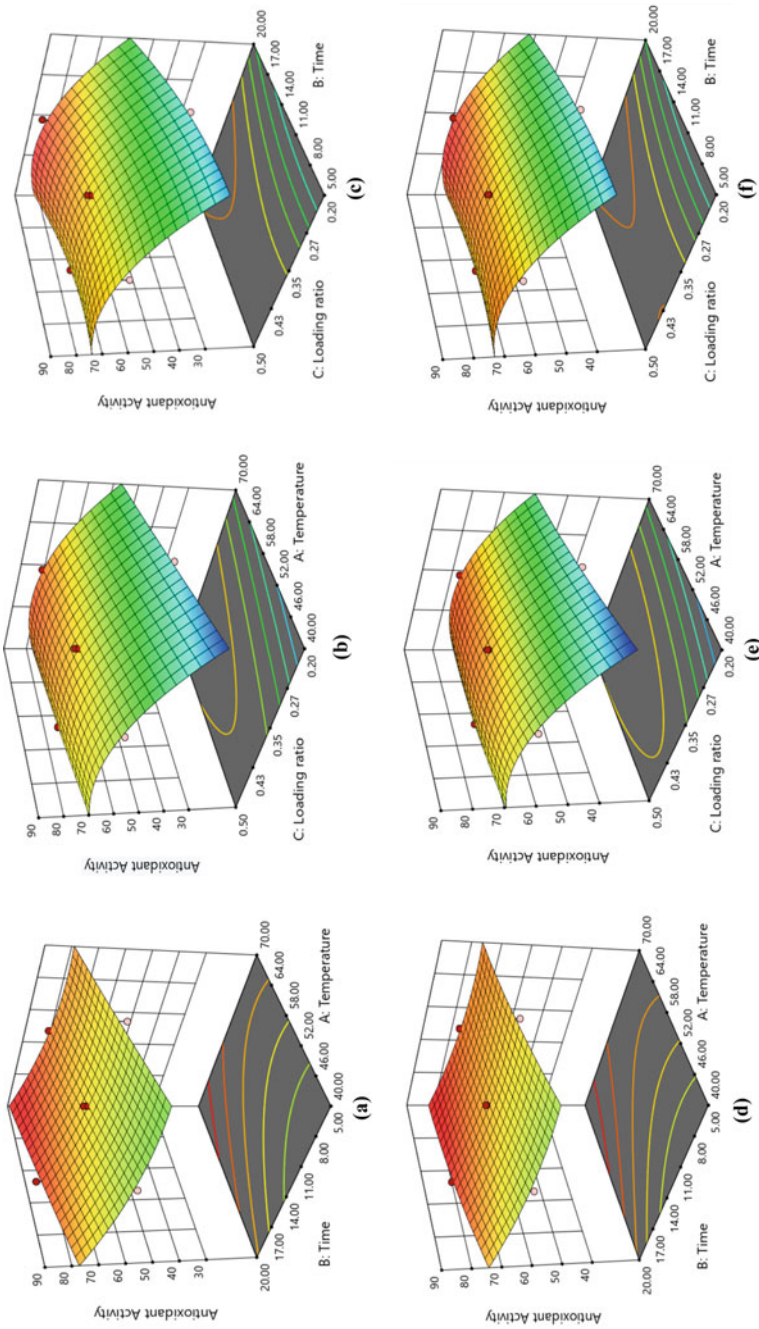


Fig. 2 ANOVA response surface plots of PPU (a-c) and PPE (d-f) during USAE

the initial alteration in SOT and SLR. Thereafter, the TFC reached a maximal value and eventually declined.

The TFC, on the other hand, varied significantly in the range of 70.67–176.08 mg QEg⁻¹ for an increment in SOT and EXT respectively in the range of 5–20 min and 40–70 °C, and for a given choice of SLR (0.2 g mL⁻¹). In this case also, a steady enhancement in the TFC yield was observed for SOT and EXT alteration. Thereafter, the TFC yield enhanced moderately.

Additionally, for a fixed SOT of 5 min, the combined effect of EXT (40–70 °C) and SLR (0.2–0.5 g mL⁻¹) varied TFC in the range of 70.67–260 mg QE g⁻¹. From the 3D RSP, it can be observed that the TFC yield increased linearly during the initial combinations of SOT and SLR. Afterwards, the TFC yield reached a maximum value and declined. Among all cases, the optimum process factors combination pertains to 0.35 g mL⁻¹ SLR, 70 °C EXT, and 20 min SOT, to achieve the highest TFC yield of 302 mg QE g⁻¹. On the TFC, the hierarchical effect of the process variables was of the order of SLR, EXT and SOT.

Impact of USAE process variables on VITAC: For alterations in SOT and SLR respectively in the range of 5–20 min and 0.2–0.5 g mL⁻¹, the VITAC significantly enhanced at 70 °C and in the range of 16.44–98.87 mg (100 g)⁻¹. The VITAC content first enhanced linearly to SOT and SLR alterations and thereafter reached a peak followed with a decline.

For a given choice of SLR of 0.2 g mL⁻¹, the VITAC, on the other hand, varied well, altering in the range of 16.44–75.23 mg (100 g)⁻¹ for a variation in SOT and EXT respectively in the range of 5–20 min and 40–70 °C. The RSP (Fig. 4a–c) affirmed linear VITAC yield increase during initial combinations of SOT and EXT. Eventually, the VITAC yield enhanced exponentially with SOT and EXT.

Furthermore, for a fixed choice of SOT of 20 min, the binary combination of EXT and SLR varied VITAC significantly in the range of 56.22–134.46 mg (100 g)⁻¹. The 3D RSP exhibited substantial VITAC yield with SLR and EXT. Thereafter, the plot affirmed a peak and subsequent sharp decline in the VITAC yield upto 0.5 g mL⁻¹ SLR.

The finest process conditions for the VITAC pertains to 0.35 g mL⁻¹ SLR, 55 °C EXT, and 12.5 min' extraction duration, at which highest VITAC yield of 134.46 mg (100 g)⁻¹ was obtained. The combination of SLR, followed by SOT and EXT, has been analysed to substantially impact on the VITAC extraction.

3.3 Optimization

For the optimisation of bioactive compounds, all independent factors were chosen for the determination of optimum values. For the PPU case, the optimum values for TPPC, VITAC and % ANOX were 148.82 mg GAEg⁻¹, 148.81 mg (100 g)⁻¹ and 87.47% for optimum process factors of 60.33 °C EXT, 19.82 min SOT and 0.40 g mL⁻¹ SLR. For the PPE case, the optimum values for TPPC, TFC and % ANOX were 138.58 mg GAEg⁻¹, 312.20 mg QE g⁻¹ and 81.10% for optimum

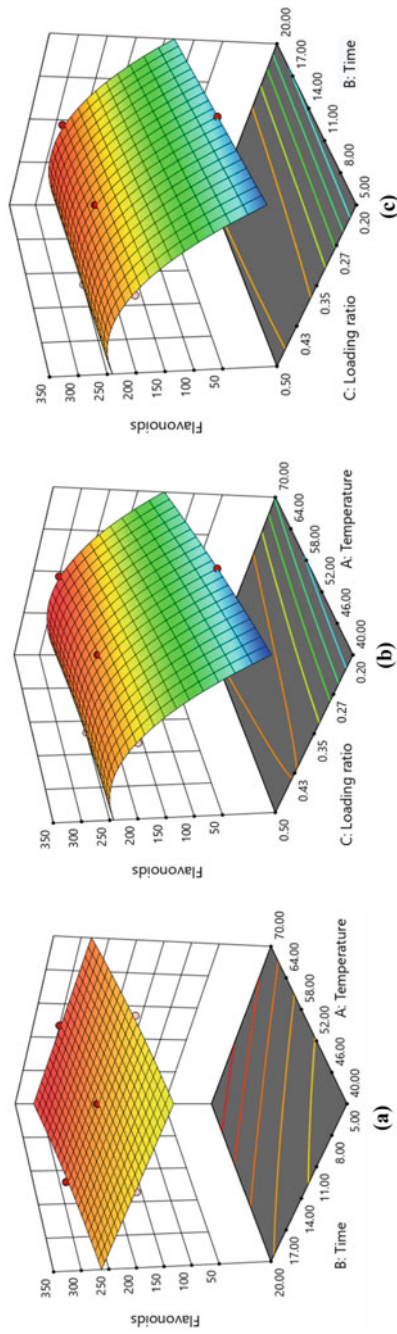


Fig. 3 TFC response surface plots of PPE (a–c) during USAE

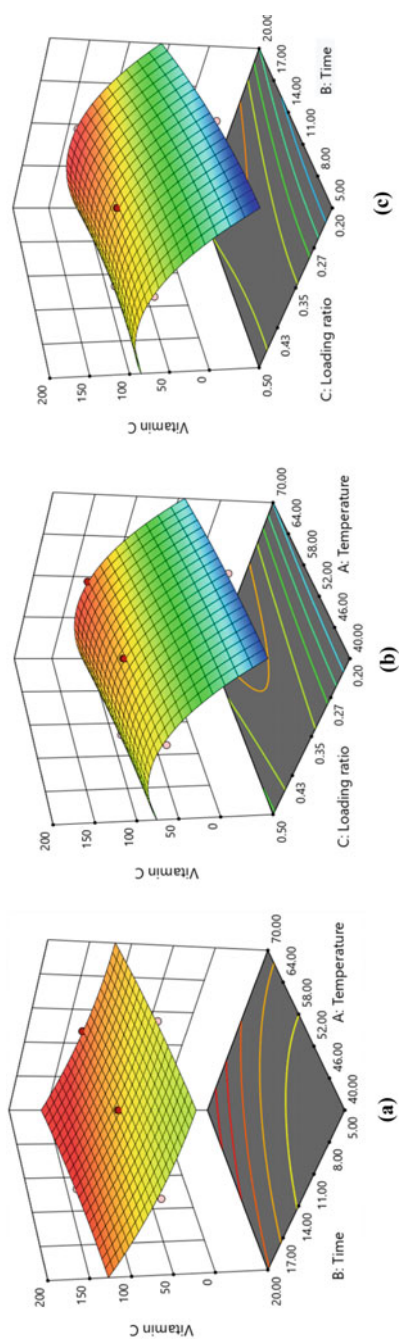


Fig. 4 VTAC response surface plots of PPU (a–c) during USAE

process condition of 66.60 °C EXT, 18.53 min SOT and 0.40 g mL⁻¹ SLR. Thus, for similar SLR, both PPU and PPE exhibited optimum extraction. Furthermore, the other independent factors, namely EXT and SOT had a variant impact on the bioactives yield for the PPU and PPE cases. This has been ascribed to various kinds of prevalent bioactive metabolites and their constitution in PPU and PPE [9]. Thereby, it can be easily analysed that the PPU has larger constitution of bioactives than PPE.

3.4 Comparative Effect of Ultrasonic Extraction Process Variables for Papaya Peel and Pulp System

A thorough analysis of the finest data for USAE of PPU and PPE confirmed that both PPU and PPE are good sources of bioactives. The USAE technique yielded higher bioactives for PPU in comparison with the PPE due to different cellular structure of PPU with respect to PPE [26]. Also, it can be affirmed from the experimental data that the SLR has been important factors in enhancing the yield of bioactives. Thereby, it has been hypothesised that the difference in concentration between the vegetable and the liquid media enabled the solute transport into the solvent [30]. Furthermore, cavitation caused by USAE has been proven to customize the onset of a number of mechanical processes, including collision of particles and breakage of the cell wall [31].

The yield patterns for both PPU and PPE exhibited distinct trends. For the PPU case, at an EXT of 55 °C, a SLR of 0.35 g mL⁻¹, and a SOT of 20 min, the USAE indicated the highest yield of TPPC, ANOX and VITAC respectively as 145.21 mg GAEg⁻¹, 85.83%, 139.5 mg (100 g)⁻¹. On the other hand, the PPE affirmed highest yield of TPPC of 126.51 mg GAE g⁻¹, and TFC of 302 mg QE g⁻¹ for 70 °C EXT, 0.35 g mL⁻¹ SLR, and 12.50 min SOT. However, the optimal ANOX of 80.36% was obtained at an EXT of 55 °C, a SLR of 0.35 g mL⁻¹, and a SOT of 20 min. The possible reason for the same could be the susceptibility of the thermolabile bioactive compounds during extreme EXT. On the other hand, a suitably larger EXT can enhance solubility of solute through the enhancement of solute's diffusion coefficient. However, even higher EXT can cause some temperature sensitive substances to deteriorate [32]. When the impact of various factors was analysed for the PPU case, it was inferred that only SLR and SOT had the greatest impact on the TPPC and VITAC yield. On the other hand, for ANOX, SLR followed by EXT had a profound effect on the response variable. Similarly, for the PPE case, SLR, EXT along with SOT, had a significant effect on the response variable. The hierarchical impact of degrees of freedom of the USAE was as per the order: SLR > EXT > SOT. In summary, it can be stated that the USAE method of sonication was better for the isolation of bioactive constituents from PPU in comparison with the PPE.

3.5 Literature Comparison

The results obtained in this work were compared with the previous work (Table 5). The study revealed that among all extraction techniques, the USAE was the most efficient. According to the literature, USAE had a greater extraction efficiency in comparison with the HWE. The ultrasound waves disrupt the cellular structure, and thereby ensure more contact between the sample and solvent and thereby enhance the release of the targeted constituents and substantially improved the extraction efficiency [21]. Further, it is well known that EXT enhancement during HWE may improve the solvent's permeability into the cell and thereby enhance the solubility of the targeted constituents into the solvent [33]. However, it may cause degradation of certain bioactive compounds and hence results in lower extraction efficiency [34]. Thus, such hypothesis may partially explain the reduced adequacy of the HWE in combination with USAE. Furthermore, it can be seen that USAE method extracted higher extraction of bioactives in the range of the PPU in comparison with the PPE. Numerous important generalizations can be deduced from the results outlined in Table 5. Notably, the finest results were obtained using USAE of PPU system, which permitted the extraction of 145.21 mg GAE g⁻¹ TPPC, 85.8% ANOX and 139.5 mg (100 g)⁻¹ VITAC at 55 °C, 20 min, and 0.35 g mL⁻¹ SLR. For the PPE, the finest values were 126.51 mg GAE g⁻¹ TPPC and 302 mg QE g⁻¹ TFC, at 70 °C, 12.5 min and 0.35 g mL⁻¹ and for ANOX, the finest values were 80.36% ANOX at 55 °C, 12.5 min and 0.35 g mL⁻¹. The sensitivity of antioxidants at extremely high temperatures could be one reason for this. Incidentally, the research carried out by the research group [9] confirmed that the MAE resulted 1154.6% lower TPPC in comparison with the finest reported USAE-PPU system data in this work. This might be because of the significantly reduced SLR of 0.016 g mL⁻¹ (2400% lower) reported in the prior art. Otherwise, it could be due to inefficient extraction procedure, including drying of the papaya sample at 50 °C and MAE [9]. Three important reasons for such large substantial variations could be the use of dried powder for extraction, the significant involvement of cavitation during ultrasound and the low levels of SLR cited in the literature. The research conducted by our research team on HWE has confirmed several explanations. For example, the HWE performed in our study at the optimised condition demonstrated much greater TPPC levels than those published previously [9]. Furthermore, one research group confirmed that the HHPE-UE yielded 11,434.8% lower TPPC and 101.08% lower VITAC concentrations, as well as 60% lower SLR [26]. It might be due to the significantly lower SLR of 0.25 g mL⁻¹ used in the study.

Considering all these into account, it can be concluded that the USAE with raw PPU and PPE systems is the most effective method for the extraction of desirable bioactive constituents rich in TPPC, TFC, VITAC and ANOX. As a result, the significance and novelty of this work has been justified. The reported prior art, according to Table 5, did not prioritise RSM-based USAE recovery of bioactives from PPU and PPE system. In addition, the impact of processing parameters on the recovery of bioactives was also not targeted in any prior art.

Table 5 Comparative summary of optimal data obtained in this study with the prior art data

Extraction process	Temperature (°C)	Time (min)	Solid to solvent ratio (g mL ⁻¹)	Total polyphenols (mg GAE g ⁻¹)	Total flavonoids (mg QE g ⁻¹)	Antioxidant activity (%)	Vitamin C (mg 100 g ⁻¹)	Literature
MAE	NA	3	0.016	11.86	0.43	3920 µmol TE/100 g	NA	[9]
HHPE-UE	NA	5-15	0.25	1.29	NA	20.6 (mM TE/100 g)	74	[26]
HWE of PPU	60.33	19.82	0.40	105	NA	76.50%	89.4	This work
HWE of PPE	66.60	18.53	0.40	88.5	145	71.5%	NA	This work
USAE of PPU	60.33	19.82	0.40	148.8	NA	87.47%	148.8	This work
USAE of PPE	66.60	18.53	0.40	138.6	312	81.1%	NA	This work

4 Conclusions

Using response surface approach, the impact of USAE process factors such as SOT, SLR, and EXT on the response variables such as TPPC, TFC, VITAC and % ANOX of PPU and PPE systems were investigated. The study confirmed that the SLR had a substantial effect on the recovery of bioactives during the USAE. In terms of SLR, EXT and SOT, the optimal conditions for the process factors and response variables differed considerably for the PPU and PPE cases. At the optimum conditions, the PPU case inferred higher TPPC and ANOX data trends than the PPE. For the PPU case, the optimum values for TPPC, VITAC and % ANOX were 148.82 mg GAE g⁻¹, 148.81 mg (100 g)⁻¹ and 87.47% at optimum process condition of 60.33 °C EXT, 19.82 min SOT and 0.40 g mL⁻¹ SLR. In addition, the findings confirmed that USAE has been efficient green extraction approach to customise maximum extraction of the bioactives in comparison with the HWE. These findings demonstrate that there is a chance to acquire new innovative technologies in the field of USAE, especially where this technology can bring several significant advantageous features and distinctive results in comparison with traditional extraction techniques. Future research should focus on optimization, predictive modelling, and scale-up integrating energy-based variables for the enhanced commercialization of the ultrasound assisted methodologies.

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Extraction of Phenolics from Yellow Passion Fruit Rind Using Supercritical Carbon Dioxide Extraction



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Abstract The passion fruit (*Passiflora edulis*) processing industry produces fruit wastes consisting of valuable compounds like phenolics, carotenoids, and flavonoids. Among these various phytochemicals, phenolics are the most common secondary metabolite in passion fruit. It contains multiple therapeutic features such as anti-inflammatory, antioxidant, and antibacterial properties. Thereby, the fruit gained popularity in several industries like medicine, food, and cosmetics. The researcher's primary concern is the cost-effective, environment-friendly extraction of chemicals from plant portions. Supercritical fluid extraction is a suitable alternative using carbon dioxide as a green solvent to recover chemicals. In this investigation, supercritical carbon dioxide extraction was used to extract phenolics from the rind powder of yellow passion fruit by using 10% ethanol as a co-solvent. The CCD model of Design Expert software was used to conduct the RSM-based optimization analysis of the extracts. The results demonstrated that the chosen independent parameters (Pressure (A), Temperature (B), and Flow rate (C)) had a significant influence on the extracted phenolic yield. The ANOVA analysis of the suggested quadratic model revealed that the model was effective (p-value <0.0001) for supercritical carbon dioxide extraction with 10% ethanol as a co-solvent. The maximum predicted yield obtained in this study was 36.87 mg GAE/g with a desirability value of 0.984. Pressure (A) 278.48 bar, temperature (B) 53.84 °C, and flow rate (C) 18.3 g/min were the optimal conditions that offered the maximal response. The study results revealed that supercritical carbon

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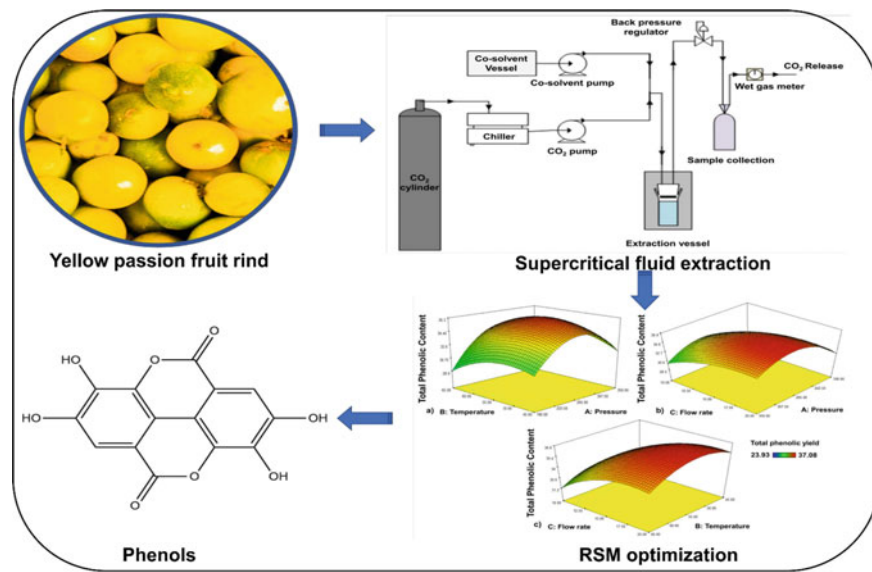
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dioxide is a green extraction technique for the extraction of phenolics from yellow passion fruit rind (YPFR).

Graphical Abstract



Keywords Ethanol · Passion fruit · Phenolics · RSM-based optimization · Supercritical carbon dioxide extraction · Yellow passion fruit rind

1 Introduction

In recent times, food waste remains as one of the very hard difficulties to be dealt. Based on the FWI (Food Waste Index) report, cited by Capanoglu, there were around 931 MT of food scraps produced in 2019 with 61% of it originating from residences, 13% from commercial and 26% from food services [1]. The food processing sector generates food waste with a high organic content in large quantities. Even though they have the potential to be repurposed in the food chain, they are mostly discarded [2]. The wastes are a plentiful source of organic bioactive chemicals that might be utilized as an ingredient for medications, cosmetics, food ingredients, and vitamins. These affordable natural food by-products open up economically appealing possibilities for their future uses. Although numerous synthetic dietary supplements are on the market, consumers have begun to reject them due to their health risks. Thus, the demand for the chemicals like phenolics, flavonoids, carotenoids, and vitamins extracted from natural sources has been rising consistently [3].

The tropical fruit passion fruit (*Passiflora edulis*) is mainly cultivated for commercial purposes in many countries. There are roughly 500 species of *Passiflora* sp., which is a member of the largest family, Passifloraceae (the family of the passion flower). This species, which is native to Brazil, is primarily found in tropical and warm temperate climates. They are infrequently found in tropical Africa, Australia, and Asia [4]. The Latin phrases “Passio” (which means “suffering”) and “floris” (which means “flower”) are the origins of the name “Passiflora”. This was found in Peru in 1569 by Spanish explorers, and missionaries in South America gave it the name because it represents Christ’s passion [5, 6]. It expands its popularity worldwide for nutritious juice production (commercially available), which has the most significant economic influence on the passion fruit industry. Around 1.5 million tonnes of passion fruit were produced globally in 2017, with Brazil being the leading grower and having great market value [7]. The juice processing industry makes a significant amount of inedible waste which is mostly discarded or utilized as fertilizer and feedstock [8]. Peel, pulp, and seed make up more than 60% of the entire amount of solid waste. [7]. These are rich in valuable chemicals such as phenolics, flavonoids, glycosides, carotenoids, vitamins, etc. Their numerous bioactivities with positive health effects, such as antioxidant capacity, anti-inflammatory activities, anti-diabetic, and anti-cancer characteristics [9, 10], are propelling up their demand scenarios on a daily basis [11]. Numerous studies conducted recently have provided evidence of the use of these phytochemicals in a variety of contexts (Table 1). Based on their bioactivity, numerous researchers demonstrated their in-vitro and in-vivo pharmaceutical applicability. Nanoparticles made from passion fruit waste were demonstrated by Nguyen affirmed both antibacterial and catalytic properties [12]. *Passiflora edulis* var. *flavicarpa* pulp’s cardioprotective properties were discovered by Soumya in an in-vitro investigation [13]. Additionally, many varieties of passion fruit have been utilized as sedatives [14], anti-diabetic agents [15], immune system boosters [16], anticancer remedies [17], and more. In the cosmetics sector, passion fruit is utilised as a moisturiser [18], antidermatophytic activity [19], anti-aging agents [20], depigmenting agent [21]. Passion fruit was used to create a variety of food products, including candy, beverages, and natural colorants. In the energy industry, passion fruit waste also produced charcoal [22], and biogas [23]. Among numerous phytochemicals, phenolics are the most typically observed secondary metabolite in passion fruit. They contain multiple therapeutic features such as antioxidant, anti-inflammatory, and antibacterial properties. Thus, the constituents in the extracted form are gaining popularity in several industries like medicine, food, and cosmetics. These plant-origin compounds protect body cells from oxidative damage caused primarily by the free radicals. This effect is due to the potent antioxidant properties of the phenolics [24].

However, the economically and environmentally feasible extraction techniques gained interest to efficiently recover targeted phytochemicals with maximum yield from various plant wastes. Several techniques for extraction (maceration, solvent extraction, soxhlet extraction, and hydrodistillation) have been used to extract active compounds present in the plant. However, these techniques are accompanied by a few drawbacks, such as a large volume of solvent consumption and low specificity [35], difficulties in releasing bound phenolics from raw materials [36, 37], and extreme

Table 1 A summary of various applications of passion fruit in alternate sectors

Sectors	Plant used	Application	References
Pharmacology	<i>P. edulis</i> peels	Catalytic activity and antibacterial properties	[12]
	<i>Passiflora edulis</i> var. <i>Flavicarpa</i> pulp	Cardioprotective	[13]
	<i>Passiflora incarnata</i> L.	Conscious sedative in dentistry	[14]
	<i>Passiflora edulis</i> Sims leaf	Anti-diabetic	[15]
	<i>P. foetida</i> fruits	Immune-enhancing activity	[16]
	<i>Passiflora mollisima</i>	Cytotoxicity	[25]
Cosmetics	<i>Passiflora edulis</i> seeds oil	Depigmenting agent	[21]
	<i>Passiflora edulis</i> f. <i>edulis</i> Sims seeds	Skin anti-aging agents	[26]
	<i>Passiflora edulis</i> var. <i>edulis</i> seed	Skin moisturizer	[18]
	<i>Passiflora foetida</i> L.	Vitamin C	[27]
	<i>Passiflora leschenaultii</i> D.C. fruits	Anti-radical and anti-diabetic	[28]
	Leaf of <i>P. caerulea</i>	Antidermatophytic activity	[19]
Food	<i>Passiflora edulis</i>	Candies	[29]
	<i>Passiflora cincinnata</i> Mast	Biopreservative	[30]
	Pericarps of passion fruits	Natural colorant	[31]
	<i>Passiflora cincinnata</i> Mast	Wine production	[32]
	<i>Passiflora edulis</i> Sims	Probiotic beverage	[33]
Energy	<i>Passiflora edulis</i> shells	Biochar	[22]
	Passion fruit seed	Antioxidant additive for biodiesel	[34]
	Peels, seeds, and shells <i>Passiflora coerulea</i> L.	Methane generation	[23]

heat, causing damage to targeted compounds (e.g., Soxhlet) [38]. Therefore, diverse solutions for the extraction of phytochemicals from bio-waste have been developed to meet the demand of a conscientious society for cost-effective and environmentally friendly approaches for sustainable production. The fundamental goal of green extraction has been to develop novel, energy-efficient extraction methods that rely on sustainable natural resources and non-hazardous substitute solvents to produce safe, high-quality extracts [39]. Ultrasound, microwave, supercritical fluid, pressurized liquid extractions, and other methods are among them which become more popular due to raising emphasis on the concentration of targeted molecules, and bioassays with high selectivity and sensitivity [40]. Among these methods, supercritical fluid extraction (SFE) is a feasible green technology [35]. In contrast to typical extraction

techniques, the supercritical solvents utilized in this procedure have various beneficial physicochemical features, such as density, viscosity, and diffusivity. Supercritical fluids' low viscosity and comparatively high diffusivity provide them with greater transport properties than liquids. Additionally, they can penetrate through solid materials more quickly, which causes extraction rates to increase. One of the major properties of a supercritical fluid is its capacity to change its density by altering its pressure and/or temperature. Since density and solubility are related, varying the extraction pressure will affect the fluid's solvent strength [39]. The high diffusivities and low viscosity make it ideal for the extraction of natural compounds like essential oils, lipids, tocopherols, and carotenoids from plants/biomass [41]. Additional advantages over traditional extraction methods include the use of GRAS solvents, increased effectiveness, and shorter extraction times [39]. Carbon dioxide (CO₂) is the most widely used solvent in supercritical fluid extraction (SFE). This is primarily due to its low cost, easy handling, and lack of harmful waste generation. Supercritical CO₂ is an ideal solvent for thermosensitive substances due to its low critical pressure (74 bar) and temperature (31 °C) [42]. The lipophilic character of CO₂, which is generally favorable for the recovery of non-polar and moderately polar chemicals, is the fundamental constraint of SFE. Small amounts of organic solvents as co-solvents like water, methanol, ethanol, etc., are frequently added to increase the capacity of solvating supercritical carbon dioxide and boost its affinity for polar molecules [2]. Several factors like co-solvent composition and quantity and extraction condition (pressure, temperature) need to be optimized to optionally extract a broader range of chemicals.

This article aims to maximize polyphenols' extraction from Passion fruit rind using supercritical CO₂. The response surface CCD model was used to optimize the factors (Temperature, Pressure, and Flow rate) for maximum extraction efficiency.

2 Experimental

2.1 Materials

The fresh yellow passion fruit (*Passiflora edulis* f. *flavicarpa*) samples were collected from the local market of Karbi Anglong, Assam, India. The fully grown, healthy fruits were washed and chopped to separate the rind and seed; and dried at 45 °C in an oven. The seeds were sealed in storage bags and stored at -20 °C. The dried rinds were grounded into fine particles and were sieved to less than 300 μm particle size with the BSS 52 mesh. The yellow passion fruit peel (YFPF) powder was stored in a refrigerator in a tightly packed Borosil glass bottle. Other reagents, i.e., absolute ethanol (Merck), sodium carbonate (Himedia), and folin-Ciocalteu (Merck), were purchased.

2.2 Optimization of Total Phenolic Content Extracted from Yellow Passion Fruit Rind

Supercritical CO₂ extraction. The extraction was performed using a Supercritical fluid extraction unit (SFE 500, Supercritical fluid Technology, Waters, USA) which contains a CO₂ pump (Waters, USA), a solvent pump (Waters, USA), an approximately 500 mL extraction cell (stainless-steel), a separation vessel, chiller (Accel 500LC, Thermo-Scientific, USA) and a pump to regulate the backpressure of the entire system (Waters, USA). Each extraction experiment entailed 20 g of powdered sample kept in the extraction cell along with a small number of glass beads to prevent the system from clogging. Ten-percent ethanol was delivered as co-solvent by a solvent pump (Waters, USA) and was mixed with supercritical CO₂ at high pressure. Prior to this mixture's entry into the extraction cell, it was heated with a heat exchanger (Waters, USA). The extractions were performed with a high-quality CO₂ mixture (10% co-solvent) flowing at a 10–20 g min⁻¹. The solvent-extract mixture was passed through the separator and depressurized to atmospheric pressure. After that, the extract was collected from the separator and kept in the refrigerator (4 °C) in airtight amber bottles for further investigation. For all trials, the extraction time was maintained at 120 min.

Experimental design. Variable levels were determined using the preliminary experimental results obtained by altering one independent factor at a time and for a fixed choice of other factors. On the basis of published literature, a range of three potential parameters have been selected for the optimization of Supercritical Carbon dioxide (SCO₂) extraction. Response Surface Methodology (RSM) based on Central Composite Design (CCD) was employed to analyze the effects of the selected parameters namely, temperature, pressure, and flow rate on the phenolic yield. Table 2 shows the experimental parameter range for SCO₂ extraction. Design-Expert® software 7.0 (Stat-Ease, inc., USA) was utilized to analyze the experimental outcomes using regression analysis.

To assess reproducibility, each experimental run was performed three times. The second-order polynomial model expresses the system that depicts the response as an independent variables' function. This is elaborated in the following mathematical expression:

Table 2 Real and coded values of the independent parameters of the SCO₂ extraction process

Variables	Codes	Coded level		
		-1	0	1
Pressure (bar)	A	180	265	350
Temperature (°C)	B	45	55	65
Flow rate (g/min)	C	10	15	20

$$Y = \beta_0 + \sum_{i=1}^n \beta_i X_i + \sum_{i=1}^n \beta_{ii} X_i^2 + \sum_{i=1, i < j}^n \sum_{j=1, i \leq j}^n \beta_{ij} X_i X_j + \epsilon \quad (1)$$

where Y —the predicted response parameter, β_0 —the constant intercept, β_i —coefficient of the linear term, β_{ii} —coefficient of the quadratic term, β_{ij} —coefficient of the interaction term, X_i and X_j —uncoded independent variable of the experiment, the symbol ϵ stands for the experimental error. Integer variables i, j , and n are used. When $i < j$ is used, β_{ij} refers to the interaction effects between X_i and X_j variables. Fisher's Satirical test (F-test) and ANOVA test with probability value ($p < 0.05$) were performed to determine the model's statistical significance. The r^2 value (regression coefficient) indicates that the model fits well with the experimental data (Fig. 1).

Determination of the Total Phenols Content (TPC) of the extracts. The Folin-Ciocalteu technique [43] was used to determine the phenolic content in the sample. A gallic acid calibration curve evaluated the total phenolic content as milligram gallic acid equivalents per gram of dried extract (mg GAE/g). In each test tube, 1 mL of tenfold diluted Folin-Ciocalteu reagent was added to an aliquot of 0.2 mL YPEP extract dissolved in Milli-Q purified water. After 5 min, 0.8 mL of 7.5% Na_2CO_3 was added, and the mixture was incubated in the dark for 30 min. The optical density was measured at 765 nm using UV-spectrophotometer (Orion Aquamate 8000, Thermo Scientific). All measurements were carried out in triplicate, and the average values were reported.

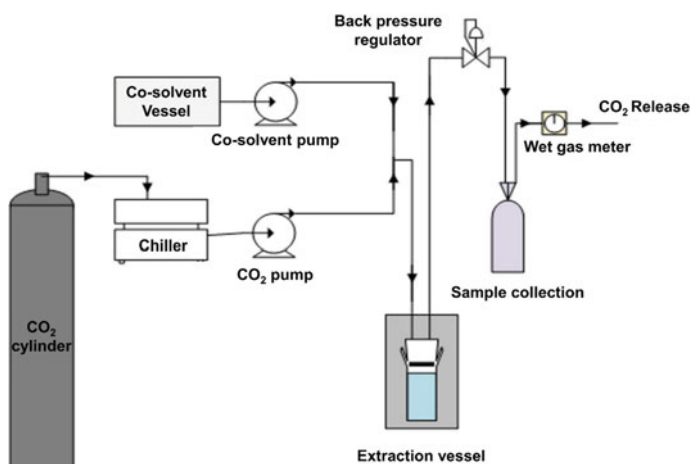


Fig. 1 Supercritical fluid extraction schematic diagram

3 Results and Discussion

3.1 ANOVA and Model Validation

The F-test of ANOVA was performed to evaluate the response of the chosen parameter on the phenolic yield using the SCO_2 -ethanol system. From the analysis, it can be deduced that the experimental and predicted phenolic yield levels were reasonably close. The significance of the model and the independent parameters were decided by p-value and F-value. The bigger the magnitude of the F-value, the lower the “Prob > F” value and hence more significant will be the analogous variable. The ANOVA analysis (Table 3) of the predicted quadratic model indicated that the model was highly significant with a p-value less than 0.0001. Therefore, this model could be used for the optimization of the extraction process. Among the independent variables, flow rate had the most significant (p-value < 0.0001) effect on the extraction yield with an F-value of 224.49, followed by pressure and temperature with F-values of 74.65 and 9.36 respectively. All quadratic terms of the process variables pressure (A^2), temperature (B^2), and flow rate (C^2) were significant and had a p-value less than 0.001. In contrast, the probability value for interaction between pressure and temperature was 0.0008. The reliability of the model and the experimental results were tested with a lack of fit test (F-value of 0.33 and p-value of 0.88) that indicated there was no proof that data was not fitted to experimental data (Table 4). The statistical model was a good fit as the adjusted regression coefficient ($r_{\text{adj}}^2 = 0.99$) was relatively close to the regression coefficient ($r^2 = 0.99$). The predicted coefficient of regression value ($r_{\text{pred}}^2 = 0.98$) demonstrated that the model could appropriately rely on the expected response range with its associated independent variable. A relatively low coefficient of variation (CV = 1.41%) was the precision and reliability of the experiment. The quadratic model came up with polynomial equations, where Eq. 2 denotes coded, and Eq. 3 represents actual factors.

$$\begin{aligned} \text{Totalphenolcontent (coded)} = & 36.16 + 1.05A - 0.37B + 1.82C + 0.76AB \\ & + 0.26AC - 0.15BC - 3.58A^2 - 1.50B^2 - 1.42C^2 \end{aligned} \quad (2)$$

$$\begin{aligned} \text{Totalphenolcontent (actualfactors)} = & -50.64 + 0.22x + 1.43y + 2.07z + 0.0009xy \\ & + 0.0006xz - 0.003yz - 0.0005x^2 - 0.015y^2 - 0.057z^2 \end{aligned} \quad (3)$$

In coded terms, A (pressure (bar)), B (temperature ($^{\circ}\text{C}$)), and C (flow rate (g/min)) were parameters. x (pressure (bar)), y (temperature ($^{\circ}\text{C}$)), and z (flow rate (g/min)) were all expressed in terms of actual parameters.

Table 3 ANOVA table for the best fit quadratic model to achieve optimal phenolic yield

Source	Sum of squares	DF	Mean square	F value	P-value Prob > F	
Model	284.80	9	31.64	157.37	<0.0001	Significant
A-Pressure	15.01	1	15.01	74.65	<0.0001	
B-Temperature	1.88	1	1.88	9.36	0.0121	
C-Flow rate	45.14	1	45.14	224.49	<0.0001	
AB	4.56	1	4.56	22.69	0.0008	
AC	0.56	1	0.56	2.79	0.1257	
BC	0.19	1	0.19	0.93	0.3564	
A ²	184.75	1	184.75	918.77	<0.0001	
B ²	32.50	1	32.50	161.63	<0.0001	
C ²	29.10	1	29.10	144.70	<0.0001	
Residual	2.01	10	0.20			
Lack of fit	0.50	5	0.099	0.33	0.8774	Not significant
Pure error	1.52	5	0.30			
Cor total	286.81	19				

3.2 Interaction Effects of the Independent Variable on the Phenolic Yield

The interaction between the independent and dependent factors on the phenolic output was demonstrated by the graphical representation of the regression equation in a 3D contour plot (Fig. 2). The individual parameters and their quadratic terms significantly affect the phenolic output with a p-value less than 0.0001. Figure 2a displayed the influence of temperature and pressure on the response (TPC output). The interaction of pressure and temperature substantially influenced the rate of extraction with a probability value of <0.001. The phenolic output increased as the pressure rapidly increased up to 270 bar and thereafter gradually decreased. With increasing pressure, the viscosity of supercritical carbon dioxide decreases at static temperature and as well increases the solubility of the supercritical carbon dioxide. Reduced viscosity decreases the intermolecular distances, thus increasing the interaction between analytes and carbon dioxide and enhancing the extraction yield [44]. In contrast, a further pressure increase beyond 270 bar caused a rapid reduction in phenolic output. As reported by Bimakr et al. [45], the diffusion rate of solutes reduced at the higher pressure is related to the supercritical fluid medium.

Figure 2b exhibits the 3D response graphic of the combined effect of pressure and flow rate. The yield increased up to the temperature of 55 °C; further, increase in the temperature, affirmed no significant improvement. Upon increasing the rate of flow of carbon dioxide from 10 to 20 g/min, the response output increased up to 15 g/min before stabilizing. This could be due to the high flow rate that drives

Table 4 Experimental data obtained from CCD design approach for the phenolic extraction using SCF process

Sl. No.	Pressure (bar)	Temperature (°C)	Flow rate (g/min)	Total phenolic content (mg GAE/g)
1	180	45	10	28.19 ± 1.06
2	350	45	10	27.94 ± 0.15
3	180	65	10	26.09 ± 0.90
4	350	65	10	28.95 ± 0.11
5	180	45	20	31.7 ± 1.36
6	350	45	20	32.6 ± 0.22
7	180	65	20	29.07 ± 1.24
8	350	65	20	32.91 ± 1.00
9	122.05	55	15	23.93 ± 0.22
10	407.95	55	15	28.08 ± 1.84
11	265	38.18	15	32.37 ± 0.36
12	265	71.82	15	31.39 ± 1.49
13	265	55	6.59	29.22 ± 1.04
14	265	55	23.41	35.00 ± 0.09
15	265	55	15	35.42 ± 0.85
16	265	55	15	36.15 ± 0.24
17	265	55	15	36.35 ± 0.57
18	265	55	15	35.86 ± 0.64
19	265	55	15	37.08 ± 0.55
20	265	55	15	36.12 ± 0.11

the solvent through the sample, that facilitates only passage around the matrix of the sample and not through the pores. Thereby, it prevented carbon dioxide from moving in and out of the sample. Due to intra-particle diffusion resistance, low rates increase the analytes' trapping ability and improve extraction efficiency. A low flow rate promotes mass transfer, extending the contact time between the solvent and the solutes of interest. As a result, the fluid's sluggish movement allows for deeper intrusion into the solute matrix while also lowering the linear velocity. This resulted in excellent efficiency of extraction [44].

Figure 2c conveyed the combined impact of temperature and flow rate on the response and for the constant pressure of the system. During the initial phase, the response yield increased rapidly with the flow rate, and thereafter reduced marginally. The effect of temperature on the extraction yield had a synonymous influence with pressure. The output increased upto an increase in the temperature of 54 °C. Thereafter, the phenolic yield was reduced due to the thermos-sensitivity of extracted compounds. Also, at constant pressure, increasing temperature decreases the solvent strength by lowering the density of carbon dioxide. Near the critical pressure, the

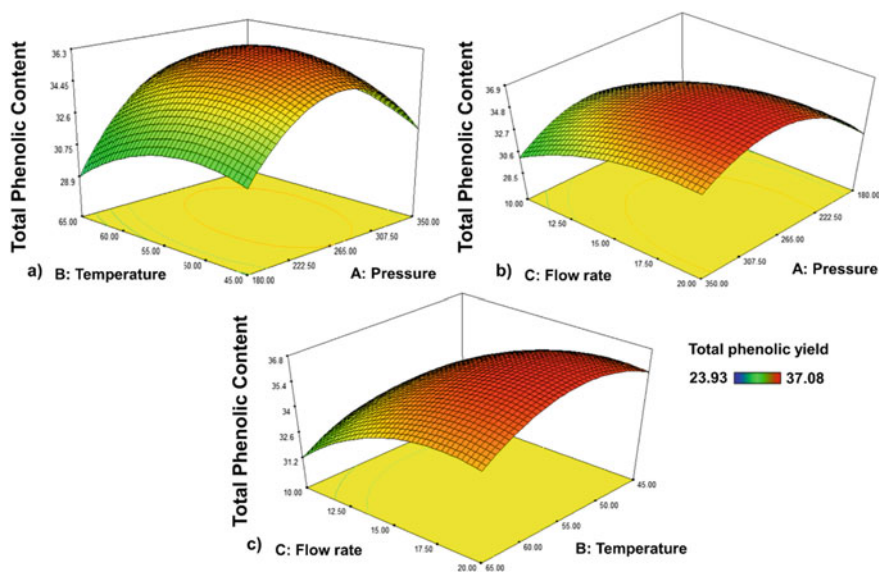


Fig. 2 3D Response surface plots of total phenolic yield with respect to variations in **a** Pressure (bar) and temperature ($^{\circ}\text{C}$); **b** Pressure (bar) and flow rate (g/min); and **c** temperature ($^{\circ}\text{C}$) and flow rate (g/min)

fluid density depends on the temperature. Therefore, the extraction yield fluctuated over a temperature range of 45–65 $^{\circ}\text{C}$. A moderate increase in temperature might cause a significantly lowered solubility of the solvent through a reduction in the density. An increasing temperature improves the mass transfer from solute to solvent and increases extraction efficiency [45].

3.3 Optimization of Process Variables

Design-Expert software's desirability function was utilized to forecast the optimal condition for achieving maximal phenolic yield within selected parameters. The predicted maximum yield was 36.87 mg GAE/g, with a desirability value of 0.984. The desirability profile to achieve maximum phenolic yield with Supercritical carbon dioxide was found at 278.48 bar, 53.84 $^{\circ}\text{C}$ temperature, and at 18.3 g/min of flow rate with 10% ethanol co-solvent (Table 5). The experiment performed at the recommended condition resulted in 36.04 mg GAE/g, and had a 2.1% deviation from the RSM predicted value. Thus, it could be stated that the suggested quadratic model could explain the phenolic yield within the chosen parameter range. Pereira found a TPC of 119 mg GAE/L in the passion fruit rind extracts, extracted with 70% ethanol as solvent by ultrasound-assisted pressurized liquid extraction at 60 $^{\circ}\text{C}$ [46]. Asimwe

Table 5 Optimized process parametric summary table for phenolic compounds extraction with SCO_2 and 10% ethanol co-solvent

Solvent	Pressure (bar)	Temperature ($^{\circ}\text{C}$)	Flow rate (g/min)	Total phenolic content (mg GAE/g)	Desirability
Supercritical carbon dioxide	278.48	53.84	18.3	36.87	0.984

extracted phenolics from purple passion fruit pulp using 80% methanol in the ultrasonic method. They observed that the TPC value of the passion fruit pulp-CMC mixture was 351.9 mg/100 g solids GAE [47]. A study on extraction of value-added compounds from two varieties of passion fruit, namely, *Passiflora edulis* Sims and *Passiflora cincinnata* Mast, by liquid–liquid extraction, using ethyl acetate and for 5 min agitation. Thereby, the total phenolic contents are 476.1 and 365 mg/kg GAE, respectively [48]. Traditional techniques such as MAE are expected to provide a lower recovery of target compounds. The low extraction efficiency of these techniques is the primary reason for the same. It has been proven that new extraction techniques are more sustainable, fast, and efficient, and due to these reasons, they yield higher phenolic content in the extract [46] (Table 6).

Table 6 Literature reported data summary of passion fruit extracted total phenolic content

Variety used	Solvent and technique used	Total phenolic content	References
<i>Passiflora edulis</i> sp. rind	70% ethanol; UAPLE	119 mg GAE/L extract	[46]
Purple passion fruit pulp	80% methanol; UAE	351.9 mg/100 g solids GAE	[47]
<i>Passiflora edulis</i> Sims ^a and <i>Passiflora cincinnata</i> Mast ^b	Ethyl acetate; liquid–liquid extraction	476.1 mg kg^{-1} GAE ^a and 365 mg kg^{-1} GAE ^b	[48]
<i>Passiflora edulis</i> f. <i>edulis</i> Sims. seed	Acidified ethanol (96%), ultrasonic-assisted maceration	0.32 g GA/g extract	[20]
<i>Passiflora edulis</i> var. <i>flavicarpa</i> seed	Methanol extraction; Soxhlet extraction	74.9 mgGAE/g	[34]
Passion fruit seed cake	CO_2 ; SFE	17.9 mgGAE/g extract	[49]
Passion fruit peel	70% ethanol; UE	382.86–428.71 μg GAE/g DM	[50]

^a*Passiflora edulis* Sims

^b*Passiflora cincinnata* Mast

4 Conclusions

SFE demonstrated a sustainable technology for the extraction of phenolics from the yellow passion fruit rind (YPFR) using carbon dioxide as a renewable green solvent. Ten-percent ethanol has been used as a solvent modifier to extract the polar compounds. According to the RSM analysis, the chosen process parameters (temperature, pressure, and flow rate) significantly impacted the phenolic output. The solvent flow rate primarily affects the outcome, followed by pressure and temperature. The RSM approach yielded the following ideal settings for maximizing phenolic content: 278.48 bar (treatment pressure), 53.84 °C (temperature), and 18.3 g/min (flow rate) for SCO₂ with 10% ethanol as a co-solvent. With desirability values of 0.984, the estimated yield was 36.87 mg GAE/g. Thus, plant waste like passion fruit rind may introduce new natural food products in the market that are healthier and prevent oxidative damage that commonly occurs in our bodies due to the processing of the food.

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Nutritional Efficacy Based Vegetables Selection for the Development of Ready to Cook Soup Mix Formulations



Kumudhini Akasapu  and Ramagopal V. S. Uppaluri 

Abstract Vegetables are a good source of nutrients and after appropriate processing, they exhibit a great potential towards the development of ready-to-cook (RTC) food products. Among various RTC foods, vegetable soup mixes are gaining a fair demand due to their ease of preparation, consumption and acceptance by wider consumer groups. Thus, effective utilization of vegetable produce will help in the development of nutritionally rich soup mix formulations. In this regard, a holistic characterization of twelve abundantly produced Indian vegetables such as gourds and beans to identify their potentiality towards the development of RTC soup mixes was carried out. For this, sensitivity of moisture content, yield and important nutritional characteristics such as antioxidant activity, total phenolic content, vitamin C, soluble protein and crude fiber in due course of heat treatments namely hot water blanching and tray drying was investigated. Initially, vegetables were dried for 12 h and the moisture content and antioxidant activity were evaluated at 1 h intervals from 6th hour onwards to infer upon the optimal drying time. Further, other nutritional parameters were evaluated for all the vegetables treated at their corresponding optimal drying time. From the findings, it was observed that among all the summer and winter vegetables, pointed gourd; spine gourd; yardlong bean and ash gourd; bitter gourd; bottle gourd possessed highest nutrients respectively.

Keywords Antioxidant activity · Blanching · Drying · Moisture content · Nutrition · Vegetable soup mix

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Abbreviations

F	Fresh
B	Blanching
D	Drying
BD	Blanched and dried
MC	Moisture content
AA	Antioxidant activity
CVITC	Vitamin
TPC	Total phenolic content
WB	Wet basis
DM	Dry matter
AG	Ash gourd
BS	Beans
BIG	Bitter gourd
BOG	Bottle gourd
P	Pumpkin
S	Squash
PG	Pointed gourd
RG	Ridge gourd
SG	Snake gourd
SPIG	Spine gourd
SPOG	Sponge gourd
YB	Yardlong bean

Highlights

- Pointed gourd, spine gourd and yardlong bean of summer possessed maximum nutrients
- Ash gourd, bitter gourd and bottle gourd of winter possessed maximum nutrients
- Tray drying was found to be effective in comparison with blanching followed by drying
- Lowest processing time was obtained for pointed gourd, bitter gourd and spine gourd

1 Introduction

Vegetable based ready to cook food products such as vegetable soup mixes are gaining a fair demand due to their ease of preparation, easy of consumption and acceptance by wider consumer groups. The major ingredients in ready to cook vegetable soup mixes are dehydrated vegetables, thickening agents and spices. Among these, dehydrated

vegetables offer major proportions of nutrients supply to the consumer. Currently, there are few commercially available soup mixes constituted with vegetables such as carrots, peas and leeks. These vegetables are typical produces of Western landscape but not India. Thus, there is a great scope to explore the potential of abundant Indian produce such as gourd and bean varieties towards the development of such vegetable soup mixes. Also, there are only few commercial soup products developed with such produce (Pumpkin and Bottle gourd) and soup mixes developed with major constitution of such abundant Indian produces will provide adequate nutrients as these vegetables are known for their promising nutritional constituents such as carbohydrates, proteins, vitamins, minerals, dietary fiber, antioxidants, and phytonutrients [10]. Also, in most cases involving dehydrated or ready to cook product development, sensory optimization followed by nutritional characterization is being followed. However, this approach may not always help in achieving a nutritionally rich dehydrated product as nutritional optimality of each vegetable is ignored. Thus, it is important to carry out an initial screening of the vegetables through holistic nutritional characterization in the due course of obtaining dehydrated vegetables. Hence, it is important to carry out holistic studies in a systematic manner to determine the influence of various heat treatments on the important nutritional characteristics of vegetables.

Several drying techniques such as sun drying, oven drying, tray drying, fluidized bed drying etc., exist for the preservation of vegetable produce through dehydration. Among these, tray dryers have the edge as being the most affordable and simple drying technology for the production of nutritionally rich dried vegetables in a short span of time [21, 32]. Also, blanching as a pre-processing technique can substantially influence the nutritional, drying rate, color and flavor characteristics of the dehydrated vegetables. Often, prior to drying, blanching is carried out to inactivate peroxidase enzyme and prevent the generation of off flavored and unaccepted colored substances due to oxidation. For vegetables such as bitter gourd, blanching plays a vital role to reduce its bitterness and thereby promote its sensory characteristics. Among alternate blanching methods, hot water blanching is the most inexpensive and easy process.

Due to variant constitution of water content and other ingredients, each vegetable has its own optimal combination of drying time and temperature to retain nutrition and yet achieve good shelf-life parameters of the dried product. Addressing these issues for cucurbits, beans and few other vegetables, the available prior art provided only few insights with respect to the role of blanching, drying [21, 32] and their combination [24, 28] as well as their optimal process parametric choice towards the production of nutritionally rich optimally dried vegetables. Despite gaining few useful insights from the available prior art, a great sparsity in terms of holistic characterization of vegetables has been observed as the influence of processing treatment on very few nutritional characteristics has been studied. For example, for bitter gourd ring samples, [32] inferred that among solar, cabinet and low temperature drying methods, the cabinet drying operated at 58–60 °C and provided the highest nutritional retention in terms of ascorbic acid (57.34 mg/100 g), total carotenoids (2.11 mg/100 g) and β -carotene (0.86 mg/100 g). For yardlong bean and pumpkin, [33] carried out investigations using sun, oven and freeze drying processes for the

evaluation of their dietary fiber content. Among all processes, the oven drying process affirmed the highest dietary fiber content of 28 and 10.5% for the bean and pumpkin samples respectively. However, the blanching pre-process investigation and its critical influence on the dried vegetable nutritional parameters and characterization of other nutritional parameters were not considered by the authors. Such approaches may not help in the appropriate selection of combinations of vegetables based on their processing and nutritional parameters in the due course of the development of nutritionally rich soup mixes. This is due to a scarcity in the comprehensive methodology and associated studies.

Considering the above cited lacunae, the article targeted a systematic holistic approach where the sensitivity of important nutritional characteristics such as antioxidant activity, total phenols, soluble protein, vitamin C and crude fiber of twelve seasonal and abundantly produced Indian vegetables towards the heat treatments namely blanching, tray drying and their combination have been addressed. The chosen summer vegetables include *Trichosanthes dioica* (Pointed gourd, PG), *Luffa acutangular* (Ridge gourd, RG), *Trichosanthes cucumerina* (Snake gourd, SG), *Momordica dioica* (Spine gourd, SPIG), *Luffa aegyptiaca* (Sponge gourd, SPOG) and *Vigna unguiculata* (Yardlong bean, YB). Similarly, the winter vegetables refer to *Benincasa hispida* (Ash gourd, AG), *Phaseolus vulgaris* (Beans, BS), *Momordica charantia* (Bitter melon, BIG), *Lagenaria siceraria* (Bottle gourd, BOG), *Cucurbita moschata* (Pumpkin, P) and *Sechium edule* (Squash, S). Having characterized the vegetables potentiality, this systematic study will provide insights for further studies in choosing the best combination of vegetables for the development of nutritionally rich soup mixes. Also, most of these chosen vegetables have been explored for their use in Naturopathy and Ayurveda and very few such as bottle gourd and pumpkin received commercial interest in terms of ready to cook soups. Despite their availability in the market, existing pumpkin and bottle gourd soups have been observed to be constituted with various preserving agents and are as well expensive. Also, these vegetables have not yet been explored for their utility in mixed vegetable soup formulations. However, their promising nutritional and sensory properties enable the scope for further exploration of these vegetables towards the development of affordable mixed vegetable soup formulations. Thus, the chosen nutritionally rich abundant vegetables have been anticipated to provide great scope towards nutritionally and taste rich ready to cook vegetable soup mixes.

2 Materials and Methods

2.1 Raw Materials

Fresh vegetables chosen for this work were procured from the vegetable store in the market complex of Indian Institute of Technology Guwahati. All vegetables were purchased fresh and were carried in tight polythene bags to avoid

any external contamination during transportation. Fresh vegetables were then immediately processed for the estimation of nutritional components.

2.2 Chemicals

Absolute methanol, Bradford's reagent, oxalic acid dihydrate, sodium hydroxide pellets, and sulfuric acid (98%) were purchased from Merck India. 2,2-Diphenyl-1-Picrylhydrazyl (DPPH) extra pure, Folin Ciocalteus Reagent and 2,6-dichlorophenol indophenol sodium salt (DCPIP) were procured from SRL Pvt. Ltd., India. Sodium bicarbonate was purchased from Rankem, India.

2.3 Sample Preparation

After procuring, the vegetables were subjected to water washing for the removal of adhering dirt and dust particles. A clean cloth was used to remove excess moisture adhering to the vegetable surfaces. All the vegetables were peeled prior to slicing. Also, P and S were deseeded after removing the peel. Except P and S, all gourd varieties were sliced into 2 mm thick circular slices. The P and S were cut into 1 mm thick rectangular slices. Further, the blanched samples were prepared by contacting the peeled vegetable samples with hot water at 95 °C for 4 min. Blanching was targeted to retain maximum content of nutrients as well as to inactivate polyphenol oxidase, peroxidase and lipoxygenase enzymes that exist in the vegetables [1, 17, 19, 29, 38]. Fresh samples were also prepared for the experimental investigations. All samples (blanched and fresh) were kept on an aluminum tray (one layer) for the onset of the drying process. Upon the completion of the drying process, powdered samples were stored in airtight pouches and were placed in a desiccator for the immediate estimation of nutritional components.

2.4 Drying of Vegetables

A laboratory scale tray dryer (Make: ICT; Model: QT) was used for the drying of vegetables. The vegetables were dried at a temperature of 60 °C. However, the corresponding drying time was varied from 6 to 12 h. An intermittent airflow drying technique was followed to ensure effective drying and retention of nutrients. An intermittent air flow velocity of 2 m/s was maintained in the tray dryer system along with 20 s run/off mode setting that ensures periodic air flow and stagnation. The drying parameters namely drying temperature and drying time were set based on the findings from the available literature [16, 21].

2.5 *Characterization of Moisture Content, Yield and Nutritional Components*

The fresh and tray dried vegetable moisture content was determined using a prior art method [8]. The yield of dried samples was determined by following the literature reported method [21]. Soluble protein content was estimated by following literature summarized procedure [21]. The fresh and tray dried vegetables crude fiber content was evaluated using the method outlined in literature [27]. Soluble protein content was estimated by following the literature reported method [21]. Vitamin C content was estimated by 2,6-dichlorophenol indophenol dye titration method and L-ascorbic acid was used as a standard [21, 27]. Folin-Ciocalteu phenol reagent procedure was followed to estimate the total phenol content [37].

Antioxidant activity was determined by following the literature summarized procedure [21]. Accordingly, 0.1 g of the dried sample was extracted with absolute methanol (20 mL) for 30 min in a sonicator (model: Elmasonic S 30H, make: Elma). For the fresh vegetable, 1 g of sample was considered. 1 mL of the solution obtained after filtration with Whatman no.1 filter paper was mixed with 3 mL of 0.002% methanolic DPPH solution. A control sample was prepared as a mixture of 1 mL absolute methanol and 3 mL methanolic DPPH solution. After thoroughly mixing with a vortex, the samples were incubated for 30 min in a dark chamber. Finally, absorbance at 517 nm was measured for control and desired samples using a UV spectrophotometer (Make: SHIMADZU, Model: UV-2600). Using these measured values, the antioxidant activity was determined using the equation:

$$AA (\%) = \frac{A_c - A_s}{A_c} \times 100 \quad (1)$$

2.6 *Evaluation of Optimal Drying Time*

The article considers moisture content and antioxidant activity of dried/blanched/blanched and dried vegetable samples as the desired critical responses. The study delineates upon the research methodology that involves optimal drying time identification based on highest antioxidant activity of the dried sample being achieved for a moisture content less than 8% on a wet basis. The methodology was chosen based on the general guidelines for dried vegetables [26]. Figures 1 and 2 illustrate the methodology associated to the evaluation of optimal drying time.

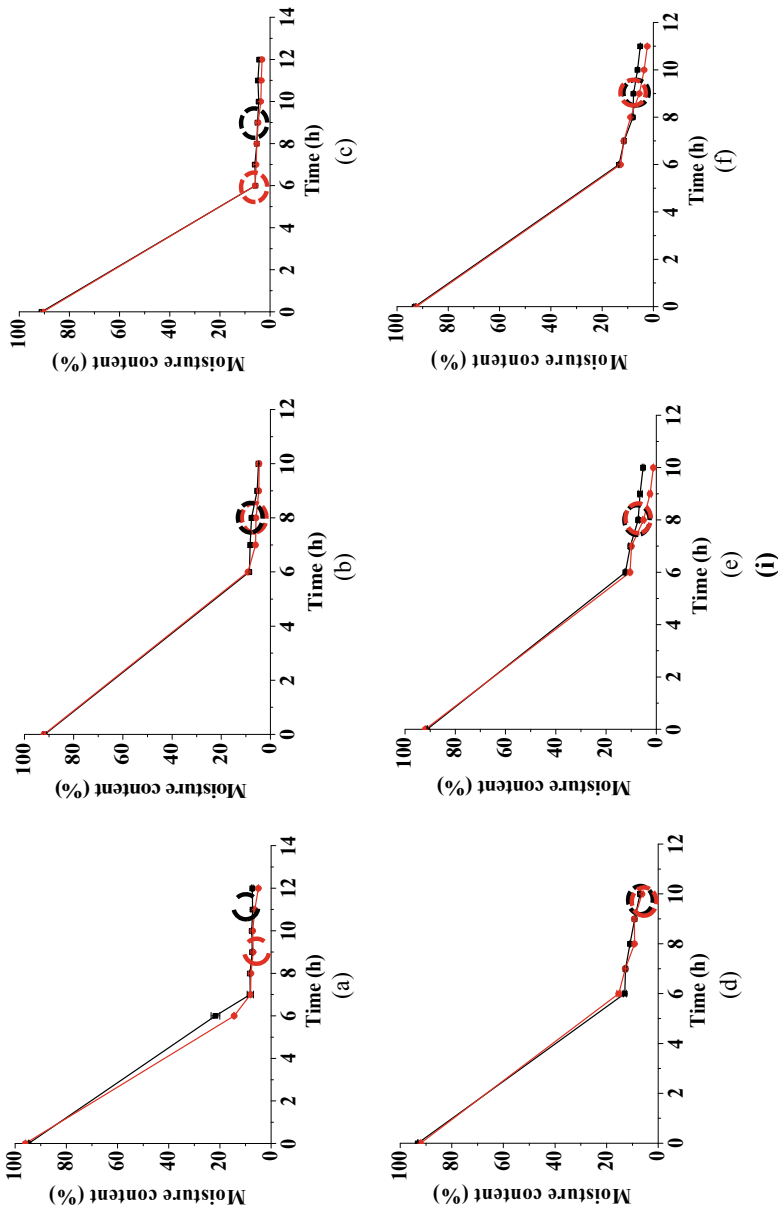


Fig. 1 Optimal drying time of dried (■) and blanched dried (●) winter vegetables w.r.t. (i) Moisture content of **a** ash gourd **b** beans **c** bitter gourd **d** bottle gourd **e** pumpkin **f** squash (ii) antioxidant activity of **a** ash gourd **b** bitter gourd **d** bottle gourd **e** pumpkin **f** squash

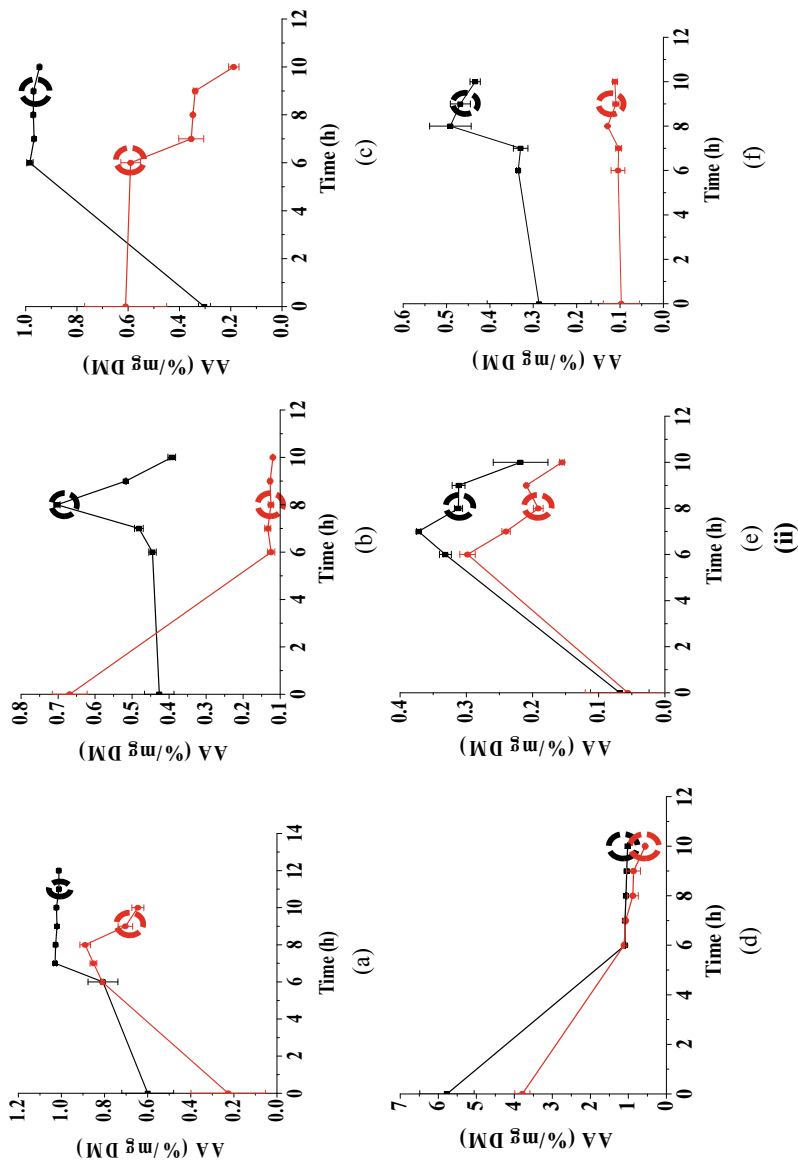


Fig. 1 (continued)

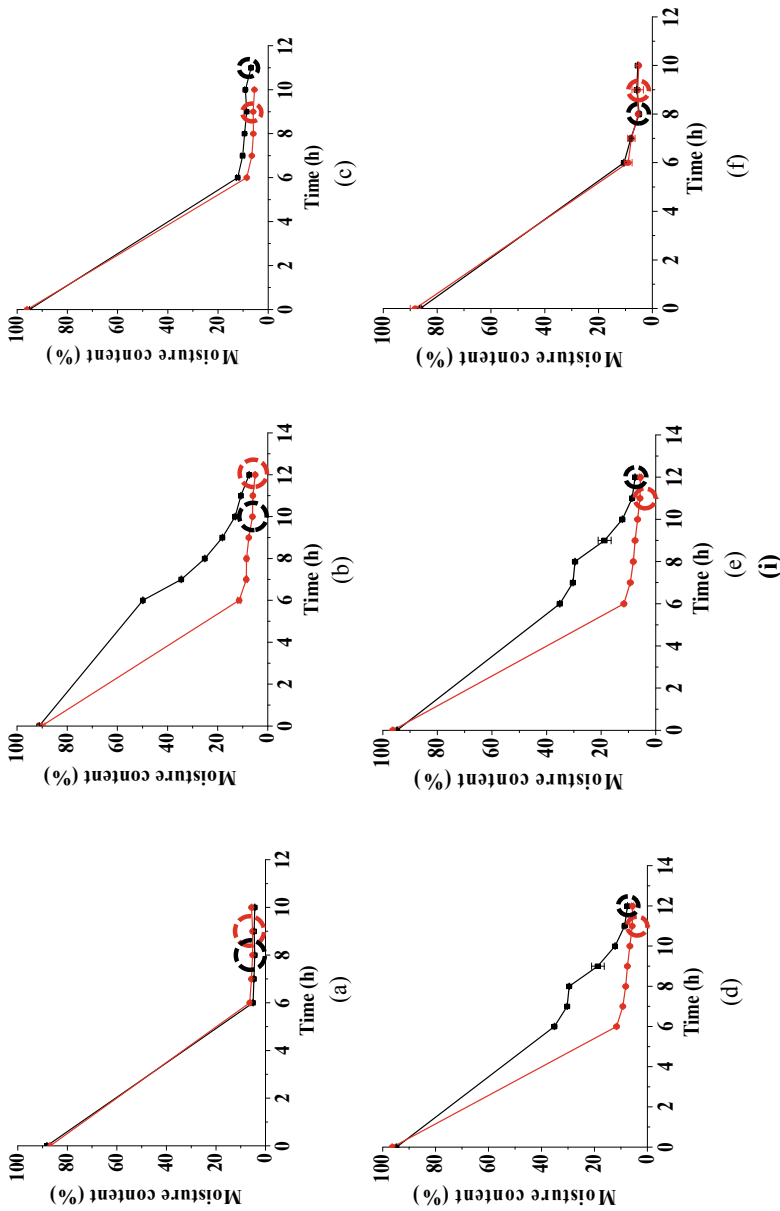


Fig. 2 Optimal drying time of dried (■) and blanched dried (●) summer vegetables w.r.t. (i) Moisture content of **a** pointed gourd **b** ridge gourd **c** snake gourd **d** spine gourd **e** sponge gourd **f** yardlong bean (ii) antioxidant activity of **a** pointed gourd **b** ridge gourd **c** snake gourd **d** spine gourd **e** sponge gourd **f** yardlong bean vegetables

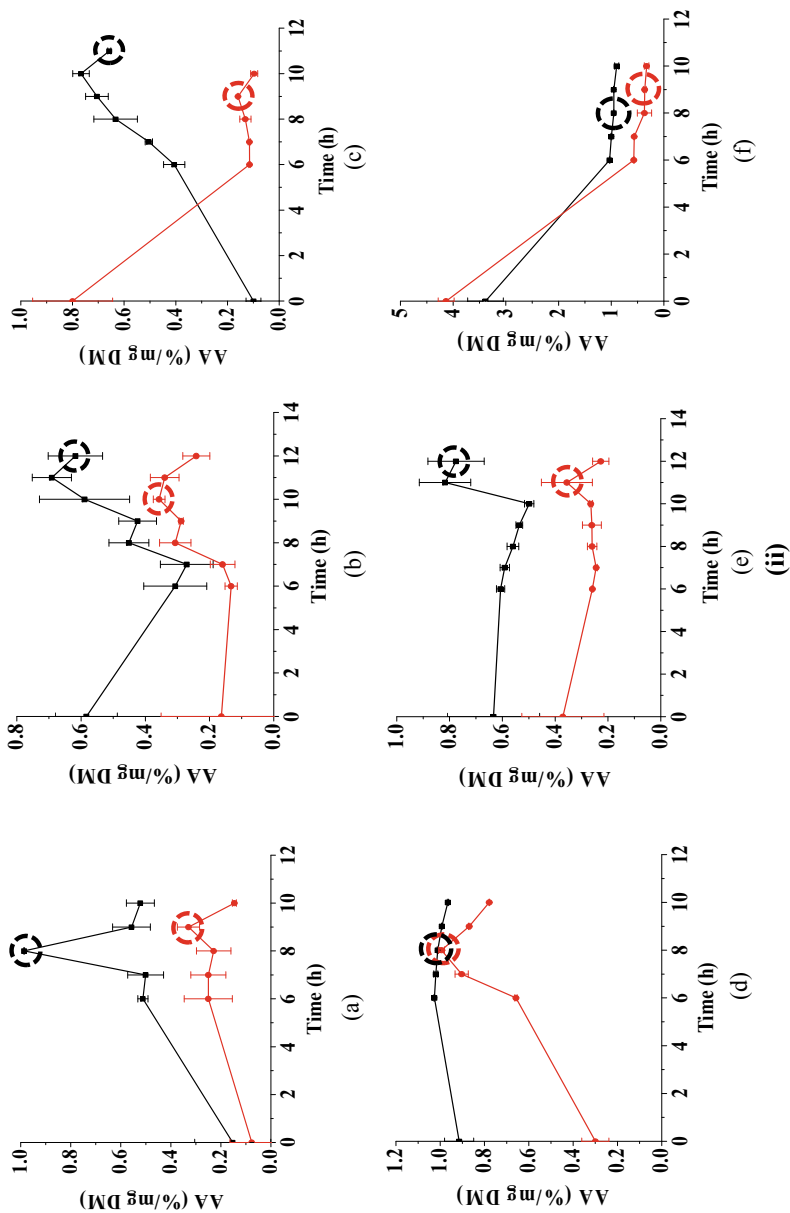


Fig. 2 (continued)

2.7 Statistical Analysis

All the experiments were carried out in triplicates. SPSS (IBM statistical analysis version 25) based ANOVA was used to perform the statistical analysis of the results. Post-hoc comparisons were carried out at $p < 0.05$ to determine the significance among the samples.

3 Results and Discussion

Table 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11 and 12 summarize the moisture, yield and nutritional parameters of fresh and optimally treated AG, BIG, BOG, BS, P, S, PG, SPIG, YB, RG, SG and SPOG respectively. As wet basis is not a standard norm to compare and contrast vegetables with variant moisture content in due course of drying, all relevant nutritional parameters have been mentioned with respect to the dry basis. However, this was not the case in the available prior art and hence for literature comparison purposes, both wet basis and dry mass basis values have been provided.

3.1 Fresh Vegetable Parameters

Among winter vegetables, AG possessed highest moisture content (94.74%) and P possessed lowest moisture content (91.20%). Among summer vegetables, SG and YB possessed highest (95.12%) and lowest (86.28%) moisture content respectively.

Among winter vegetables, BOG and P possessed highest (5.77%/mg) and lowest (0.07%/mg) antioxidant activity respectively. Among summer vegetables, highest and lowest antioxidant activity was observed for YB (3.39%/mg) and PG (0.15%/mg). Among winter vegetables, highest and lowest TPC was observed for BOG (722.02 mg/g) and S (163.90 mg/g). Among summer vegetables, YB (492.42 mg/g) and SG (135.90 mg/g) possessed highest and lowest TPC respectively. For the winter vegetables, BIG (674.60 mg/100 g) and S (78.43 mg/100 g) possessed highest and lowest VITC content respectively. Among summer vegetables, SPIG (572.52 mg/100 g) and PG (68.84 mg/100 g) possessed maximum and minimum VITC content correspondingly.

Among winter vegetables, AG (97.77%) and P (89.18%) possessed highest and lowest crude fiber content respectively. Similarly, among summer vegetables, YB (97.02%) and RG (92.06%) possessed highest and lowest crude fiber content respectively. Among winter vegetables, BS (28.26 mg/g) and P (15.00 mg/g) possessed highest and lowest soluble protein content respectively. Similarly, among the summer vegetables, SPOG (25.85 mg/g) and SPIG (11.93 mg/g) possessed highest and lowest soluble protein content respectively.

Table 1 Moisture and nutritional characteristics of fresh and optimally treated ash gourd samples

Reference	Treatment	MC (%WB)	DT (h)	Yield (%)	CF (%)	SP (mg/g DM)	AA (%/mg DM)	TPC (mg/g DM)	VITC (mg/100 g DM)
This work	F	94.74 ± 0.12 ^b	NA	NA	97.67 ± 0.10 ^d	18.66 ± 1.71 ^a	0.60 ± 0.12 ^b	272.43 ± 5.19 ^b	214.21 ± 10.71 ^b (11.27 ± 0.56)
	D	7.19 ± 0.06 ^c	11	9.38	98.45 ± 0.05 ^b	10.56 ± 0.82 ^b	1.01 ± 0.01 ^a	332.50 ± 9.45 ^a	362.19 ± 9.27 ^a (336.15 ± 8.61)
	B	96.05 ± 0.02 ^a	NA	NA	98.15 ± 0.12 ^c	19.56 ± 1.26 ^a	0.23 ± 0.17 ^c	225.33 ± 29.68 ^c	190.17 ± 8.23 ^c
[6]	BD	7.00 ± 0.11 ^d	09	5.60	98.76 ± 0.06 ^a	8.14 ± 0.20 ^c	0.70 ± 0.03 ^b	142.84 ± 5.52 ^d	193.85 ± 6.06 ^c (9.85)
[21]	F	96.05							
[31]	D	7.30	10	4.61	–	5.98	–	–	(304.37)
	BD	4.00	08	–	–	–	–	–	–

Superscripted means in a column differ significantly ($p < 0.05$; $n = 3$)

Values in parentheses refer to nutritional composition with respect to wet weight

Table 2 Moisture and nutritional characteristics of fresh and optimally treated bitter gourd samples

Reference	Treatment	MC (%WB)	DT (h)	Yield (%)	CF (%)	SP (mg/g DM)	AA (%/mg DM)	TPC (mg/g DM)	VITC (mg/100 g DM)
This work	F	91.12 ± 0.07 ^a	NA	NA	94.82 ± 0.08 ^a	16.47 ± 1.45 ^a	0.3 ± 0.02 ^c (0.04 ± 0.002)	421.81 ± 59.71 ^a	674.60 ± 3.66 ^a
	D	4.97 ± 0.06 ^d	09	10.96	94.61 ± 0.02 ^b	13.47 ± 0.03 ^b	0.97 ± 0.004 ^a	247.34 ± 11.84 ^b	559.25 ± 6.85 ^b
	B	90.50 ± 0.05 ^b	NA	NA	94.85 ± 0.15 ^a	8.21 ± 1.42 ^c	0.60 ± 0.16 ^b (0.06 ± 0.02)	177.25 ± 20.17 ^c	318.26 ± 3.42 ^d
[23]	BD	5.90 ± 0.06 ^c	06	8.03	94.33 ± 0.09 ^c	7.96 ± 0.92 ^c	0.59 ± 0.04 ^b	204.51 ± 17.05 ^{b,c}	522.87 ± 3.46 ^c
	F	-	-	-	-	-	0.05	-	-
	B	-	-	-	-	-	0.07	-	-

Superscripted means in a column differ significantly ($p < 0.05$; $n = 3$)

Values in parentheses refer to nutritional composition with respect to wet weight

Table 3 Moisture and nutritional characteristics of fresh and optimally treated bottle gourd samples

Reference	Treatment	MC (%WB)	DT (h)	Yield (%)	CF (%)	SP (mg/g DM)	AA (%/mg DM)	TPC (mg/g DM)	VITC (mg/100 g DM)
This work	F	93.20 ± 0.12 ^a	NA	NA	96.24 ± 0.11 ^c	25.04 ± 0.22 ^a	5.77 ± 0.72 ^a	722.07 ± 8.45 ^b	142.22 ± 27.78 ^a (8.08 ± 0.86)
	D	7.05 ± 0.17 ^c	10	4.30	97.56 ± 0.10 ^a	10.66 ± 0.56 ^b	1.02 ± 0.002 ^c	261.76 ± 5.08 ^c	143.45 ± 3.50 ^a
	B	92.15 ± 0.10 ^b	NA	NA	96.86 ± 0.08 ^b	8.80 ± 0.09 ^c	3.79 ± 0.2 ^b	808.11 ± 2.73 ^a	88.51 ± 10.96 ^b (6.95 ± 0.86)
	BD	6.26 ± 0.43 ^d	10	3.48	97.65 ± 0.30 ^a	7.47 ± 0.27 ^d	0.56 ± 0.02 ^c	169.00 ± 1.68 ^d	72.12 ± 6.01 ^b (13)
[31]	F	-	-	-	-	-	-	-	-
	D	-	-	-	-	-	-	-	-
[4]	B	-	-	-	-	-	-	-	(6.99)
[31]	BD	4.00	08	-	-	-	-	-	-

Superscripted means in a column differ significantly ($p < 0.05$; $n = 3$)

Values in parentheses refer to nutritional composition with respect to wet weight

Table 4 Moisture and nutritional characteristics of fresh and optimally treated bean samples

Reference	Treatment	MC (%WB)	DT (h)	Yield (%)	CF (%)	SP (mg/g DM)	AA (%/mg DM)	TPC (mg/g DM)	VITC (mg/100 g DM)
This work	F	91.76 ± 0.78 ^a	NA	NA	95.13 ± 0.08 ^c	28.26 ± 1.64 ^a	0.43 ± 0.04 ^c (0.04 ± 0.003)	272.75 ± 3.31 ^a	100.28 ± 3.95 ^b (8.26 ± 0.33)
	D	7.72 ± 0.6 ^b	08	10.06	95.35 ± 0.12 ^b	13.18 ± 0.36 ^b	0.7 ± 0.01 ^a	292.20 ± 10.24 ^a	142.45 ± 3.52 ^a
	B	92.06 ± 0.50 ^a	NA	NA	96.24 ± 0.08 ^a	14.57 ± 0.84 ^b	0.67 ± 0.05 ^b	224.62 ± 16.58 ^b	96.97 ± 4.10 ^b
[9]	BD	5.83 ± 0.26 ^c	08	9.24	96.09 ± 0.11 ^a	9.15 ± 0.04 ^c	0.12 ± 0.01 ^d	154.36 ± 7.66 ^c	85.75 ± 3.45 ^c (8.6)
[34]	F	90.39	-	-	-	-	-	-	-
[19]	F	-	-	-	-	-	(0.02)	-	-
	BD	13.96	05	-	-	-	-	-	-

Superscripted means in a column differ significantly ($p < 0.05$; $n = 3$)

Values in parentheses refer to nutritional composition with respect to wet weight

Table 5 Moisture and nutritional characteristics of fresh and optimally treated pumpkin samples

Reference	Treatment	MC (%WB)	DT (h)	Yield (%)	CF (%)	SP (mg/g DM)	AA (%/mg DM)	TPC (mg/g DM)	VITC (mg/100 g DM)
This work	F	91.20 ± 0.03 ^b	NA	NA	89.18 ± 0.15 ^b	15.00 ± 0.49 ^b	0.07 ± 0.04 ^c	215.17 ± 11.85 ^a	207.00 ± 3.70 ^a (18.22 ± 0.33)
	D	7.21 ± 0.42 ^c	08	6.00	90.16 ± 0.08 ^a	15.94 ± 0.75 ^a	0.31 ± 0.01 ^a	143.69 ± 1.70 ^c	135.60 ± 3.51 ^b
	B	91.95 ± 0.05 ^a	NA	NA	89.83 ± 0.13 ^a	8.50 ± 0.10 ^c	0.06 ± 0.06 ^c	173.32 ± 4.85 ^b	114.31 ± 4.04 ^c
	BD	5.08 ± 0.38 ^d	08	3.75	90.11 ± 0.47 ^a	7.16 ± 0.38 ^d	0.19 ± 0.01 ^b	34.76 ± 5.43 ^d	120.68 ± 3.43 ^c (14.12)
[9]	F	–	–	–	–	–	–	–	–
[3]	B	90.60	–	–	–	–	–	–	–

Superscripted means in a column differ significantly ($p < 0.05$; $n = 3$)

Values in parentheses refer to nutritional composition with respect to wet weight

Table 6 Moisture and nutritional characteristics of fresh and optimally treated squash samples

Reference	Treatment	MC (%WB)	DT (h)	Yield (%)	CF (%)	SP (mg/g DM)	AA (%/mg DM)	TPC (mg/g DM)	VITC (mg/100 g DM)
This work	F	93.06 ± 0.34 ^a	NA	NA	95.25 ± 0.12 ^b	19.50 ± 0.47 ^a	0.29 ± 0.12 ^b	163.90 ± 0.86 ^a	78.43 ± 4.66 ^b
	D	7.78 ± 0.08 ^c	09	6.33	95.78 ± 0.07 ^a	13.34 ± 0.36 ^b	0.47 ± 0.02 ^a	166.57 ± 6.98 ^a	89.60 ± 3.53 ^a
	B	92.45 ± 0.12 ^b	NA	NA	95.34 ± 0.12 ^b	7.83 ± 0.24 ^c	0.10 ± 0.04 ^c	102.87 ± 2.09 ^b	42.28 ± 4.31 ^d
	BD	5.45 ± 0.11 ^d	09	3.60	95.84 ± 0.08 ^a	7.51 ± 0.37 ^c	0.11 ± 0.01 ^c	76.32 ± 5.00 ^c	69.52 ± 3.44 ^c
Shara and Mussa [30]	F	–	–	–	–	–	–	–	75.56
	B	–	–	–	–	–	–	–	38
[14]	BD	15.15	7.25	–	–	–	–	–	–

Superscripted means in a column differ significantly ($p < 0.05$; $n = 3$)

Table 7 Moisture and nutritional characteristics of fresh and optimally treated pointed gourd samples

Reference	Treatment	MC (%WB)	DT (h)	Yield (%)	CF (%)	SP (mg/g DM)	AA (%/mg DM)	TPC (mg/g DM)	VITC (mg/100 g DM)
This work	F	88.27 ± 0.10 ^a	NA	NA	95.74 ± 0.66 ^a	12.86 ± 0.28 ^a	0.15 ± 0.15 ^c	162.30 ± 1.83 ^b	68.84 ± 2.77 ^c (8.08 ± 0.33)
	D	4.34 ± 0.08 ^d	08	6.55	93.61 ± 0.55 ^b	13.34 ± 0.36 ^a	0.99 ± 0.001 ^a	171.35 ± 2.16 ^a	237.54 ± 6.80 ^a
	B	86.93 ± 0.32 ^b	NA	NA	96.17 ± 1.43 ^a	5.65 ± 0.25 ^c	0.08 ± 0.09 ^c	127.26 ± 1.64 ^d	41.67 ± 6.58 ^d
	BD	5.19 ± 0.07 ^c	09	9.00	95.55 ± 0.74 ^a	7.51 ± 0.37 ^b	0.33 ± 0.04 ^b	131.57 ± 2.17 ^c	156.48 ± 9.08 ^b
[16]	F	92.00	-	-	-	-	-	-	-
	B	91.50	-	-	-	-	-	-	-
[10]	BD	7.50	11	-	-	-	-	-	-
	F	-	-	-	-	-	-	-	(9.33)

Superscripted means in a column differ significantly ($p < 0.05$; $n = 3$)

Values in parentheses refer to nutritional composition with respect to wet weight

Table 8 Moisture and nutritional characteristics of fresh and optimally treated spine gourd samples

Reference	Treatment	MC (%WB)	DT (h)	Yield (%)	CF (%)	SP (mg/g DM)	AA (%/mg DM)	TPC (mg/g DM)	VITC (mg/100 g DM)
This work	F	88.09 ± 0.12 ^a	NA	NA	96.52 ± 0.51 ^a	11.93 ± 0.33 ^b	0.91 ± 0.07 ^b	201.40 ± 1.32 ^c	575.52 ± 17.91 ^b (68.54 ± 2.13)
	D	6.83 ± 0.07 ^c	08	11.80	95.19 ± 0.15 ^b	14.20 ± 0.03 ^a	1.01 ± 0.003 ^a (0.94 ± 0.002)	241.22 ± 2.93 ^a	733.68 ± 9.24 ^a
	B	87.41 ± 0.12 ^b	NA	NA	96.31 ± 0.18 ^a	7.27 ± 0.33 ^d	0.30 ± 0.06 ^c (0.04 ± 0.01)	168.68 ± 2.95 ^d	319.20 ± 15.72 ^c
[10] [15] [23]	BD	5.96 ± 0.05 ^d	08	8.70	95.80 ± 0.62 ^{a,b}	8.28 ± 0.03 ^c	0.99 ± 0.013 ^{a,b}	206.83 ± 2.28 ^b	305.53 ± 15.85 ^c (39.40)
	F	84.23	-	-	-	-	-	-	-
	D	-	-	-	-	-	(0.83)	-	-
	B	-	-	-	-	-	(0.07)	-	-

Superscripted means in a column differ significantly ($p < 0.05$; $n = 3$)

Values in parentheses refer to nutritional composition with respect to wet weight

Table 9 Moisture and nutritional characteristics of fresh and optimally treated yardlong bean samples

Reference	Treatment	MC (%WB)	DT (h)	Yield (%)	CF (%)	SP (mg/g DM)	AA (%/mg DM)	TPC (mg/g DM)	VITC (mg/100 g DM)
This work	F	86.28 ± 0.34 ^a	NA	NA	97.02 ± 1.09 ^b	17.73 ± 1.54 ^a	3.39 ± 0.03 ^b	492.42 ± 1.56 ^a	154.67 ± 2.37 ^a
	D	4.90 ± 0.33 ^b	08	11.00	98.82 ± 0.83 ^a	12.43 ± 0.45 ^b	0.95 ± 0.004 ^c	273.90 ± 7.69 ^d	150.08 ± 9.05 ^{ab}
	B	88.07 ± 1.95 ^a	NA	NA	98.69 ± 0.04 ^a	7.02 ± 0.41 ^c	4.13 ± 0.15 ^a	400.10 ± 11.69 ^b	140.09 ± 2.73 ^b
	BD	5.46 ± 2.12 ^b	09	8.25	97.94 ± 0.49 ^{ab}	6.93 ± 0.42 ^c	0.36 ± 0.03 ^d	289.70 ± 6.01 ^c	111.24 ± 6.88 ^c
[33]	F	88.74	-	-	-	-	-	-	-
	BD	13.96	-	-	-	-	-	-	-
[19]		-	05	-	-	-	-	-	-

Superscripted means in a column differ significantly ($p < 0.05$; $n = 3$)

Table 10 Moisture and nutritional characteristics of fresh and optimally treated ridge gourd samples

Reference	Treatment	MC (%WB)	DT (h)	Yield (%)	CF (%)	SP (mg/g DM)	AA (%/mg DM)	TPC (mg/g DM)	VITC (mg/100 g DM)
This work	F	91.20 ± 0.03 ^a	NA	NA	92.06 ± 0.39 ^b	21.58 ± 0.10 ^a	0.58 ± 0.10 ^a	135.90 ± 9.37 ^b	128.04 ± 6.40 ^a (11.27 ± 0.56)
	D	7.43 ± 0.04 ^c	12	4.05	97.96 ± 0.34 ^a	18.43 ± 0.06 ^b	0.62 ± 0.08 ^a	170.76 ± 5.26 ^a	99.41 ± 3.51 ^b
	B	90.30 ± 1.40 ^b	NA	NA	98.26 ± 0.43 ^a	9.31 ± 0.27 ^c	0.16 ± 0.19 ^b	91.05 ± 1.62 ^d	77.44 ± 3.35 ^c (7.51 ± 0.33)
	BD	6.09 ± 0.06 ^d	10	1.87	97.91 ± 0.61 ^a	9.49 ± 0.01 ^c	0.36 ± 0.02 ^b	124.42 ± 4.16 ^c	67.99 ± 3.46 ^d
[31]	F	-	-	-	-	-	-	-	(13)
[4]	B	-	-	-	-	-	-	-	(6.99)
[31]	BD	4.00	08	-	-	-	-	-	-

Superscripted means in a column differ significantly ($p < 0.05$; $n = 3$)

Values in parentheses refer to nutritional composition with respect to wet weight

Table 11 Moisture and nutritional characteristics of fresh and optimally treated snake gourd samples

Reference	Treatment	MC (%WB)	DT (h)	Yield (%)	CF (%)	SP (mg/g DM)	AA (%/mg DM)	TPC (mg/g DM)	VITC (mg/100 g DM)
This work	F	95.12 ± 0.04 ^b	NA	NA	93.27 ± 0.97 ^b	20.68 ± 0.98 ^a	0.10 ± 0.03 ^b	320.40 ± 43.82 ^a	107.75 ± 13.53 ^b
	D	6.82 ± 0.24 ^c	11	4.29	98.74 ± 0.44 ^a	14.79 ± 0.48 ^b	0.66 ± 0.01 ^a	167.06 ± 1.92 ^b	203.55 ± 3.49 ^a
	B	96.05 ± 0.05 ^a	NA	NA	97.88 ± 0.61 ^a	15.82 ± 0.45 ^b	0.80 ± 0.16 ^a	179.22 ± 13.14 ^b	190.17 ± 16.47 ^a
	BD	5.94 ± 0.01 ^d	09	2.73	97.90 ± 0.99 ^a	6.85 ± 0.03 ^c	0.16 ± 0.003 ^b	100.83 ± 4.78 ^c	103.82 ± 3.46 ^b
[4]		64	-	-	-	-	-	-	-
	B	-	-	-	-	-	-	-	(6.99)
[31]	BD	4.00	08	-	-	-	-	-	-

Superscripted means in a column differ significantly ($p < 0.05$; $n = 3$)

Values in parentheses refer to nutritional composition with respect to wet weight

Table 12 Moisture and nutritional characteristics of fresh and optimally treated sponge gourd samples

Reference	Treatment	MC (%WB)	DT (h)	Yield (%)	CF (%)	SP (mg/g DM)	AA (%/mg DM)	TPC (mg/g DM)	VITC (mg/100 g DM)
This work	F	94.79 ± 0.12 ^b	NA	NA	96.63 ± 0.44 ^a	25.85 ± 0.48 ^a	0.63 ± 0.17 ^a	283.62 ± 12.87 ^b	111.74 ± 6.24 ^a
	D	7.56 ± 0.03 ^c	12	6.00	96.82 ± 0.29 ^a	17.82 ± 0.18 ^c	0.78 ± 0.11 ^a	164.31 ± 0.64 ^c	117.83 ± 7.04 ^a
	B	96.36 ± 0.20 ^a	NA	NA	93.12 ± 1.03 ^b	24.76 ± 0.85 ^b	0.37 ± 0.16 ^b	413.50 ± 24.69 ^a	98.02 ± 8.94 ^b
	BD	5.73 ± 0.08 ^d	11	1.72	95.97 ± 0.76 ^a	7.99 ± 0.16 ^d	0.36 ± 0.10 ^b	169.87 ± 4.93 ^c	77.69 ± 5.98 ^c

Superscripted means in a column differ significantly ($p < 0.05$; $n = 3$)

3.2 Influence of Blanching and Tray Drying on Moisture Content and Drying Time

Figures 1 and 2 depict the moisture content and optimal drying time of dried and blanched dried vegetables. Moisture content of blanched winter vegetables varied from 90.50 (BIG) to 96.05% (AG). Among the blanched summer vegetables, SPOG and PG indicated highest (96.36%) and lowest (86.93%) moisture content respectively. These findings for blanched P and PG agree with those reported in the relevant prior art [3, 16].

Among the optimally dried winter vegetables, BIG (9 h) and S (9 h) possessed lowest (4.97%) and highest (7.78%) moisture content. Among optimally blanched dried winter vegetables, P and AG possessed the lowest (5.08%) and highest (7.00%) moisture content for a drying time of 8 and 9 h respectively. The results obtained for dried AG and blanched dried BOG are in good agreement with the findings of [21, 31].

Among summer vegetables, lowest (4.34%) and highest (7.56%) moisture content was obtained for optimally dried PG (8 h) and SPOG (12 h). Among blanched and optimally dried summer vegetables, lowest (5.19%) and highest (6.09%) moisture content was obtained for PG (9 h) and RG (10 h). Among all optimally dried vegetables, the obtained results affirm a moisture content less than 8 percent (wet basis) and hence good shelf life characteristics based on general rules of thumb [26]. Thus, the investigations inferred that, except for PG and YB, the moisture removal rate was faster for blanched vegetables. This is due to the faster transport of moisture from the inner sections to the outer surface due to the tissue softening achieved through the blanching process [25].

3.3 Influence of Blanching and Tray Drying on Antioxidant Activity

Antioxidant activity of blanched winter and summer vegetables varied from 0.06 (P)—3.79%/mg (BOG) and 0.08 (PG)—4.13%/mg (YB) respectively. The results obtained for fresh BS, BIG and S and blanched SPIG are in agreement with the findings reported in the relevant prior art [7, 15, 23, 34]. It has been perceived that blanching had a significant effect on the antioxidant activity of the fresh vegetables. However, the effect was positive for few vegetables and detrimental for few other vegetables. Among the chosen winter and summer vegetables, the antioxidant activity of AG, BOG, P, S, PG, RG, SPIG and SPOG reduced after blanching. Similar trend was reported in the prior art for green peas [41] and long bean [38]. This reduction may be ascribed to the loss of water-soluble antioxidant compounds due to leaching. However, for the BS, BIG, SG and YB vegetables, the antioxidant activity increased after blanching. Similar trend was reported in the relevant prior art for green BS [36] and BIG [23]. For these enhancing trends, the possible reason can be correlated

to the inactivation of peroxidase enzyme during blanching to thereby reduce the pro-oxidants content in the processed vegetable [5].

Among dried winter vegetables, P and BOG possessed lowest (0.31%/mg) and highest (1.02%/mg) respectively. Among dried summer vegetables, RG and SPIG possessed lowest (0.62%/mg) and highest (1.01%/mg) antioxidant activity respectively. The percent variation in antioxidant activity of dried and blanched dried vegetables with respect to the fresh vegetables has been illustrated in Fig. 3. The figure depicts that drying alone fostered enhanced antioxidant activity for most vegetables but not BOG and YB. Such positive influence of drying upon the antioxidant activity can be ascribed to the formation of new antioxidant compounds through the Maillard reaction along with the release of bounded antioxidants due to the disruption of cell wall [20]. On the contrary, a detrimental influence of drying on the antioxidant activity. May be correlated to the heat sensitivity of most antioxidant constituents in the processed vegetable [28].

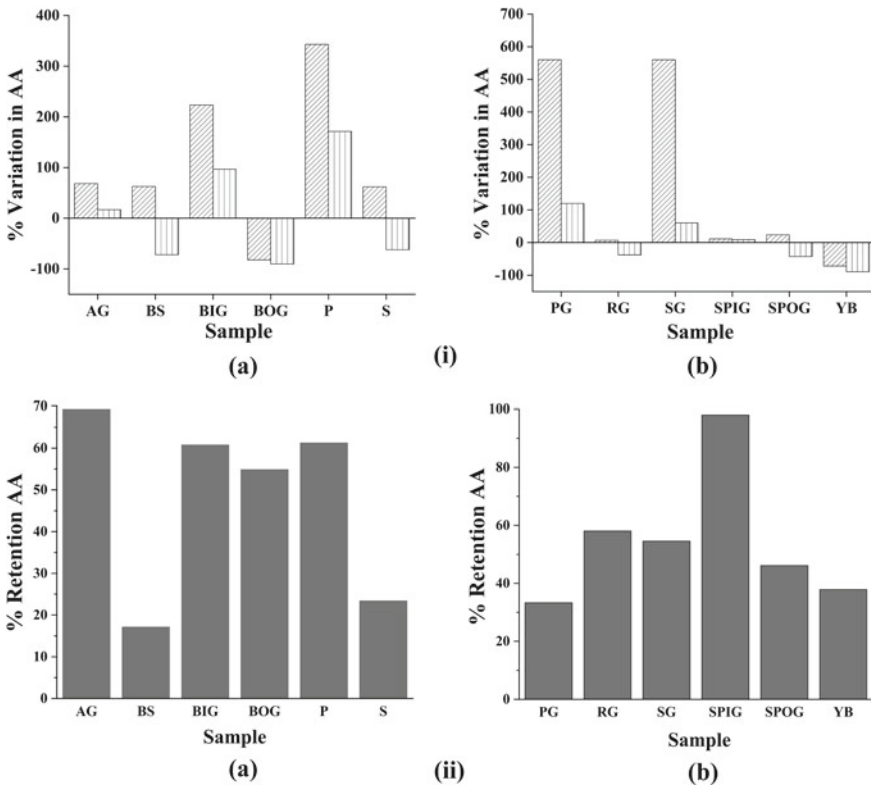


Fig. 3 (i) Percentage variation in AA of dried (//////) and blanched dried (|||||) sample with respect to fresh sample of **a** winter vegetables **b** summer vegetables (ii) percentage retention of AA in blanched dried samples compared to dried samples **a** winter vegetables **b** summer vegetables

For the case involving blanching followed with drying, the positive influence of the processing scheme on the antioxidant activity was observed for only few vegetables (AG, BIG, P, PG, SG and SPIG). Similar trend was reported for Moringa leaves in the relevant prior art [40]. Such findings have been correlated to the inactivation of polyphenol oxidase enzyme during blanching and release of bound antioxidant compounds due to cell wall disruption, and formation of new antioxidant compounds during drying. However, for all other vegetables, a negative influence was observed due to the combined processing scheme. Such detrimental influence has been reported for cabbage by [28]. The authors indicated the potential role of heat sensitivity of antioxidant compounds in due course of vegetable drying. Among all winter vegetables, the combined processing scheme indicated lowest (0.11%/mg) and highest (0.70%/mg) antioxidant activity for S and AG respectively. Among all summer vegetables, the corresponding lowest and highest values were obtained for SG (0.16%/mg) and SPIG (0.99%/mg) respectively. Further, for all vegetables, a combination of blanching and drying always indicated lower antioxidant activity than those achieved through the tray drying process alone. This has been attributed to the comparatively prolonged heat exposure of vegetables during the combined process scheme.

A comparative enhancement of the combined scheme towards percentage retention of antioxidant activity has been presented in Fig. 3. Among winter vegetables, maximum (69.31%) and minimum (17.14%) retention were indicated for AG and BS respectively. Further, among summer vegetables, SPIG and PG indicated maximum (98.02%) and minimum (33.33%) retention respectively. Also, it is interesting to note that the antioxidant activity retention of all vegetables except BS and S was close to or more than 50%. The findings thereby affirm that for vegetables such as BIG and SPIG blanching can be used as a preprocessing treatment for the development of dried vegetables with good sensory and nutritional aspects. The detrimental/enhancing trends of antioxidant activity for various considered treatments have been analyzed to be due to a combination of several chemical and surface phenomena. These include synergistic effects of antioxidants and polyphenolic compounds, influence of bioactive constituents in the raw material, heat sensitive behavior of antioxidant and polyphenolic constituents and cell wall structure of the vegetables. In this regard, it shall be noted that tougher cell walls may not get disrupted during the suggested process schemes and hence they do have a strong influence on the observed trends [11, 35]. Optical microscope-based imaging has been useful to analyse the toughness and rupturability of the cell wall. To corroborate with this hypothesis, optical microscope images were obtained for various cases and the hypothesis has been validated with relevant insights associated to the cell wall rupturing effect (Fig. 4).

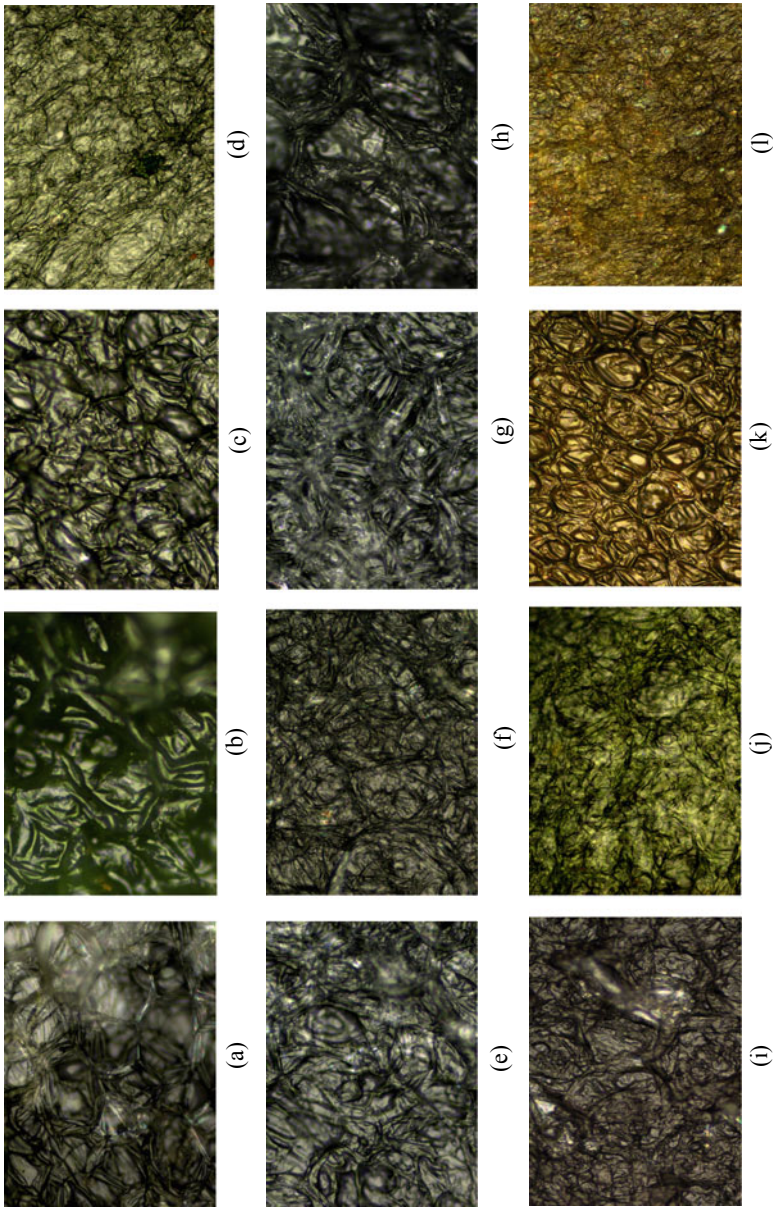


Fig. 4 Optical microscope images (20X magnification) depicting cell wall structure of **a** BS (F) **b** BS (B) **c** BS (D) **d** BS (BD) **e** BOG (D) **f** BOG (BD) **g** S (D) **h** S (BD) **i** BIG (BD) **j** BIG (D) **k** P (D) **l** P (BD) **m** SP1G (D) **n** SP1G (BD) **o** SG (D) **p** SG (BD)

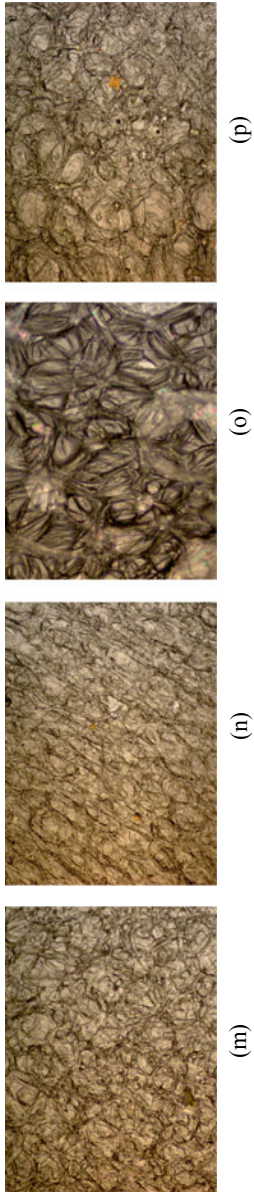


Fig. 4 (continued)

3.4 Influence of Blanching and Tray Drying on Total Phenol Content

TPC of blanched winter and summer vegetables varied from 102.87 (S)—808.11 mg/g (BOG) and 91.05 (RG)—413.50 mg/g (SPOG) respectively. The results depicted a significant effect of blanching on TPC. Among the chosen winter and summer vegetables, blanching resulted in the TPC reduction of all vegetables but BOG and SPOG.

Among dried winter vegetables, AG (332.50 mg/g) and P (143.69 mg/g) possessed highest and lowest TPC respectively. Among dried summer vegetables, YB (273.90 mg/g) and SPOG (164.31 mg/g) possessed highest and lowest TPC respectively. Among all winter vegetables subjected to combined processing scheme, BIG (204.51 mg/g) and P (34.76 mg/g) indicated highest and lowest TPC respectively. Among all summer vegetables, the corresponding highest and lowest values were obtained for YB (289.70 mg/g) and SG (100.83 mg/g) respectively. The percent variation in TPC of dried and blanched dried vegetables with respect to the fresh vegetables has been illustrated in Fig. 5. The figure depicts that drying alone fostered enhanced TPC for AG, BS, S, PG, RG and SPIG. For the case involving blanching followed with drying, the positive influence of the processing scheme on the TPC was observed for only SPIG. For most vegetables subjected to various treatments, the similar trend observed for TPC and antioxidant activity can be correlated to the synergistic influence between both constituents [42]. However, the varied trends may be attributed to factors like sensitivity of few compounds to heat treatments and changes in the cell wall structures when subjected to varied treatments. A comparative enhancement of the combined scheme towards percentage retention of TPC has been presented in Fig. 5. Among winter vegetables, maximum (82.68%) and minimum (24.19%) retention were indicated for BIG and P respectively. Further, among summer vegetables, YB and SG indicated maximum (105.77%) and minimum (60.36%) retention respectively.

3.5 Influence of Blanching and Tray Drying on Vitamin C Content

Among all chosen vegetables, except SG, blanching proved to be unfavorable for the retention of vitamin C content. The possible reason for such vitamin C enhancement in SG could be due to inactivation of ascorbic acid oxidase in due course of blanching [22]. The contrary trend has been due to the leaching-based vitamin C loss driven with its water-soluble nature. Among blanched winter vegetables, BIG (318.26 mg/100 g) and S (48.28 mg/100 g) possessed highest and lowest vitamin C content. Similarly, among blanched summer vegetables, PG (41.67 mg/100 g) and SPIG (319.20 mg/100 g) possessed the lowest and highest vitamin C contents respectively.

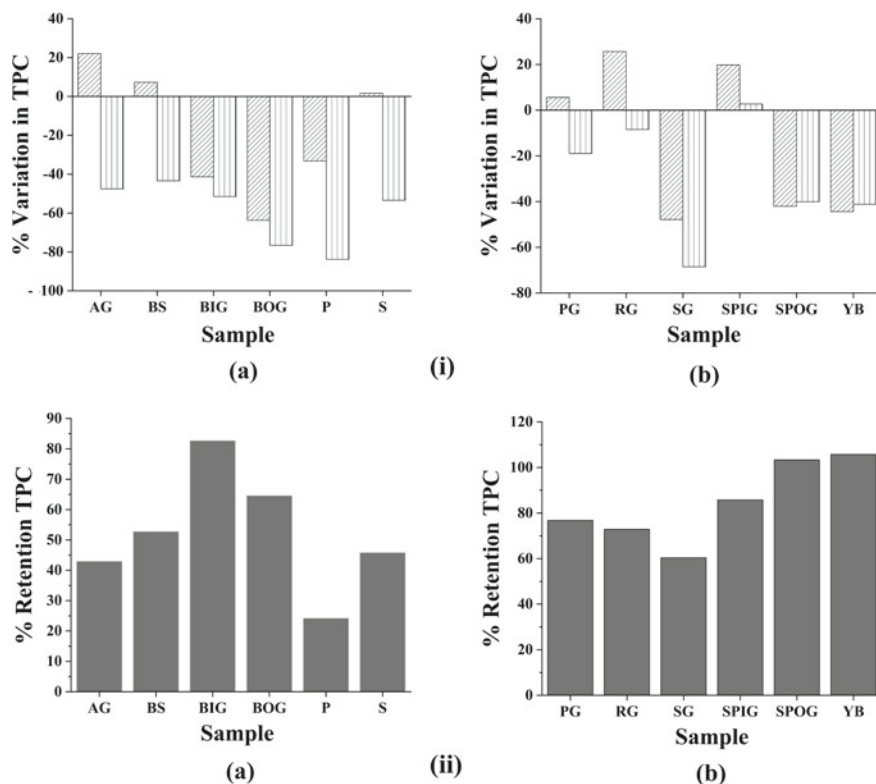


Fig. 5 (i) Percentage variation in TPC of dried (//////) and blanched dried (|||||) sample with respect to fresh sample of **a** winter vegetables **b** summer vegetables (ii) percentage retention of TPC in blanched dried samples compared to dried samples **a** winter vegetables **b** summer vegetables

Figure 6 depicts the percentage variation in the vitamin C content of dried and blanched dried vegetables with respect to the fresh vegetables. It has been observed that drying alone facilitated an enhancement in the vitamin C content of most vegetables but not BIG, P, RG and YB. Such pertinent enhancement could be possibly due to the intact structure of the cell wall matrix that prevented the thermal or oxidative damage of the vitamin C constituents [13]. In few vegetables, the detrimental influence of drying on the Vitamin C content can be corroborated with its heat sensitivity characteristics and oxidation based loss [18, 20, 28]. Among the dried winter vegetables, lowest (89.60 mg/100 g) and highest (559.25 mg/100 g) contents of vitamin C were observed for S and BIG samples respectively. Among dried summer vegetables, RG and SPIG possessed lowest (99.41 mg/100 g) and highest (733.68 mg/100 g) vitamin C content respectively.

For the case of combined blanching and drying scheme, except PG, all other vegetables indicated reduction in the vitamin C content. Similar trend was reported in the relevant prior art [24, 28]. Leaching during blanching and thermal and oxidative

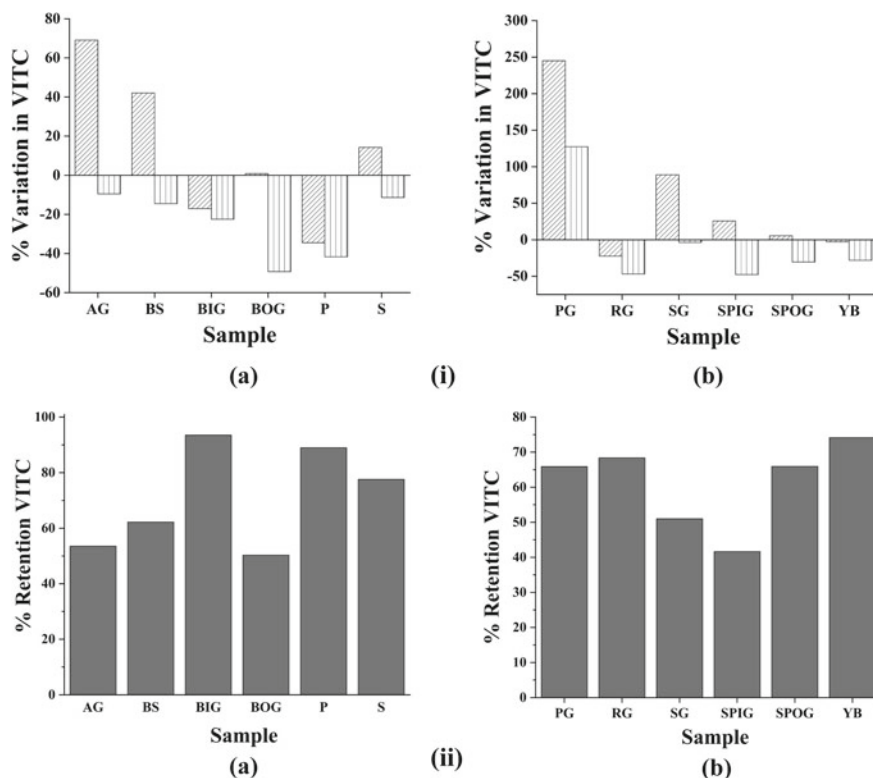


Fig. 6 (i) Percentage variation in VITC of dried (//////) and blanched dried (|||||) sample with respect to fresh sample of **a** winter vegetables **b** summer vegetables (ii) percentage retention of TPC in blanched dried samples compared to dried samples **a** winter vegetables **b** summer vegetables

degradation during drying have been accounted to be primarily responsible for such detrimental influence of the combined processing scheme. Among blanched dried winter vegetables, S and BIG possessed minimum (69.52 mg/100 g) and maximum (522.87 mg/100 g) vitamin C content respectively. Similarly, among blanched dried summer vegetables, RG and SPIG possessed minimum (67.99 mg/100 g) and maximum (103.82 mg/100 g) vitamin C content respectively. The percent retention of vitamin C content of blanched dried vegetables with respect to dried vegetables has been depicted in Fig. 6. The illustration infers that despite processing ensuring a reduction of vitamin C content, the retention was close to 50% for SPIG and more than 50% for all other vegetables. Among winter vegetables, BIG and BOG refer to maximum (93.49%) and minimum (50.28%) retention. Corresponding maximum and minimum retention among summer vegetables refer to YB (74.12%) and SPIG (41.64%) respectively. For all vegetables, Vitamin C content of dried vegetables was higher in comparison with the corresponding blanched dried vegetables. This is due

to the leaching of vitamin C during blanching followed with heat sensitive constituent loss in due course of prolonged exposure to hot air during drying [28].

3.6 Influence of Blanching and Tray Drying on Soluble Protein Content

Among blanched winter vegetables, AG (19.56 mg/g) and S (7.83 mg/g) possessed highest and lowest soluble protein content. Among blanched summer vegetables, SPOG (24.76 mg/g) and PG (5.65 mg/g) possessed highest and lowest soluble protein content respectively. Among dried winter vegetables, P (15.94 mg/g) and AG (10.56 mg/g) possessed highest and lowest soluble protein content. Similarly, among dried summer vegetables, RG (18.43 mg/g) and PG (13.34 mg/g) possessed highest and lowest soluble protein content respectively. Among all winter vegetables subjected to combined processing scheme, BS (9.15 mg/g) and P (7.16 mg/g) possessed highest and lowest soluble protein content. Similarly, among summer vegetables subjected to combined processing, RG (9.49 mg/g) and SG (6.85 mg/g) possessed maximum and minimum soluble protein content. In most cases, a reduction in soluble protein content in due course of various treatments has been observed. However, for very few cases, a marginal increase has been noticed. However, the enhancement was observed to be very minimal. This reduction could be ascribed to the leaching of soluble compounds during blanching and removal along with moisture during drying [21].

3.7 Influence of Blanching and Tray Drying on Crude Fiber Content

Among all optimally treated winter vegetables, AG (98.45%) and P (90.16%) possessed highest and lowest crude fiber content respectively. Similarly, among summer vegetables, crude fiber content of dried and blanched dried vegetables varied from 93.61–98.82% and 95.55–97.94% respectively. Among dried and blanched dried summer vegetables, YB and PG possessed highest and lowest amounts of crude fiber content respectively. In most winter and summer vegetables, it has been observed that blanching and drying resulted in marginal enhancement of the crude fiber content. This marginal enhancement may be attributed to the varying total solid content of the sample due to the losses of sugar, mineral and vitamin constituents [39]. For both PG and SPIG, drying fostered a marginal reduction in the crude fiber content. This is attributed to the heat based solubilization of fiber compounds such as cellulose, hemicellulose, and pectin [2].

3.8 Effect of Blanching and Drying on Yield

Among dried and blanched dried winter vegetables, the yield varied from 4.30–10.96% and 3.48–9.24% respectively. Among dried winter vegetables, BOG (4.30%) and BIG (10.96%) samples possessed lowest and highest yield respectively. Similarly, among blanched dried winter vegetables, BOG (3.48%) and BS (9.24%) provided lowest and highest yield respectively. Among dried summer vegetables, RG (4.05%) and SPIG (11.80%) affirmed lowest and highest yield. However, among blanched dried summer vegetables, the SPOG (1.72%) and PG (9.00%) possessed lowest and highest yield respectively. In summary, dried vegetables facilitated higher yield characteristics in comparison with the blanched dried vegetables. Such generalized inference is in agreement with those being inferred for broccoli by [12]. This comparatively greater yield is attributed to the specific losses in due course of the blanching process.

3.9 Identification of Vegetables for Soup Mix Formulations

Among dried winter vegetables, the best vegetables correspond to AG due to appropriate processing time (11 h), highest antioxidant activity (1.01%/mg), TPC (332.50 mg/g) and vitamin C (362.19 mg/100 g); BIG due to appropriate processing time (9 h), highest antioxidant activity (0.97%/mg), TPC (247.34 mg/g) and vitamin C (559.26 mg/100 g) and BOG due to appropriate processing time (10 h), highest antioxidant activity (1.02%/mg), TPC (261.76 mg/g) and appropriate vitamin C (143.45 mg/100 g) content. Similarly, blanched and dried BIG is the best due to lowest processing time (6 h), high antioxidant activity (0.59%/mg), TPC (204.51 mg/g) and highest vitamin C (522.87 mg/100 g) content.

Among dried summer vegetables, the best vegetables correspond to PG due to appropriate processing time (8 h), highest antioxidant activity (0.99%/mg) and vitamin C (237.54 mg/100 g); SPIG due to appropriate processing time (8 h), highest antioxidant activity (1.01%/mg) and vitamin C (733.68 mg/100 g) and YB due to appropriate processing time (8 h), highest antioxidant activity (0.95%/mg) and appropriate vitamin C (150.08 mg/100 g) content. Similarly, blanched and dried SPIG is the best due to appropriate processing time (8 h), highest antioxidant activity (0.99%/mg), TPC (206.83 mg/g) and vitamin C (305.53 mg/100 g).

4 Conclusions

A systematic study for holistic characterization of nutrients of various summer and winter vegetables to identify their potential towards the development of ready to cook soup mixes has been addressed for the first time in this work. In summary, AG;

BIG; BOG and PG; SPIG; YB can be inferred to be the most relevant vegetables to represent maximum constitution in the winter and summer vegetable-based dry soup mix formulations respectively due to their proportionate and adequate nutritional composition. Another important inference being drawn from conducted investigation is that both blanching-drying and drying processes are competent to achieve the desired nutritional characteristics. However, among these, for all vegetables except BIG and SPIG, only drying is recommended for the considered responses due to the higher retention during drying. It is well known that vegetables such as BIG and SPIG have to be inevitably blanched to mitigate bitterness and enhance sensory characteristics of associated soup mix formulations. However, though a reduction has been observed in such cases, the nutritional content was still comparable to that of only dried vegetables and a retention of more than 50% was observed in the nutritional parameters. From the perspective of processing time, among dried and blanched dried winter vegetables, lowest processing time was achieved for P (8 h) and BIG (6 h) respectively. Similarly, among dried and blanched dried vegetables, lowest processing time was achieved for PG (8 h) and SPIG (8 h) respectively. The important inferences drawn from the present investigation will be further used towards the development of nutritionally rich ready to cook vegetable soup mixes.

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Food Product Design and Development

Sensory and Functional Qualities of Fruit Leather Prepared from Guava (*Psidium Guajava*), Papaya (*Carica papaya* L.) and *Mirika Tenga* (*Parameria polyneura*)



Ginisha Kalsi , Luna Dutta Baruah , and Moloya Gogoi 

Abstract Fruit leather is a fruit based product prepared by drying of fruit purees. Its chewable texture, low fat composition, high dietary fibers and carbohydrate content make it a nutritious value-added alternative to whole fruits as a source of nutrients. The objective of the research was to use locally available and lesser known tropical minor fruits to derive a nutritionally enriched finger food for the elderly with the aim to provide them with essential micronutrient rich food product. Fresh guava, papaya, *mirika tenga* were pureed and mixed in different ratios and heated to 85 °C to inactivate the enzymatic activity. Citric acid (0.2%) was added as a preservative. The mixture was poured in stainless steel trays and dried in a cross flow cabinet dryer at 60 °C until the moisture content was 14–16%. The TSS, pH, titratable acidity and the moisture content of the final leathers were analyzed. The sensory analysis was carried out with the help of Hedonic Scale by 15 panelists to determine the overall acceptability of the developed product. The aim was to preserve the fruits and study about their shelf life to develop the fruit leather without the addition of pectin and reduced amount of sugar. It can be concluded that the product could be packaged in polypropylene and stored at ambient conditions without any detrimental alterations to nutritional, physicochemical and organoleptic properties.

Keywords Fruit leather · Papaya · Guava · *mirika tenga*

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1 Introduction

Fruits are widely recommended in diets of all age groups due to their health-promoting properties. In recent times, under the influence of rapid globalization, there has been a massive alteration in diets across the globe. This has led to homogenization and urbanization of food habits leading to shrinkage of diversity of existing food system. The food policies framed for sustainable agriculture target more upon the production and availability of agricultural crops at lower prices rather than emphasizing on nutritional security. The resultant outcome is extinction of the nutritious species from food menus which include the indigenous and native minor fruits. These fruits constitute varying concentrations of different essential nutrients required by the human body for its growth, development and repair functionalities. Fruits should be regularly included in the diet since they are an excellent source of nutrients including minerals, fibre, carbohydrates, antioxidants and other phytonutrients. Malnutrition amongst the elderly geriatric population, predominantly deficiency of micronutrients such as calcium, magnesium, iron, folate, zinc etc., is common among the elderly population because of an imbalanced and insufficient diet that lacks in essential nutrients. Thereby, it can lead to concomitant malabsorption syndrome [1]. The frequent consumption of fruits and vegetables can lower the risk of acquiring a number of chronic diseases, according to evidence [2]. Nutritional deficiencies are exacerbated by age-related eating issues caused by decreasing appetite, interrupted eating habits, and poor dental health. Consuming fruits and vegetables may help prevent the incidence or advancement of various geriatric diseases [3]. Fruit-based finger foods could be an effective nutritional plan for healthy aging. Fruit leather could be a potential finger food for the elderly in the context that they can eat between meals as a snack due to being abundantly constituted with essential vitamins and minerals [4].

Fruit leather which is also known as fruit bar or fruit slab is a reconstituted and restructured sheet-shaped dehydrated dietary fruit-based product made from fruit pulp or fruit puree of different fruits [5]. The fruit concentrates are mixed with appropriate quantities of sugar without the addition of food additives and complex preservatives. The only preservative used in the product is citric acid for prolonged shelf life of the fruit leather. The addition of sugar/sucrose gave the leather sweetness to enhance the taste and balance the flavor and increased the total soluble solids content. Due to problems of dentition, fallen teeth, and dentures, the foods for the elderly should be soft, chewable and easily digestible. Hence, fruit leather serves as a snack which is loaded with nutrients and can be eaten anytime. The most important principle for planning newly developed fruit leather is to provide a food product which is nutritionally dense, visually appealing, flavorful, tasteful and of appropriate texture or consistency. North-east India has a vast diversity of underutilized tropical fruits grown in hot and humid sub-tropical regions. Considering the nutrition, availability, seasonality, taste, mass acceptability and postharvest needs, three tropical fruits of North-East India were purposively selected for the development of the fruit based leather, namely, papaya (*Carica papaya* L.), guava (*Psidium guajava*) and mirika tenga (*Parameria polyneura*). This study aimed at developing a standardized

method for the formulation of nutritious leather in terms of sensory acceptability and functional attributes.

2 Materials and Methods

2.1 Sample Collection

The fruits namely guava (*Psidium guajava*), papaya (*Carica papaya* L.), and *mirika tenga* (*Parameria polyneura*) were obtained from the local fruit market located in A.T. Road, Jorhat, Assam. The fruits collected were fresh, ripe and mature. The sugar was bought from a local grocery shop located in Gar-Ali, Jorhat, Assam. The experiments were conducted at the Food Science Laboratory, Department of Food Science and Nutrition, College of Community Science, Assam Agricultural University, Jorhat, Assam.

2.2 Preparation of Mixed Fruit Leather

In general, two steps are involved to achieve the mixed fruit leather. These are the addition of the necessary components such as sugar, preservatives, and dehydration. The process might vary depending upon the type of fruits used, additional ingredients, technology used for drying, temperature, time, etc. The fresh fruits were thoroughly washed to wash off any harmful and undesirable substances. The fruits were peeled with a stainless steel knife and the peels were discarded. Papaya and guava flesh was sliced into pieces after removing the seeds. The chopped pieces of the fruits were separately put into the mixer grinder to blend them into homogenous fruit puree. In the case of *mirika tenga*, the seed was removed and the pulp was used. After homogenizing 100 g of fruits, the TSS, pH and moisture content were determined. The fruit purees were mixed together in three different combinations in the ratio of 60:30:10; 50:30:20; 40:30:30 for papaya, guava and *mirika tenga* puree respectively. In all three formulations, the content of guava puree was purposely kept constant (30%) so as to maintain it as a source of natural pectin found in guava pulp. Thereby, papaya and *mirika tenga* were altered. The purees were heated in a heavy base iron kadhai at the medium flame on a gas stove. Sugar was added gradually and mixed thoroughly by continuous stirring. The sweetness of the fruits differs due to its ripeness. Hence, the amount of sugar to be added depends upon the ripeness. The total amount of sugar added was measured to obtain the desired TSS level of 25–30% for fruit leather. The mixture was heated up to 85 °C to inhibit the enzymatic activity in the fruits. The pH of the samples was adjusted through the addition of 0.2% citric acid during preparation. The citric acid was weighed and then dissolved in a small amount of puree to be added to the cooking mixture. The citric acid was

added after the mixture was taken off from the flame. The mixture was taken off from the flame after ensuring that the measured TSS reached the desired value. The mixture was poured gently as 1.00 cm thick layer in glass petri plates and placed onto the aluminum trays of the cabinet dryer at 60.0 ± 2.0 °C. Thereby, a constant moisture level of targeted 14–16% for fruit leather was reached for a drying time of around 12–15 h. This is as per the desired level of dryness of a fruit leather [6]. The leather was frequently monitored to check its dryness and softness until the desirable attributes of the leather were obtained.

3 Physico-chemical Analysis

3.1 Total Soluble Solids (TSS)

Total soluble solids are a crucial indicator of food quality since it quantifies the amount of soluble solids present in a liquid. It reveals how sweet a fruit or food item is and how the TSS influences its taste. The test is carried out by a refractometer in which the liquid fruit extract is spread across the detector in the form of a uniform thin film. Based on the ratio of the speed of light in a vacuum to the speed of light in the food sample, the values are reported in Brix% [7].

3.2 pH

The pH value of the fruit leather was determined with the digital pH meter (model EUTECH Instruments, A&D Company, Limited pH 700) in the laboratory of the Department of Food Science and Technology, Assam Agricultural University. About 2 g of the fruit leather sample was crushed into a paste using a mortar and pestle. It was dissolved in 10 mL of distilled water and stirred appropriately until complete homogenization was achieved. The resultant readings were noted. This procedure was carried out for both the samples.

3.3 Titratable Acidity

The titratable acidity is expressed as % citric acid and is determined by titration of a known amount of reconstituted sample solution with 0.1 N NaOH using phenolphthalein indicator. About 1 g of the sample was dissolved in distilled water and made up to a volume of 10 ml. The prepared solution of 10 mL was titrated against 0.1 N NaOH using 0.1% w/v phenolphthalein indicator solutions. The acidity was calculated and expressed as percent (anhydrous solution). This procedure was carried out

for both the samples.

$$\% \text{Titrateable Acidity}_{\text{(as citric acid)}} = \frac{\text{Titrate Value} \times N_2 \times V_2 \times V_o}{V_1 \times P} \quad (1)$$

where N_2 = Normality or molarity of sodium hydroxide NaOH, V_2 = Titer Volume, V_1 = Volume of extract used, V_o = Total volume of extract, P = Weight of the sample taken.

3.4 Moisture Content Analysis

The moisture analysis was conducted in the laboratory of the Department of Food Science and Technology, Assam Agricultural University, Jorhat with the digital moisture meter and MX-50 with the scale of 0.01%/Max 51. About 1 g of sample was taken to analyze the moisture content of the fruit leather. The result is obtained from the percentage evaluation of the difference between the wet weight and the dry weight of the sample.

3.5 Colour Analysis

The color analysis was conducted in the Department of Food Science and Nutrition, College of Community Science, Assam Agricultural University, Jorhat and determined by the Colorimeter (model HunterLab. Reston VA) being equipped with the sensor ColorQuest XE "CQX4284". Using a CR-400 measuring head, the illuminance value of D65/10 was represented in terms of L^* (darkness/whiteness), a^* (greenness/redness), and b^* (blueness/yellowness) by the instrument. For each colour parameter, multiple measurements were taken, and the mean of the triple values was determined for each leather sample. The device features three-dimensional colour differences using coordinates in a consistent colour space (Lab). Three orientations are identified by the letters L^* , a^* , and b^* in the uniform colour space: light to dark, red to green, and blue to yellow [8].

3.6 Sensory Analysis

Sensory analysis was conducted at the Sensory Evaluation Laboratory of the Department of Food Science and Nutrition, College of Community Science, Assam Agricultural University, Jorhat in order to reveal product acceptability and consumer satisfaction by the trained, the semi-trained and the untrained panel members. A sensory panel made up of 15 people was used in the sensory evaluation to assess

the organoleptic qualities of the developed fruit leather. The consumer acceptability of the leather sample was assessed using a hedonic test with nine levels (1: dislike extremely, 2: dislike very much, 3: dislike moderately, 4: dislike slightly, 5: neither like nor dislike, 6: liked slightly, 7: liked moderately, 8: liked very much, 9: liked extremely) for five parameters (1: appearance/color, 2: taste/ flavor, 3: smell/odor, 4: texture/mouth feel, and 5: overall acceptability). 7 trained people—which included the Faculty of College of Community Science, Assam Agricultural University, and 8 untrained people (students) of the University campus were involved. A ballot sheet was given to the panelists containing all the details for evaluation. The volunteer panelists were provided with the samples displayed on a plate, water cup, knife, tissue napkin, etc. The panelists marked down the scores on the ballot sheets and the results were eventually evaluated.

4 Results and Discussion

4.1 Total Soluble Solids (TSS)

The individual °Brix value of the fruit puree of papaya, guava and *mirika tenga* were 9.0 ± 0.1 , 7.0 ± 0.1 , 3.0 ± 0.1 °Brix respectively. The TSS of the blended puree was recorded after the addition of sugar. Sample 1 (S1) consisting of papaya, guava and *mirika tenga* in the respective ratio of 60:30:10 showed a TSS of was 28°Bx, Sample 2 (S2) in the respective ratio of 50:30:20 had 27.5°Bx and for Sample 3 (S3) in the respective ratio of 40:30:30 respectively had 30°Bx. The total soluble solids of the dried final products were higher than the fresh blends. The final TSS content observed for S1 was 58.2 ± 1.8 °Bx, 57.5 ± 1.24 °Bx for S2 and for S3 it was 58.8 ± 1.32 °Bx (Table 1).

Table 1 Total soluble solids (TSS) of the developed mixed fruit leathers (all the values are mean \pm SD of 3 independent determinants)

Sample code	Sugar (%)	TSS (°Bx) fruit blends before drying	TSS (°Bx) final product
S1 (60:30:10)	14.0	28 ± 0.5	58.2 ± 1.8
S2 (50:30:20)	13.4	27.5 ± 1.4	57.5 ± 1.24
S3 (40:30:30)	15.0	30 ± 0.3	58.8 ± 1.32

Table 2 pH of the developed mixed fruit leathers (all values are mean \pm SD of 3 independent determinants)

Sample code with formulation	Sugar (%)	pH
S1 (60:30:10)	14.0	5.60 \pm 0.03
S2 (50:30:20)	13.4	5.54 \pm 0.04
S3 (40:30:30)	15.0	5.52 \pm 0.08

4.2 pH

The pH is the hydrogen potential and indicates the degree of concentration of hydrogen ions and thereby the acidity level of the food product. The determination of pH of food products is important as it determines the time and temperature at which the food should be handled, stored, and processed for the effective elimination of microbiological and enzymatic activity. Thereby, it can be ensured that the food is restricted from deterioration and spoilage [9]. The highest pH value (5.60 \pm 0.03) was observed in S1 and the lowest pH value (5.52 \pm 0.08) was observed in the fruit leather, S3 prepared with papaya, guava, and *mirika tenga* in the respective ratio of 40:30:30 and 15 percent sugar. The hydrocolloid addition aids in the reduction of the pH level of fruit leather [10]. Therefore, the marginal differences in the pH are attributed to the variations in the amount of sugar added to the fruit blend during the puree processing phase (Table 2).

4.3 Titratable Acidity

The titratable acidity assay determines and predicts the impact on the flavor constituent on the total acidity in foods. The titratable acidity conducted for S1 consisting of papaya, guava, and *mirika tenga* in the proportion of 60:30:10 respectively derived as 1.52 \pm 0.02 per cent acidity in 1 g of the sample whereas, the same carried out for S2 consisting of papaya, guava, and *mirika tenga* in the proportion of 50:30:20 respectively were derived as 1.684 \pm 0.24 per cent acidity in 1 g of the sample. For S3 consisting of papaya, guava, and *mirika tenga* in the proportion of 40:30:30 respectively the value was 1.578 \pm 0.32 per cent. Therefore, the acidity in 1 g of S2 was found to be the highest while the acidity in 1 g of S1 was the lowest (Table 3).

Table 3 Titratable acidity of the developed mixed fruit leathers (all values are mean \pm SD of 3 independent determinants)

Sample code with formulation	Sugar (%)	Titratable acidity in 1 g sample (%)
S1 (60:30:10)	14.0	1.52 \pm 0.02
S2 (50:30:20)	13.4	1.684 \pm 0.24
S3 (40:30:30)	14.07	1.578 \pm 0.32

Table 4 Moisture content analysis of the developed mixed fruit leathers (all values are mean \pm SD of 3 independent determinants)

Sample code with formulation	Sugar (%)	Moisture content (%)
S1 (60:30:10)	14.0	15.72 \pm 0.82
S2 (50:30:20)	13.4	15.18 \pm 0.81
S3 (40:30:30)	14.07	14.07 \pm 0.76

Fig. 1 Functional properties of the fruit leather



4.4 Moisture Content Analysis

The final moisture content of the samples was recorded till a constant level was reached and is presented in Table 2. The minimum moisture content (14.07 \pm 0.76) was observed for S3 and maximum for S1 (15.72 \pm 0.82) and at the same drying temperature (60.0 \pm 2.00 °C). The leather with the highest amount of sugar (15%), S3, had the lowest moisture content. It was also found that the addition of solids reduced the moisture content of persimmon fruit leather [11]. The reduction in moisture level due to the addition of hydrocolloids could be due to the effect on water holding capacity of fluid and semisolid foods [12] (Table 4; Fig. 1).

4.5 Colour Analysis

The color of any developed product is of paramount importance in the context that it influences consumer preference. For each leather sample, the mean of the three duplicate values for each colour parameter was determined. The developed mixed fruit leathers can be described as yellowish-brown. The colour assay derived for S1 consisting of papaya, guava, and *mirika tenga* in the proportion of 60:30:10 respectively was derived as the mean of the following values of the index values and were measured as Lightness (L*) as 48.38 \pm 0.43, redness (+a*) as 8.23 \pm 0.86, yellowness (+b*) as 11.46 \pm 1.16. The colour assay derived for S2 consisting of papaya, guava, and *mirika tenga* in the proportion of 50:30:20 respectively was derived as the mean of the following values of the index values and were measured as lightness (L*) as 48.73 \pm 0.72, redness (+a*) as 8.03 \pm 1.04, yellowness (+b*)

Table 5 Colour analysis of the developed mixed fruit leathers (all values are mean \pm SD of 3 independent determinants)

Sample code with formulation	Sugar (%)	Lightness (L*)	Redness (+a*)	Yellowness (+b*)
S1 (60:30:10)	14.0	48.38 \pm 0.43	8.23 \pm 0.86	11.46 \pm 1.16
S2 (50:30:20)	13.4	48.73 \pm 0.72	8.03 \pm 1.04	11.66 \pm 0.80
S3 (40:30:30)	15.0	42.36 \pm 0.43	8.43 \pm 0.86	10.66 \pm 1.06

as 11.66 \pm 0.80. The colour assay derived for S3 consisting of papaya, guava, and *mirika tenga* in the proportion of 40:30:30 respectively was derived as the mean of the following values of the index values and were measured as lightness (L*) as 42.36 \pm 0.43, redness (+a*) as 8.43 \pm 0.86, yellowness (+b*) as 10.66 \pm 1.06. Few previous studies have reported that the differences in colour parameters are related to the types and amounts of the constituents present in the product [13]. The increased sugar concentration in the combined fruit pulp content caused the leather to achieve a brown colour and indicated alterations in the product's hue (Table 5).

4.6 Sensory Analysis

The sensory analysis of any newly developed food product is crucial for consumer satisfaction and acceptance prior to its successful commercialization of the product in the market. During sensory analysis, trained sensory panelists must distinguish between and describe both quantitative and qualitative sensory features [14]. These aspects are important to be quantified so as to critically understand the food and eating behavior of consumers. A nine-point hedonic scale that posits participants' preferences exist on a continuum and that their responses being divided into like and dislike was used to conduct the sensory study.

The sensory analysis of the developed mixed fruit leather was conducted with the help of a 9 point hedonic scale and the results are presented in Table 6 (Figs. 2, 3 and 4).

Table 6 Sensory evaluation scores for different mixed fruit leather (9 point Hedonic scale)

Sample code with formulation	Sugar (%)	Colour	Taste	Aroma	Texture	Overall acceptability
S1 (60:30:10)	14.0	7.8 \pm 0.67	8.0 \pm 0.65	7.4 \pm 0.63	7.3 \pm 0.83	7.8 \pm 0.61
S2 (50:30:20)	13.4	7.0 \pm 0.80	7.0 \pm 0.65	6.87 \pm 0.64	6.73 \pm 0.59	7.4 \pm 1.23
S3 (40:30:30)	15.0	7.8 \pm 0.67	6.4 \pm 0.99	7.2 \pm 0.7	7.4 \pm 0.71	6.3 \pm 1.23

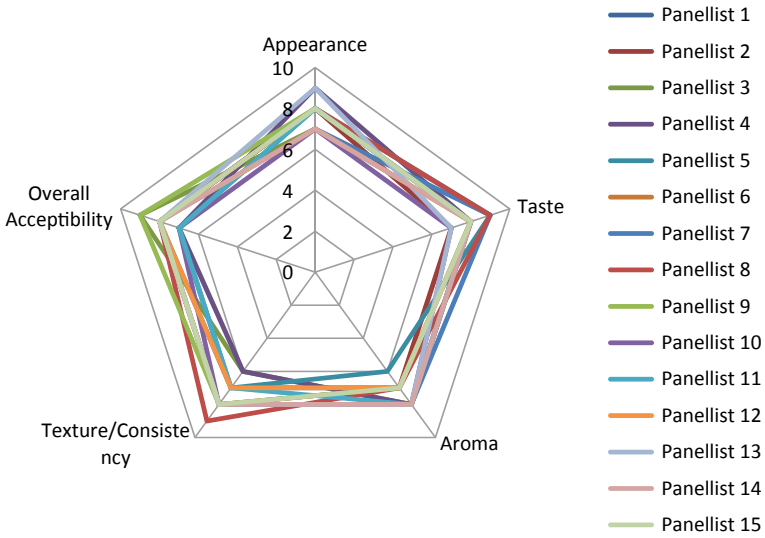


Fig. 2 Sensory analysis evaluation of the fruit leather S1

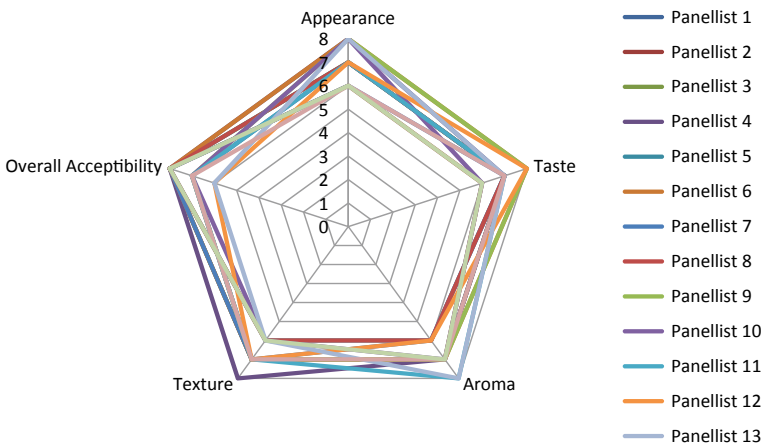


Fig. 3 Sensory analysis evaluation of the fruit leather S2

Colour. Colour is one of the most important parameter of the organoleptic properties of any newly developed food product as it has a vital impact on the consumers due to its ability to attract consumers. The colour of the food product is critical for evaluation as the appearance is determined mostly by the surface colour, and it is the first sensation perceived by the consumer. The colour preference index of both the fruit leather S1 (60% papaya: 30% guava: 10% *mirika tenga*) and the fruit leather S3 (40% papaya: 30% guava: 30% *mirika tenga*) were the same as 7.8 ± 0.67 while the

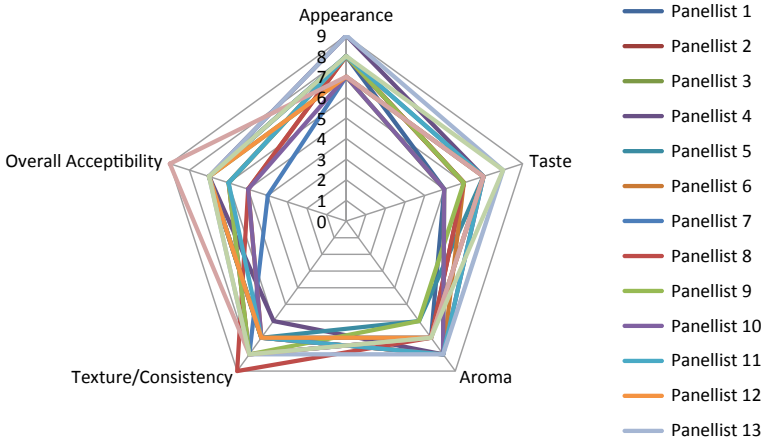


Fig. 4 Sensory analysis evaluation of the fruit leather S3

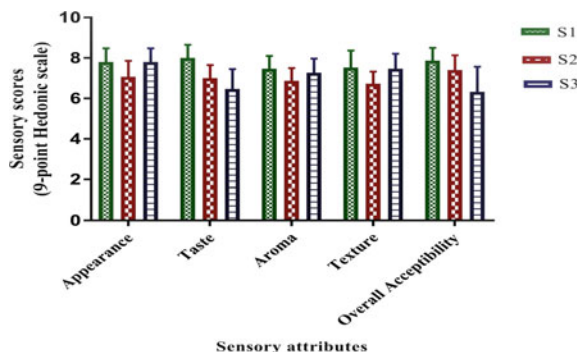
same for S2 was 7.07 ± 0.80 . Thus, the colour of the S1 and S3 was equally liked by the panelists in comparison with the S2 (50% papaya: 30% guava: 20% *mirika tenga*).

Aroma. Aroma depends upon the type and quantity of the volatile compounds present in the fruits. The aroma of the fruits also varies due to the components present in them. The odorant substances are volatile which are perceived by the receptors cells in the smell organ. The aroma of the fruit leathers differed from each other. The aroma preference means score of the mixed fruit leather S1 was 7.40 ± 0.63 whereas for S2, it was 6.87 ± 0.64 and for S3 it was 7.20 ± 0.7 . Therefore, the consumer preference based on aroma for the fruit leather S1 (60% papaya: 30% guava: 10% *mirika tenga*) was the highest while for fruit leather S2 (50% papaya: 20% guava: 30% *mirika tenga*), it was the lowest.

Taste. The taste was influenced by the composition, proportion, and species or variety of the fruits used. Although different fruits often share varied taste characteristics, each fruit has a specific and distinctive taste that depends upon the combination and concentration of the fruits used. The perception threshold of taste of every individual differs. As one of the most vital parameters of the food quality, for every individual, the taste varies due to the sensitivity to taste which is determined through the genetic protein receptors that exist in the taste buds cells of the tongue. The taste preference mean score of the mixed fruit leather S1 was 8.0 ± 0.65 whereas for S2, it was 7.0 ± 0.65 and for S3 it was 6.40 ± 0.99 . Therefore, the consumer preference for the taste of the fruit leather S1 (60% papaya: 30% guava: 10% *mirika tenga*) was the highest while the taste preference for the fruit leather S3 (40% papaya: 30% guava: 30% *mirika tenga*) was the lowest.

Texture/Consistency. The texture or consistency refers to the property of food that can be sensed or felt by touch, tongue, teeth or palate. The texture of the food is judged by the physical attributes of food during mastication. The texture profile

Fig. 5 Sensory evaluation of mixed fruit leather samples S1, S2 and S3



analysis is perceived in three stages of ingestion namely initial consisting of viscosity, hardness, brittleness; masticating consisting of gumminess, adhesiveness, chewiness, softness or hardness; and residual consisting of general mouth feel and breakdown rate [15]. The texture preference mean of the mixed fruit leather S1 was 7.30 ± 0.83 whereas for S2, it was 6.73 ± 0.59 and for S3 it was 7.47 ± 0.71 . Therefore, the consumer preference for the texture of the fruit leather S3 (40% papaya: 30% guava: 30% *mirika tenga*) was the highest whereas the texture preference for the fruit leather S2 (50% papaya: 30% guava: 20% *mirika tenga*) was the lowest.

Overall acceptability. The overall acceptability ranking varied for the freshly prepared fruit leathers achieved and with the inclusion of 10, 20, and 30 per cent *mirika tenga*. The overall acceptability of the mixed fruit leather S1 was 7.80 ± 0.61 whereas for S2, the overall acceptability was 7.40 ± 1.23 and the overall acceptability for S3 was 6.30 ± 1.23 . Therefore, the consumer preference for the fruit leather S1 (60% papaya: 30% guava: 10% *mirika tenga*) was the highest while the consumer preference for the fruit leather S3 (40% papaya: 30% guava: 30% *mirika tenga*) was the lowest (Fig. 5).

5 Conclusions

This article delineates upon the role of various factors in altering the physicochemical characteristics and sensory characteristics of several fruit leathers. Further studies could be carried out to evaluate the nutritional composition of the developed product and the bioavailability of nutrients. Packaging studies for long-term storage could also be carried out for sustainable production and marketing. The developed fruit leather affirmed a reduction in the moisture content and pH value. The sensory properties of the product showed that the fruit leathers' appearance (colour), flavour, and texture developed using papaya, guava and *mirika tenga* in the ratio of 60:30:10 showed the highest overall acceptability followed by the one with the respective ratio of 50:30:20. This study proves that mixed fruit leather made up of papaya, guava, and *mirika tenga* can be successfully developed with economically viable

resources. This research outcome can be applied for newer product development in terms of alternative and supplementary nutritional therapy. It might eventually open up avenues for societal development in terms of contribution to snacking for healthy aging. It would also provide impetus to the cultivation, production, and consumption of indigenous minor fruits of North-east India.

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Formulation and Characterization of Atta/Multigrain Atta-Aizong Rice-*Sechium edule* Flour Mix Baked Chips



Srimonti Dutta , Pankaj Kalita , and Ramagopal V. S. Uppaluri 

Abstract Through a comprehensive literature study, it has been analyzed that a significant research gap exists in the preparation and characterization of baked chips using combinations of grain and fruit flours. Henceforth, this was addressed in the conducted study. Composite flour-based chips were obtained using Aizong rice, atta (whole wheat flour), multigrain atta (multigrain whole wheat flour), and *Sechium edule* flour in different ratios, and optimization studies of baking parameters for chips preparation were conducted to achieve the best formulation. While, atta, multigrain-atta, and Aizong rice flour were varied in percentages of 0–100% (wt%), the *S. edule* flour was varied by 10–30% (wt%). Baking temperature and time were varied from 110–200 °C and 3–25 min, respectively. Experimental analyses were conducted to determine the moisture content, water absorption capacity, oil absorption capacity, swelling capacity, and bulk density for atta, multigrain-Atta, *S. edule* flour, and Aizong rice. Sensory analyses of best grain flour-based and fruit-grain flour-based baked chips were carried out. Most acceptable grain-based chips and grain-fruit flour-based chips were assessed in terms of color, crispiness, flavor, taste, hardness, aftertaste, and overall acceptance. Optimal chips formulations in both categories were determined by performing sensory analysis of the best chips obtained at variant combinations of baking process parameters.

Keywords Grain flour · Fruit flour · Chips · Formulation · Dough chips · Baking parameters optimization · North-East India

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Abbreviations

BD	Bulk Density
CAGR	Compound Annual Growth Rate
DPPH	2,2-Diphenyl-1-picrylhydrazyl
F	Formulation
GAE	Gallic Acid Equivalents
OAC	Oil Absorption Capacity
OTG	Oven Toaster Grill
QE	Quercetin Equivalents
RSM	Response Surface Methodology
SC	Swelling Capacity
SE	<i>Sechium edule</i>
SF	Sample Formulation
USDA	United States Department of Agriculture
WAC	Water Absorption Capacity

1 Introduction

Between June 2017 and April 2000, the packaged food industry became the 13th largest sector to receive direct foreign investment in India [1]. By 2023, the Indian snacks and confectionery industry is expected to expand at a compound annual growth rate (CAGR) of 11.5% [2].

Snack food products are relished and consumed by individuals of different ages and ethnicities around the world [3]. In the recent past, snack food enterprises have accelerated their efforts to introduce innovation in their product range to cater to customer desires. The Indian healthy snack food industry is expected to exceed INR 1 billion due to the intelligent snacking habits and lifestyle improvements that got emphasized after the onset of Covid-19 [4]. Current consumer awareness is reflected in changes in their buying habits, and today, items are carefully purchased based on their taste and nutritional content. As a result, there exists a demand for variety in nutritional dietary snacks. Furthermore, as people develop more mindfulness of their intake [5], individuals want healthier food options to be readily available for consumption. Aside from their nutritional value, functional foods provide numerous benefits to human health, such as reducing the chances of cardiovascular problems, aiding in digestion, diabetes, and controlling blood sugar [6, 7]. In the coming years, the Indian chips industry is predicted to increase at a CAGR of greater than 9% [8]. In contrast to the rapid growth of the chips industry, there has been a substantial gap in the varieties of healthy chips in the Indian market.

Annually, India produces around 107.59 million tonnes of wheat. As the world's second-largest wheat producer, India accounts for 13.53% of global wheat production [9]. Wheat features a high constitution of carbohydrates, and thereby serves as an

excellent nutritional energy source. Wheat flour-based chips can be produced by mixing them with other flours and spices. The utilization of wheat in the formulation of chips will allow consumers to consume wheat in between meals and thereby add variety to traditional labor-intensive and meal-oriented products such as wheat-based products (roti or chapati).

Aizong rice is a rice variety indigenous to the states of North-Eastern India. To date, it has been consumed only in the form of cooked rice in the North-East Indian households. In an attempt to widen the scope of this rice in the rest of the Indian states and increase the commercial and value-added prospects of the grain variety in the Indian chips industry, Aizong rice flour has been used in this research along with other flours to optimally formulate baked chips.

Sechium edule, also known as Chayote, Squash, Isquash, Piskut, Sikut, Mirliton, Choko, Buddha's palm, and Chow-chow, is an edible fruit of the gourd family Cucurbitaceae. The *S. edule* plant is native to Central America's mountains, where it was initially tamed by the Aztecs [10]. *S. edule* has long been used in Mexico to treat renal disorders and regulate excessive blood pressure [11]. Although the plant is native to Mexico, there exists a significant variety in North-Eastern Indian states and notably in Mizoram, Meghalaya, and Sikkim [12, 13]. The plant is also widely planted in the above states as a significant home garden crop for both selling and self-utility [14]. According to WebMD, *S. edule* contains essential vitamins and minerals and is a rich source of fiber and carbohydrates [15].

SE is a fleshy fruit having a high moisture content (89–95 g/100 g dw) [16–20], and is consumed as a vegetable after cooking it in various ways in different parts of India. U.S. Department of Agriculture (USDA) has presented a comprehensive list of quantitative values of various nutritional parameters of raw, cooked without salt, and cooked with salt in their FoodData Central database [16]. In the presented findings, a comparative observation can be made from the variation in the values of nutritional components can be seen with respect to the cooking methodology and the addition of salt while cooking the raw *S. edule*. In relevant literature [17], fruits and roots of *S. edule* have been said to be major components of the Latin American diet and are an important exported agricultural product as well. A detailed study has been done in the above literature on the taxonomy, origin, uses and properties, diversity and genetics, breeding, production, ecology, agronomy, and limitations of *S. edule*. In another relevant literature [18], an investigation of physicochemical properties for 10 diversities of *S. edule* fruit has been conducted. They have reported the high moisture content of *S. edule* fruit which ranged between 89.3 and 94.2%, along with the determination of different nutritional parameters such as carbohydrate content, protein content, crude fibre, vitamin C, and mineral ash, which ranged from 4.12–4.98%, 0.77–1.05%, 4.88–5.89%, 14.6–22.3%, and 0.245–0.321, respectively. An investigation of the nutrient composition of *S. edule* fruits, leaves, and stems has been conducted in the relevant literature [19]. Through the analyses for determination of the values of dry matter, total ash, crude protein, ether extract, crude fiber, nitrogen-free extract, acid insoluble ash, calcium, and phosphorus, they were found to be 5.92%, 5.97%, 14.88%, 0.83%, 7.53%, 70.79%, 0.01%, 0.25%, and 0.93, respectively. From a carbohydrate and mineral-based study on *S. edule* conducted in the

relevant literature [20], it has been observed that the carbohydrate, dietary fiber, and mineral content of *S. edule* fruits vary significantly when analyzed with and without the peel. With peel, the total sugar, reducing sugar, non-reducing sugar, starch, and crude fiber were observed to be 6.09, 5.32, 0.77, 1.22, 5.59 g/100 g dw, respectively; whereas, without peel, the same nutritional elements were found to be 6.69, 6.42, 0.47, 1.56, and 4.41 g/100 g dw, respectively. Determination of the bioactive composition of *S. edule* fruits has been conducted in several literatures [21–25] in terms of antioxidant activity, flavonoids, phenolics, and carotenoids. These respectively altered as 6.1–1503.96 $\mu\text{g/ml}$, 0.15–2.33 g QE/100 g dw, 0.06–6.18 g GAE/100 g dw, and 0.0083–6.01 g BE/100 g dw. A summary of the nutritional, and bioactive properties of uncooked and cooked *S. edule* fruit has been presented in Table 1.

In this article, *S. edule* fruit has been dried, and ground, and the obtained flour was used in oven-baked chips formulations.

Table 1 Nutritional, and bioactive properties of uncooked and cooked *S. edule* fruit

Sl No.	Property	Parameter	Un-cooked	Cooked (without salt)	Cooked (with salt)	References
1.1	Nutritional (per 100 g)	Moisture (g)	89–95	93.4	93.4	[16–18]
1.2		Energy (kcal)	19–31	24	22	[16, 17]
1.3		Carbohydrates (g)	3.5–7.7	5.09	4.5	[16–18]
1.4		Soluble sugar (g)	1.66–3.30	–	–	[17]
1.5		Reducing sugar (g)	6.420	–	–	[20]
1.6		Non-reducing sugar (g)	0.470	–	–	[20]
1.7		Total sugar (g)	6.69	1.89	1.89	[16, 20]
1.8		Starch (g)	1.56–0.20			[16, 20]
1.9		Crude fibre (g)	4.88–7.53	–	–	[18, 19]
1.10		Total dietary fibre (g)	1.7–7.6	2.8	2.8	[16, 17]
1.11		Total lipids (g)	0.10–0.30	0.48	0.48	[16]
1.12		Proteins (g)	0.77–1.74	0.62	0.62	[16–18]
1.13		Crude protein (g)	14.88	-	-	[19]
1.14		Fat (g)	0.1–0.3	-	-	[17]
1.15		Ash (g)	0.245–5.870	0.38	0.97	[16–19]
1.16		Calcium (mg)	12–25	13	13	[16, 17, 19]

(continued)

Table 1 (continued)

Sl No.	Property	Parameter	Un-cooked	Cooked (without salt)	Cooked (with salt)	References
1.17		Iron (mg)	0.20–0.78	0.22	0.22	[16, 17]
1.18		Zinc (mg)	0.28–0.74	0.31	0.31	[16]
1.19		Magnesium (mg)	12–15.40	12	12	[16, 17, 19]
1.20		Phosphorus (mg)	4–60	29	29	[16]
1.21		Potassium (mg)	125–337.87	173	173	[16]
1.22		Sodium (mg)	1–3.6	1	237	[16]
1.23		Copper (mg)	0.12–0.87	0.11	0.11	[16]
1.24		Manganese (mg)	0.19–0.49	0.169	0.169	[16]
1.25		Selenium (μ g)	0.2	0.3	0.3	[16]
1.26		Vitamin C (mg)	7.7–22.3	8	8	[16–18]
1.27		Thiamin (mg)	0.025–0.030	0.026	0.026	[16, 17]
1.28		Riboflavin (mg)	0.029–0.040	0.04	0.04	[16, 17]
1.29		Pantothenic acid (mg)	0.249	0.408	0.408	[16, 17]
1.30		Niacin (mg)	0.400–0.500	0.42	0.42	[16]
1.31		Vitamin A (mg)	5	-	-	[17]
1.32		Vitamin B-6 (mg)	0.076	0.118	0.118	[16]
1.33		Folate, total (μ g)	93	18	18	[16]
1.34		Chlorine, total (mg)	9.2–14.64	10.5	10.5	[16]
1.35		Vitamin E (mg)	0.12	0.14	0.14	[16]
1.36		Vitamin K (μ g)	4.1	4.7	4.7	[16]
2.1	Bioactive	Antioxidants (DPPH, IC ₅₀ , μ g/ml)	6.1–1503.96	-	-	[21–24]
2.2		Flavonoids (g QE/100 g dw)	0.15–2.33	-	-	[21–25]
2.3		Phenolics (g GAE/100 g dw)	0.06–6.18	-	-	[21–24]
2.4		Carotenoids (g BE/100 g dw)	0.0083–6.01	-	-	[21, 23–25]

2 Materials and Methodologies

2.1 Materials

Grain-based composite flour: Aizong rice, Aashirvaad brand's whole wheat flour, i.e., atta (nutritional properties per 100 g—protein: 10.9 g, fat: 1.7 g, carbohydrate: 77 g, sugar: 5.4 g in 77 g, dietary fiber: 10.5 g) and Aashirvaad brand's multigrain atta (ingredients—wheat flour (atta): 90.9%, multigrain flour mixture: 9.1% (defatted soya flour (5.2%), oat flour (1.5%), psyllium husk powder (1.1%), degermed corn-flour (0.9%), bengal gram flour (0.9%); nutritional properties per 100 g—protein: 14.5 g, fats: 1.7 g, iron: 4.4 mg, vitamin B1: 0.5 mg, sodium: 10.8 mg, carbohydrate: 72.9 g of which sugar: 5 g, dietary fiber: 13 g) were used to prepare the composite flour.

Grain and fruit-based composite flour: Aizong rice, Aashirvaad atta, Aashirvaad multigrain atta, and *S. edule* flour were used to prepare the grain and fruit-based composite flour.

Spices: In composite flour mixtures, spices used were salt (brand—Aashirvaad), turmeric powder (brand—Everest), red chili powder (brand—Everest), black pepper powder (brand—Everest), ginger garlic paste (brand—Catch), sunflower oil (brand—Fortune), and rice bran oil (brand—Fortune).

2.2 Methodologies

Preparation of flours: Aizong rice flour and *S. edule* flour were prepared after procuring commercially available Aizong rice and *S. edule* grown locally in North-East India.

Aizong rice flour: Commercially available Aizong rice was bought from the Market Complex of IIT Guwahati, Assam. The rice was washed thrice under running water to remove all impurities and was kept for drying at room temperature for a period of 12 h. Thereafter, the cleaned and dried rice was ground in a mixer-grinder (Make: Philips, Model: HL 1606, Rating: 230 V, 50 Hz, 500 W) and sieved using a 335- μ m sieve to obtain the Aizong rice.

S. edule flour: The fruit, *S. edule*, was purchased from the market complex of the Indian Institute of Technology, Guwahati. Subsequently, it was cleaned with running water, and the water adhering to the surface was removed using a soft towel. The cleaned *S. edule* was peeled and sliced into sheets of thickness 1 mm with a stainless-steel slicer. From a comprehensive study on *S. edule* drying in relevant literature [26], where RSM-based numerical optimization was done to observe the drying parameters to obtain minimal moisture content and maximum vitamin C and antioxidant activity, the ideal drying parameters were determined to be 62.5 °C and 7 h. Following the above study, the so-obtained 1 mm *S. edule* slices were dried in a hot air oven by placing them in a single layer on steel trays for 7 h at 62.5 °C.

Table 2 A summary of trial and error based grain flour and grain flour-fruit flour combinations being targeted for the preparation of baked chips

Formulation	Composite flour composition
F1	100% Atta
F2	100% Aizong rice flour
F3	100% Multigrain-atta
F4	25% Atta, 75% Aizong rice flour
F5	50% Atta, 50% Aizong rice flour
F6	75% Atta, 25% Aizong rice flour
F7	80% Atta, 20% Aizong rice flour
F8	25% Multigrain-atta, 75% Aizong rice flour
F9	50% Multigrain-atta, 50% Aizong rice flour
F10	75% Multigrain-atta, 25% Aizong rice flour
F11	80% Multigrain-atta, 20% Aizong rice flour

Dried slices were ground using a mixer-grinder, which was intermittently kept to rest at regular time intervals to avoid substantial temperature rise and nutritional deterioration of powdered *S. edule*. Finally, the *S. edule* flour was obtained after sieving the powdered *S. edule* using a 335-micron sieve.

Composite flour dough preparation: Composite grain flours were prepared with alternate proportions of whole wheat atta, multigrain atta, and Aizong rice flour. These have been summarized in Table 2, and depicted in Figs. 1 and 2. The composition of the three flours was varied in levels—0, 20, 25, 50, 75, 80, and 100%.

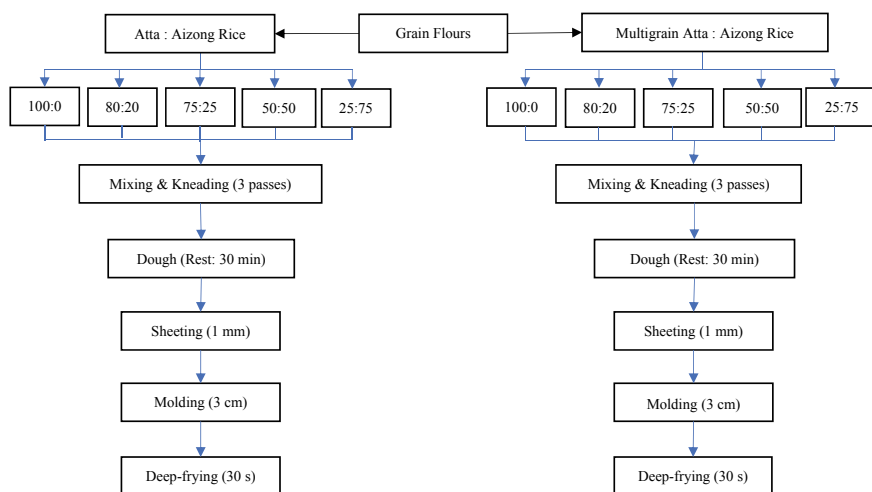


Fig. 1 Preliminary investigations targeting the optimization of atta, multigrain-atta, and Aizong rice flour ratios in the composite dough

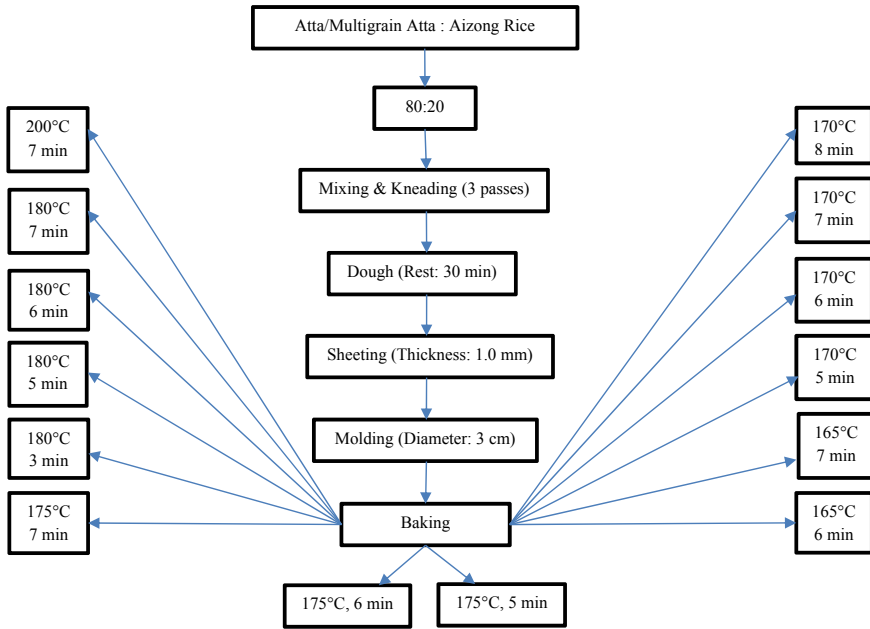


Fig. 2 A schematic of trial and error based baking process parametric optimization studies for the composite grain-flour based baked chips preparation

For the preparation of composite grain and fruit-flour dough, the best composite grain flour composition was identified through the baking process, and the atta/multigrain-atta was replaced with *S. edule* flour at 10, 20, and 30 wt% constitutions.

Homogenization of composite flour mixtures was carried out using a food processor (Home Plus Magic 400-W Atta Kneader) for 1 min. After mixing, 63% by weight drinking water and 3% oil were incorporated into the mix, and the mixture was kneaded for 5 min. The dough was then kept in a glass bowl and covered with a lid to prevent drying for 30 min.

Design of Experiments: Preliminary experiments were conducted by adopting a trial and error methodology with the deep-frying process. Thereby, composite flour mix and dough consistency range was evaluated for the successful formulation of chips. The procedure has been pictorially represented in Fig. 1. Considering the identified formulations that were confirmed for the successful formation of chips in the preliminary experiments, secondary experiments were conducted in an OTG (Oven Toaster Grill) to optimize the baking parameters. The procedure has been pictorially represented in Figs. 2 and 3.

After subjecting the composite flour dough to rest for proper hydration for 30 min at room temperature, it was rolled into 1 mm thin sheets using a pasta roller (Harivar Mart Stainless Steel 3 in 1 Pasta Maker Noodles Cutter Roller Machine). Thereafter, it was cut into chips having a diameter of 3 cm using a cookie cutter. The chips

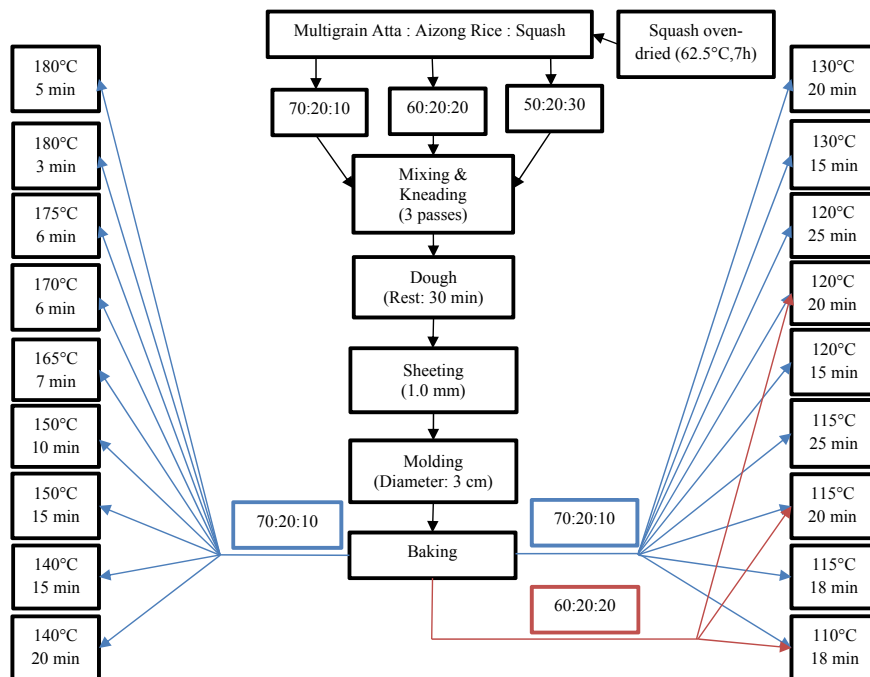


Fig. 3 A schematic of trial and error based baking process parametric optimization studies for the fruit and composite grain-flour based baked chips preparation

were placed in a single layer with even spacing on an aluminum foil atop a steel tray and loaded into an OTG (Philips HD6975/00 25 L Digital Oven Toaster Grill) for the baking process. Baking temperature and time were varied from 110–200 °C and 3–20 min, respectively, and thereby, these were optimized. Baked chips were cooled by placing them on tissue paper at ambient temperature and were then stored in ziplock bags.

Determination of properties. Physicochemical and functional properties in terms of moisture content, bulk density, water absorption capacity, oil absorption capacity, and swelling capacity of atta, multigrain atta, Aizong rice flour, and *S. edule* flour have been determined.

Determination of moisture content: The moisture content was assessed using the literature reported method of oven drying [27]. In this, the samples were pre-weighed and kept inside a preheated to 105 °C hot air oven for 14 h. Then, when the samples' weight reached a constant value, they were removed from the oven, and the final weight of samples was noted. Thereby, the respective moisture percentages were determined.

Water absorption capacity: The technique detailed in relevant literature [28, 29] was used to determine the water absorption capacity. 1 g sample flour and

10 mL distilled water were mixed and kept to rest for 30 min at room temperature. The mixture was then centrifuged at $2000 \times g$ for 30 min, and finally, relevant measurements were conducted.

Oil absorption capacity: The technique detailed in relevant literature [28, 29] was used to determine the oil absorption capacity. To do so, 1 g sample flour and 10 mL rice bran oil were mixed and kept to rest for 30 min at room temperature. The mixture was then centrifuged at $2000 \times g$ for 30 min, and finally, relevant measurements were conducted.

Swelling capacity: The technique detailed in relevant literature [30, 31] was used to determine the swelling capacity. Sample flour was taken and filled upto 10 mL in a 100 mL measuring cylinder. Thereafter, the rest of the volume was made up with distilled water and upto the 50 mL mark. The mouth of the measuring cylinder was covered, and the sample flour and water were mixed by inverting it. This was repeated after 2 min. Thereafter, with a resting period of 8 min, the volume occupied by the sample was noted.

Bulk density: The technique detailed in relevant literature [31] was used to determine the bulk density. 50 g sample flour was taken in a measuring cylinder. Thereafter, the cylinder was tapped till all volume variations got terminated. Finally, the weight per unit volume was evaluated to determine the bulk density.

Sensory analysis of chips: The sensory evaluation of formulated chips was conducted using the nine-point Hedonic scale [32]. The attributes evaluated were aroma/flavor, taste, appearance, color, crispiness, after-taste, and overall acceptability. For each formulated sample, panelists provided a rating of the above characteristics using the Hedonic scale. In this, the scores represented were: 1—dislike extremely, 2—dislike very much, 3—dislike moderately, 4—dislike slightly, 5—neither like nor dislike, 6—like slightly, 7—like moderately, 8—like very much and 9—like extremely.

3 Results

3.1 Physicochemical and Functional Properties of Flours

Experimental results of the functional properties of a variety of flours, namely atta, multigrain-atta, Aizong rice flour, and *S. edule* flour, have been provided in Table 3. The determined properties provide information on how the different flours would interact with water and oil and other factors that can be used to judge up on preparation of processed food items such as baked chips.

The analysis for the assessment of the quantity of moisture in the flour samples did convey that the highest value of moisture content was observed for atta (whole wheat) flour ($14.15 \pm 0.54\%$) and the lowest for *S. edule* flour ($10.80 \pm 0.65\%$).

The highest WAC was observed in *S. edule* flour ($350 \pm 15.45\%$) and the lowest in atta ($135 \pm 14.68\%$). Water absorption capacity or features describe a product's

Table 3 Physicochemical and functional properties of grain and fruit flours

S. No.	Property	Type of flour			
		Atta	Multigrain-atta	Aizong rice	<i>S. edule</i>
1	Moisture (%)	14.15 ± 0.54	12.51 ± 0.26	11.70 ± 0.57	10.80 ± 0.65
2	Water absorption capacity (%)	135 ± 14.68	210 ± 11.79	183 ± 12.44	350 ± 15.45
3	Oil absorption capacity (%)	142 ± 9.21	148 ± 10.16	121 ± 13.32	155 ± 14.19
4	Swelling capacity (%)	17.60 ± 1.81	17.15 ± 1.31	15.20 ± 0.83	1 ± 0.68
5	Bulk density (g/cc)	0.61 ± 0.12	0.63 ± 0.09	0.81 ± 0.16	0.69 ± 0.05

potential to interact with scarce quantities of water [33]. *S. edule* flour has a high water absorption capacity, which might imply that it has a significant quantity of fibers. Else, it might also be due to non-protein elements such as carbohydrates that can absorb and retain water. Water absorption capability is primarily a functional feature of proteins and is also influenced by carbohydrates and the flour's particle properties. An essential role of protein in flour is with respect to its ability to absorb water. Such a property of protein plays a crucial part in a variety of food items, such as baked food items and soups. These products necessitate upon strong protein and water interactions [34].

The highest value of OAC was observed for *S. edule* flour (155 ± 14.19%) and the lowest in Aizong rice flour (121 ± 13.32%). The ability of dietary protein to bind water and oil is determined by intrinsic characteristics such as amino acid content, protein structure, and hydrophobicity. Since *S. edule* flour has the most significant oil absorption capability, it may be superior to rice flour as a flavor retainer. The capacity of these flours' proteins to form a bonding with oil is that makes them suitable in the processing of food that demands maximum oil absorption. Oil absorption capability is another significant feature for the determination of the flour's appropriateness in aiding taste improvement and improved mouthfeel in due course of the food preparation. Oil absorption capacity is beneficial for fragrance preservation, enhanced palatability, and increased mean life of baked items [35]. However, the oil absorption capacity of *S. edule* flour is not very high. This is the desired metric of contemporary low-fat food trends.

The value of SC was found highest for atta (17.60 ± 1.81%) and lowest for *S. edule* flour (1 ± 0.68%). The swelling capacity of flour depends on particle size, processing method, and other factors. According to relevant literature [36], parboiled rice has a greater swelling capacity than raw rice. The swelling capacity of composite flours rose with rising levels of rice and reduced with increasing levels of wheat flour incorporation. This behavior is strongly correlated with the proportion of starch content in various kinds of flours. Such a feature is contributed by the enlargement

of starch granules as well as the resistance of the larger granules to thermal dissolution or fragmentation [37].

In the analysis for bulk density, which was conducted to observe the heaviness of the four different flours (atta, multigrain-atta, Aizong rice, and *S. edule*), similar densities (0.61 and 0.63 g/cc) were observed for the two types of wheat flours i.e. atta and multigrain-atta. Aizong rice flour scored the highest with a bulk density of 0.81 g/cc, while BD of *S. edule* flour was closer to the wheat flours with a score of 0.69 g/cc.

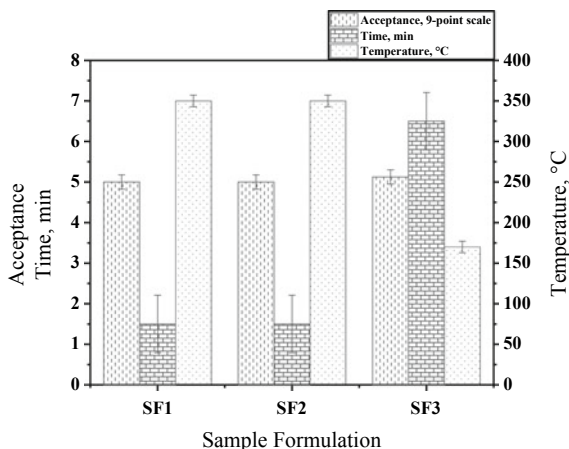
3.2 Formulation of Grain-Based Chips

Search for competent formulations based on deep-frying process: In the preliminary investigations, the primary focus was on varying proportions of atta, multigrain-atta, and Aizong rice flour in the dough to obtain an optimal formulation in terms of stickiness, rollability, and crispiness after preparing the chips. Atta, multigrain-atta, and rice flour were varied in percentages of 0–100%, and chips were prepared using the deep-frying technique. Dough formulations with good handling characteristics and with successful crispy chips were identified to constitute atta (75–80%): rice flour (20–25%) and multigrain atta (75–80%): rice flour (20–25%) as the possible formulation scope.

Identification of optimal formulations based on baking process parameters: It was noted that the hardness of chips increased with an increase in rice flour percentage. Hence, for further analysis and optimization of baking parameters, multigrain atta-Aizong rice flour percentages of 80 and 20%, respectively, were fixed. To optimize baking parameters, baking temperature and time were varied from 165–200°C and 3–9 min, respectively. The highest temperature at which crispy chips were obtained was at 180 °C for 7 min. However, the color was very dark. Hence, the temperature was reduced gradually, by maintaining the baking time of ± 2 min of that at which crispiness in chips was observed. Finally, crispy chips with good appearance and texture were obtained for the baking process parameters of 175 °C, 6 min, and 165 °C, 7 min.

Sensory Analysis: The sensory analysis of best grain-based chips in terms of properly cooked and crispy chips, consisted of atta, multigrain-atta, and Aizong rice flour, in SF1 (75% Atta, 25% Aizong rice flour), SF2 (75% Multigrain-atta, 25% Aizong rice flour), and SF3 (80% Multigrain-atta, 20% Aizong rice flour) formulations was conducted, and the results have been graphically depicted in Fig. 4. The sensory ratings given for the parameters of color, crispiness, flavor, taste, hardness, aftertaste, and overall acceptance have been observed to vary marginally for variations in formulations and baking process parameters. Best sensory ratings were obtained for SF3 (80% Multigrain-atta, 20% Aizong rice flour) formulations with baking process parameters of 165 °C, 7 min, and an overall acceptance of 5.25 ± 0.25 .

Fig. 4 Optimization of baking parameters and determination of best composite grain flour formulation based baked chips

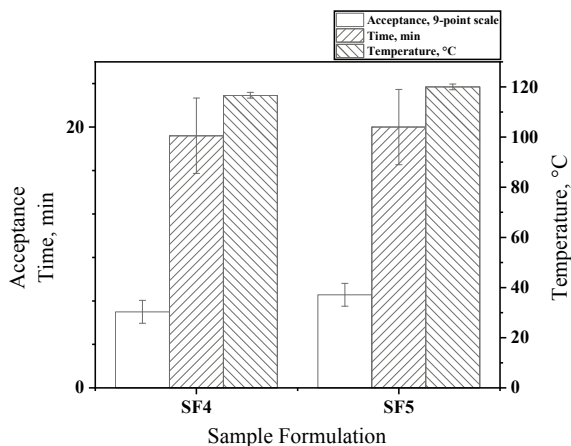


3.3 Formulation of *S. edule*-Based Chips

Identification of optimal formulation based on baking process: During the study of dough composition and process parameter optimization with SF2 and SF3, the best results were obtained with 80% multigrain atta and 20% rice flour, and the corresponding baking process parameters were 175 °C, 6 min, and 165 °C, 7 min. With the intention to gradually reduce the percentage of multigrain-atta, *S. edule* flour was added to the composite flour mixture in percentages of 10, 20, and 30% and thereby through the replacement of multigrain-atta. For the formulation with the composite mix, 70% Multigrain-atta, 20% Aizong rice flour, and 10% *S. edule* flour, the best results were obtained for baking process parameters of 120 °C, 20 min, and 115 °C, 20 min. Similar results were obtained when the composite flour mixture constitution of 60% Multigrain-atta, 20% Rice flour, and 20% *S. edule* flour. However, when a greater quantity of multigrain atta was replaced with *S. edule* flour in the composite mixture, (such as 50% Multigrain-atta, 20% Rice flour, and 30% *S. edule* flour), the dough handling capacity reduced as the stickiness increased and rollability decreased. Thus, the formulated dough could not be used for making chips.

Sensory Analysis: The sensory analysis of best fruit and grain flour-based chips was obtained in terms of properly cooked and crispy characteristics of atta, multigrain-atta, Aizong rice, and with *S. edule* flour. Thereby, SF4 (70% Multigrain-atta, 20% Rice flour, 10% *S. edule* flour) and SF5 (60% Multigrain-atta, 20% Rice flour, 20% *S. edule* flour) formulations were subjected to sensory analyses. The results have been graphically shown in Fig. 5. Best sensory ratings were obtained for chips made with formulation SF5 (60% Multigrain-atta, 20% Rice flour, 20% *S. edule* flour), with baking process parameters of 120 °C, 20 min, and with an overall acceptance of 6.75 ± 0.39 .

Fig. 5 Optimization of baking parameters and determination of best composite grain and fruit flour formulation based baked chips



4 Conclusions

Among the functional properties of the considered flours, the water absorption capacity of *S. edule* flour was observed to be significantly higher than the other flours. This might be due to its high fiber content. On the other hand, swelling capacity was almost negligible. This might be due to low starch content and necessitates further investigation. Atta and Aizong rice flour have low water and oil absorption capacities, and thereby confirmed upon their average quality as flavor retainers in processed food products. *S. edule* flour has higher water and oil absorption capacities without being too high in comparison to the considered grain flours. Thus, the fruit flour will be a better flavor retainer and will also aid in low-fat chips formulation. Due to this, *S. edule* flour incorporated formulation resulted in better-flavored chips, as well as aided in a higher score during the sensory analysis. The optimal *S. edule* flour percentage in composite flour mixture for the successful formulation of acceptable chips was found to be 20 wt%. While the addition of *S. edule* flour to grain flour led to a reduction in the hardness of chips as compared to only grain flour-based chips, they also resulted in darker chips. The addition of rice flour was observed to play a critical role in the crispiness and hardness of chips and was seen to be proportionally related. However, the incorporation of greater than 25% rice flour in the composite flour mixture resulted in crumbly dough consistency. It was noted that grain and fruit-flour-based chips required much lesser baking temperature and greater baking time than the solely grain-based chips.

During baking parameter optimization, it was noticed that the increase in baking temperature led to a darkening of color in chips, but the increase in baking time (till +2 min) at a constant temperature resulted in harder chips without significant change in the color of chips. The best formulation in terms of overall acceptability was found to be for the formulation SF5 (60% Multigrain-atta, 20% Rice flour, 20% *S. edule* flour), which consisted of the maximum possible percentage of *S. edule* flour integration in the formulation of wheat, Aizong rice, and *S. edule* flour-based healthy baked chips. Also, it shall be noted that while good quality grain flour-based

chips could be achieved at higher baking temperatures and shorter baking time, the fruit flour integrated chips were best achieved at lower temperatures and prolonged baking time. These alterations may not significantly reduce energy consumption but can aid in formulating baked chips with better nutritional characteristics.

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Development and Sensory Evaluation of Cookies from Nutrient Rich Composite Flour



Kasturi Pusty , Mriganka Shekhar Borah , Ajita Tiwari , and Sheriful Alam

Abstract The formulation of non-wheat flour from tuber-cereal-legume-oilseed combinations, as well as the replacement of gluten in cereal-based bakery products, remains a contemporary significant challenge. This article investigates the utility of composite flour in the development of cookies using tuber crops viz. sweet potato and taro. Sweet potato flour was blended with sorghum flour, chickpea flour and flax seed flour at the ratios of 80:10:5:5 (SP1), 70:15:10:5 (SP2), 60:20:15:5 (SP3), 50:25:20:5 (SP4), 40:30:25:5 (SP5). Also, composite blends of taro flour incorporated with sorghum flour, chickpea flour and flax seed flour in different ratios as 80:10:5:5 (TF1), 70:15:10:5 (TF2), 60:20:15:5 (TF3), 50:25:20:5 (TF4), 40:30:25:5 (TF5) were considered. The physicochemical and functional properties of composite flour confirmed significant differences. The sensory attributes evaluated in sweet potato-based composite flour cookies differed significantly. On the other hand, taro-based CF cookies exhibit no significant difference ($p > 0.05$) in appearance, color, flavour and texture. The formulation of composite flour cookies with improved nutritive and organoleptic properties was accomplished using blends of 60% sweet potato flour and 70% taro flour. Thereby, the methodology confirmed that it is possible to develop nutritious cookies with acceptable sensory characteristics using tuber-based composite flours.

Keywords Composite flour · Cookies · Nutrient · Health · Sorghum · Sensory · Tuber crops

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1 Introduction

Today, food consumers are becoming increasingly aware and hence worried about their health. Hence, they seek food items that enhance their immunity factors. They choose foods with high protein, carbohydrate and fiber content to preserve their health and mitigate possible risks such as cardiovascular disease, diabetes, weight gain, and so on. As a result, there is a new market trend to create a product that combines health advantages with good sensory features. In the present study, cookies from composite flour were prepared using flours of sweet potato, taro, sorghum, chickpea and flax seeds. The aim of the study was to develop nutrient enriched cookies with high constitution of carbohydrate, protein, fiber content. In numerous studies, composite flour has been defined as a blend of various flours derived from legumes, cereals, tubers, roots or other raw materials. These are used to make breads, pastas and baked goods and thereby develop traditional or new products [1]. Composite flour (CF) is best suited for cookie production than bread production. This is due to its ready-to-eat form, relatively long storability, widespread intake, and excellent taste. Also, the incorporation of composite flour is becoming essential due to its ability to impart enriching characteristics to any food product. Several researches revealed that the composite flour is suitable for producing bakery products [2]. A research group experimented with cookie recipes based on wheat and legume or cereals [3]. Utilizing composite flour prepared with millet, rice, soybean and tiger nut, a high quality and functional cookie was prepared along with the carboxyl methyl cellulose binder [4]. However, for the targeted combination of sweet potato or taro-based flour along with the flours of sorghum, chickpea and flax seed has not been addressed till date.

In this study two tuber crops were chosen i.e., taro and sweet potato. Taro (*Colocasia esculenta*) is one of the most extensively farmed edible aroids. It is a very important and old crop. It is high in phosphorus, vitamin C, calcium, iron, riboflavin, thiamine and niacin which are all essentially required for human nutrition. Colocasia has wide variability and a large number of local cultivars are grown in different parts of India. It has been reported that the flour obtained from corms and cormels through the drying and milling processes contains easily digestible starch. Thus, the said flours have been commonly deployed as infant food [2].

Sorghum (*Sorghum bicolor*) is an African grain being now grown all over the world as a staple food. It slows the growth of malignant tumors, protects against diabetes and insulin resistance, lowers cholesterol, and aids in the treatment of human melanoma. Sorghum grain is a vital food for millions across parts of Africa and Asia. It is one of the most important food and animal feed crops in arid and semi-arid regions of the world. Several potential economic, environmental and social benefits exist for the food grain. In most parts of the world, sorghum is a significant grain crop and a key dietary staple. The grain is an endemic crop to Africa. Many rural populations continue to rely on it as their sole source of nutrition. This is particularly true in the more drought-prone regions of the north India. In the country, the crop offers more

household food security than the maize. After wheat, rice, maize, and barley, the grain has fifth place in terms of its significance.

Chickpea (*Cicer arietinum*), a legume that provides several health benefits, was also used in this study. It is one of the oldest legumes known and cultivated from ancient times in both Asia and Europe. This pea is also known as garbanzo beans. Chickpeas are high in fiber and protein for vegetarians, and manganese for energy generation. It also has high iron content, blood sugar stabilization feature, and a low glycemic index (GI). Chickpeas can help to reduce LDL and total cholesterol in due course of its consumption on a regular basis.

Flaxseed (*Linum usitatissimum*) also known as linseed, is mainly considered as an oilseed crop. It is one of the oldest crop with abundance of beneficial nutrients. A good amount of omega-3 fatty acid, α -linolenic acid (ALA), dietary fiber, protein, lignin do exist in flaxseed. Arginine, aspartic acid, and glutamic acid are relatively abundant in flaxseed proteins, but lysine, methionine, and cystine are the limiting amino acids. Flaxseed contains nutrients called lignans that assist to mitigate cancer growth. As a versatile ingredient, the seed can enhance the taste and texture of almost any recipe.

Cookies are cheaper food products being consumed by almost everyone in society. Among other processed foods, some of the reasons for their widespread appeal include diverse taste, quick availability, longer shelf life, and low cost. Cookies are nutritious treats that transform unpalatable dough into an appetizing product with heat in an oven [2]. They provide significant source of energy due to being regarded as a concentrated food. This is due to higher carbohydrate, fat, and low moisture contents [3]. Maintenance of a product's sensory qualities is essential in the development of cookies with improved nutritional value. Hamdani et al. (2020) developed a rice-chickpea composite flour which has the potential to serve as a novel base material for baked foods. The finished product earned higher customer acceptability ratings in addition to being gluten-free. The substitution of chickpea flour for rice flour at a rate of 20% had a substantial influence on its physico-chemical properties [4]. However, it shall be noted the general acceptance of the consumers remains the most important factor for the evaluation of the applicability of a newly developed product [5]. The texture of cookies is mostly determined by the gelatinization of starch. Hence, no need arises for the formation of a gluten network. Further, cookies have several other advantages that emphasize upon their simple work through, easy and scalable throughput for manufacturing and delivery [6]. Cookies play a major role in the snack food industry. This is due to their unique taste, crunchy texture, and digestibility. Cookies are often baked either to be crisp or just be moist enough and remain soft. They are an excellent source of vitamin supplements [7]. Cookies can be baked in a variety of forms and with a range of ingredients such as chocolate, butter, peanut butter, almonds, or dry frost [8]. This article presents relevant research methodology as a part of a larger effort to encourage the use of CF, which are achieved from flour derived from locally farmed tuber crops and other flours such as sorghum flour, chick pea flour, flax seed flour. Thereby, the CF with high-nutrient content will impart similar characteristics to the baked cookies. The main approach of the study was to

develop nutrient-rich low cost cookies from composite flour and assess upon their physico-chemical, functional and sensory properties.

2 Material and Methods

2.1 Sample

Fully matured tubers of taro (*C. esculenta*) and sweet potato (*Ipomea batatas*) planted during the month February 2018 and harvested in January 2019 were obtained from the AICRP on Tuber Crops scheme (Horticulture), Assam Agricultural University and in the month of January. Chick pea (*C. arietinum*), sorghum (*S. bicolor*) flour and flax seed (*L. usitatissimum*) flour were procured from the local market.

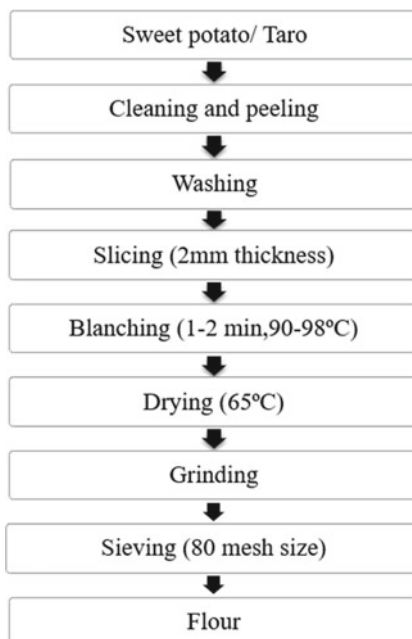
2.2 Methods

Prior to processing, sweet potatoes and taro corms were cleaned well using water and peeled with a sharp knife. After peeling, washing of the potatoes and taros were once again repeated. Thereafter, they were sliced into thin pieces of 2 mm thickness and were blanched for 2 min with distilled water (DsW) at 90–98 °C (Fig. 1). After 2 min, the slices were removed with a strainer and were spread on trays. The slices were dried in a tray dryer at 65 °C and for a time such that moisture content was 4–5%. Thereby, the dried sweet potato slices and taro slices were milled into flour using a grinder and the flour was sieved using 80 mesh sieve. The flour was kept in an air tight container for later use. The chickpea brought from the market were thoroughly washed to get rid of extraneous materials. Cleaned chickpea granules were then roasted in a pan for 10–15 min. The roasted granules were grounded in an electric grinder to achieve its powder. Thereafter, it was sieved with 80 mesh sieve. The obtained flour was stored in an airtight container for its later use. Sorghum and flax seed flours were procured from the market and were kept in air tight containers for the preparation of CF.

2.3 Formulation of Blends

Ten types of blends were prepared to achieve composite flour mix. A sweet potato-based blends contained sweet potato flour, sorghum flour, chickpea flour and flax seed flour in different ratios viz. 80:10:5:5 (SP1), 70:15:10:5 (SP2), 60:20:15:5 (SP3), 50:25:20:5 (SP4), 40:30:25:5 (SP5). Similarly, blends containing taro flour in different ratios viz. 80:10:5:5 (TF1), 70:15:10:5 (TF2), 60:20:15:5 (TF3), 50:25:20:5

Fig. 1 Process flow diagram for the preparation of sweet potato and taro flour



(TF4), 40:30:25:5 (TF5) were also prepared (Table 1). Thereafter, different blends of sweet potato-based and taro-based CF were used for cookie development. Using a 9-point hedonic scale, the developed cookies were then assessed for sensory attributes.

2.4 Determination of Physico-chemical Properties of Composite Flour

The CF mix were analyzed for their moisture, ash, crude fat, crude protein, crude fiber and total carbohydrate contents by using established analytical techniques [9]. The moisture content, crude fat content and crude fiber content were analyzed using AOAC methods [9].

Ash percentage: The ash percentage was calculated using the AOAC method [9]. To do so, a 2 g powdered sample was placed in a silica crucible, charred with a low Bunsen flame, and then ignited in the muffle furnace at 600 °C for 6 h. The ash percentage was determined by dividing the ash weight by the weight of the sample collected and multiplying the result by 100. The relevant expression is as follows:

$$\text{Ash content (\%)} = \frac{\text{Weight of the ash}}{\text{Weight of the sample taken}} \times 100 \quad (1)$$

Table 1 A summary of targeted sweet potato and taro-based composite flour formulations

Treatments	Flours					Treatments					Flours			
	Sweet potato (%)	Sorghum (%)	Chickpea (%)	Flax seed (%)		T1	T2	T3	T4	T5	Taro (%)	Sorghum (%)	Chickpea (%)	Flax seed (%)
S1	80	10	5	5		T1					80	10	5	5
S2	70	15	10	5		T2					70	15	10	5
S3	60	20	15	5		T3					60	20	15	5
S4	50	25	20	5		T4					50	25	20	5
S5	40	30	25	5		T5					40	30	25	5

Crude protein: The crude protein content of the CF flour was measured using the micro Kjeldahl technique [9]. To calculate total nitrogen, the nitrogen organic form in the sample was first converted into inorganic form (ammonium sulfate) through wet digestion with concentrated sulfuric acid in the presence of catalysts, potassium sulfate and copper sulfate, followed by ammonium sulfate decomposition by excess alkali (40% NaOH). After collecting the liberated ammonia in a 4% boric acid solution, it was titrated with normal hydrochloric acid (0.1 N). In a digestion tube, 500 mg was determined by titrating with 0.1 N HCl in the conical flask until the color altered from blue green to light pink. The relevant expression for crude protein estimation is as follows:

$$\text{Protein in g/100 g of sample} = \frac{(\text{Titre value} - \text{Blank}) \times \text{N of H}_2\text{SO}_4 \times 14}{\text{Weight of the sample} \times 1000} \times 100 \times 6.25 \quad (2)$$

Total carbohydrate: Anthrone method was used to determine the total carbohydrate content of the samples [10]. In a boiling tube, hundred milligram of each sample was placed and 5 mL of 2.5 N HCl was added. Then, by neutralizing it with solid sodium carbonate, the effervescence was stopped. The volume was increased to 100 mL before centrifugation. After filtering the supernatant, distilled water (DsW) was added in the test tubes and the supernatant was divided into 0, 0.2, 0.4, 0.6, 0.8, 1 mL. Anthrone reagent (4 mL) was added in the sample tube and boiled for eight min. The absorbance was measured at 630 nm in a spectrophotometer. A standard graph was prepared for the absorbance versus standard concentration. The total carbohydrate content in the composite flour was evaluated from the graph. The investigation was carried out in triplicate, and the mean value was noted. Thereby, the total carbohydrate index was determined using the expression:

$$\text{Total carbohydrate in 100 mg of the sample} = \frac{\text{mg of glucose}}{\text{volume of test sample}} \times 100 \quad (3)$$

2.5 Functional Parameters of Composite Flour

Water absorption capacity (WAC): The flour WAC was determined using the procedure outlined by Sosulski et al., 1976 [11]. In ten mL DsW, 1 g sample was dissolved and kept at 30 °C for half an hour. Thereafter, centrifugation was conducted at 3,000 rpm or 2000 × g for 30 min. The WAC was measured using the fraction of water bound per gram flour and with the expression:

$$\text{WAC} = \frac{\text{weight of water absorbed (ml)}}{\text{sample weight (g)}} \quad (4)$$

Oil absorption capacity (OAC): For OAC determination, 1 g sample was combined with soybean oil of 10 mL and was kept aside at 30 °C for half an hour. Thereafter, centrifugation at 30 min or 2000 g × for 3000 rpm was conducted [11]. Prior to the reweighing the tubes, the oil was extracted. The oil was then drained from the tubes by keeping them inverted for 30 min. Then, the OAC was determined by calculating the amount of oil bound per gram of sample and with the expression:

$$\text{OAC} = \frac{\text{weight of the sample after extracting oil} - (\text{weight of centrifuge tube} + \text{weight of the sample})}{\text{weight of the sample}} \quad (5)$$

Foam capacity (FC) and Foam stability (FS): The FC and FS were determined using the method described by Narayana et al., 1982 [12]. In a graduated cylinder, fifty mL of DsW was poured and 1.0 g of CF flour was mixed. To achieve foaming, the mixture was thoroughly agitated for 5 min. After 30 s whipping, the foam volume of TF was expressed as FC and with the expression:

$$\text{FC \%} = \frac{\text{Volume of foam after whipping} - \text{volume of foam before whipping}}{\text{volume of foam before whipping}} \times 100 \quad (6)$$

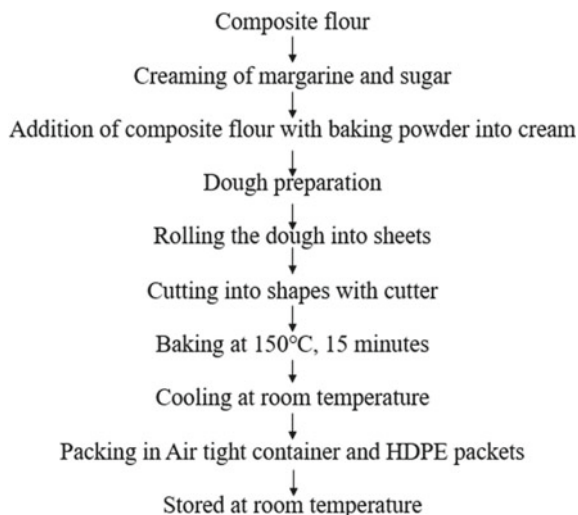
To determine foam stability, after one hour of whipping, the foam volume was determined and expressed as FS.

Water solubility (WS): The WS of CF was analyzed using the method given by Anderson et al., 1969 [13]. In thirty mL of DsW, the flour sample (2 g) was dissolved and was boiled at 90 °C for 15 min. Eventually, the sample was cooled and poured to centrifuge tubes, and was mixed at 3000 rpm for 10 min. To determine the solid content, the supernatant was poured into an evaporating basin and the weight of the residue was noted. The dry solid weight was obtained by drying the supernatant at 110 °C. Thereby, WS was determined using the expression:

$$\text{WS\%} = \frac{\text{weight of dissolved solids in supernatant}}{\text{weight of flour sample}} \times 100 \quad (7)$$

Swelling capacity (SC): The swelling capacity (SC) of CF was determined using the method given by Okaka et al., 1977 [14]. To do so, 10 mL of the CF sample was poured in a 100 mL graduated cylinder. To make up the final volume, 50 mL of distilled water was added. To mix the sample, the graduated cylinder was turned over and tightly covered. Again after 2 min, the suspension was flipped and left to remain stagnant for another 8 min. After eight mins, the volume was determined to represent the SC [14].

Fig. 2 Flow chart for the preparation of composite flour-based cookies



2.6 Preparation of Cookies

The Cookies were developed from sweet potato and taro-based CF by following the procedure reported by Barooah et al., 2015 [15]. Margarine (75 g), sugar (40 g), baking powder (2 g), salt (1 g) were used as fixed ingredients for the development of 100 g of composite flour-based cookies. The procedure has been depicted in Fig. 2.

2.7 Sensory Assessment of Developed Cookies

The developed cookies from sweet potato and taro-based CF were subjected to organoleptic examination with 15 judges. For each cookie sample, the judges panel evaluated its sensory characteristics using a hedonic rating scale of 1–9 (with 1 denoting extreme dislike and 9 denoting extreme like). Quality characteristics such as appearance, color, flavor, texture, taste, and general acceptance were assessed for the developed cookies. The sensory scores were statistically analyzed using Analysis of Variance (ANOVA).

2.8 Statistical Analysis

The variance analysis of the acquired data was performed using completely randomized design (CRD) for various treatments and in accordance with AOAC, 2000 [5]. The significance was confirmed with 5% level of significance ($p < 0.05$).

3 Results and Discussion

3.1 Physico-chemical Analysis

For the alternate samples of composite flour (TF1–TF5 and SF1–SF5), a substantial variation was noticed in the proximate composition (moisture, ash, crude protein, crude fat, crude fiber and total carbohydrate). While sweet potato-based CF (Fig. 3) had a moisture content of 5.66–6.51%, the taro-based CF had a moisture content of 6.59–8.87%. Thus, when different flours were mixed in varied ratios, the moisture content of sweet potato-based CF and taro-based CF gradually increased.

For the SP1–SP5, (sweet potato-based CF formulations) the ash content altered as 2.46–2.06%. However, in taro-based CF formulations, the ash content ranged between 5.23 and 3.43% (TF1–TF5). These trends were due to the increased contribution of sweet potato and taro and their rich mineral content in comparison to sorghum, chickpea and flax seed flour. The protein content increased with reduced content of sweet potato and taro.

The parameter altered 4.88–7.38 g/100 g in sweet potato-based composite flour and 6.62–8.85 g/100 g in taro-based CF (Fig. 4). Due to enhanced chickpea and sorghum constitution, the protein level may have increased in the later formulations. With mature amino acid constitution, the chickpeas serve as an affordable source of protein. The fat content in sweet potato & taro-based CF ranged from 4.13 to 6.50% and 5.50 to 7.16% respectively. This could be due to the fact that unlike sorghum, chickpea and sweet potato, the taro uses starch to store its energy. The addition of

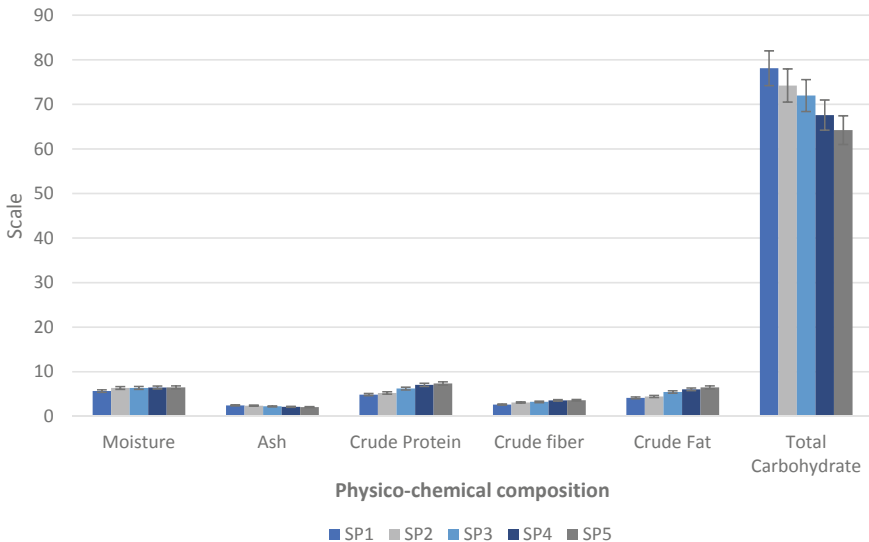


Fig. 3 Physico-chemical characteristics of sweet potato-based composite flour

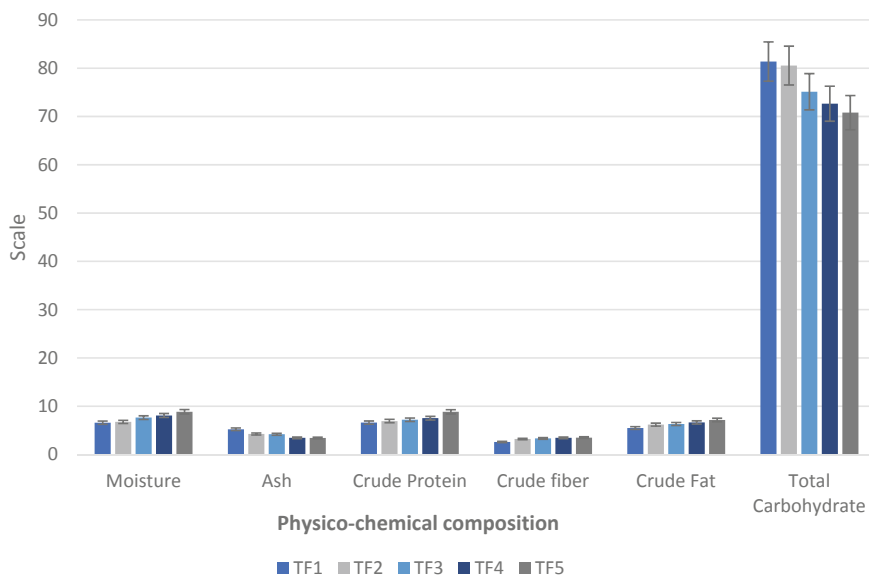


Fig. 4 Physico-chemical properties of taro-based composite flour

chickpea and sorghum flour, which have a little greater fat content than sweet potato and taro flour, could be attributed for the increasing trend in the CF.

The crude fiber content of sweet potato-based CF was between 2.63 and 3.61%. For taro-based CF, these were 2.61–3.51% (Fig. 4). An increasing trend was observed in crude fiber content for sweet potato and taro-based composite flour. The trend is directly correlated with the percentage incorporation of sorghum and chickpea flour. In human metabolism, carbohydrates are primarily needed to provide energy besides other metabolic functions. In the present investigation, the total carbohydrate content of sweet potato-based composite flour (Fig. 3) altered from 78.11 to 64.21 g/100 g. For taro-based composite flour, the parameter altered as 81.37–70.80 g/100 g. The decreasing trend in the carbohydrate content in the test sample could be due to the reduced inclusion of sweet potato and taro proportion. This is due to the fact that both sweet potato and taro are rich in carbohydrate.

3.2 Functional Parameters

The analyzed functional parameters of CF were WAC (mL/g), OAC (mL/g), FC (%), FS (%), WS (%), SC (%). The WAC of sweet potato-based composite flour (Table 2) varied as 2.64–2.86 mL/g. In the taro-based composite flour, the parameter altered as 3.17–3.85 mL/g. Higher WAC can be attributed to the presence of more hydrophilic components. The WAC increasing content could be due to the comparatively richer

Table 2 Functional parameters of sweet potato-based composite flour

Treatments	WAC (mL/g)	OAC (mL/g)	FC (%)	FS (%)	WS (%)	SC (%)
SP1	2.64	1.43	4.33	1.51	11.80	13.30
SP2	2.69	1.92	6.39	2.36	14.63	12.40
SP3	2.72	2.43	8.89	3.65	16.30	9.36
SP4	2.76	3.36	12.40	4.53	17.48	5.26
SP5	2.85	3.49	13.71	5.80	18.25	4.36
S.Ed(\pm)	0.02	0.04	0.11	0.05	0.19	0.19
CD (5%)	0.06	0.07	0.26	0.13	0.44	0.43

protein and carbohydrate content of chickpea and sorghum flour. The OAC of sweet potato and taro-based composite flour was altered as 1.43–3.49 mg/mL and 1.04–2.49 mg/mL respectively. An increasing trend in the OAC was observed. This can be primarily attributed to the fat and fiber content in sorghum, chickpea and flax seed flour. The capacity of the flour to froth is based on the availability of flexible protein molecules that lower water's surface tension [16].

An increasing trend in FC was observed in sweet potato and taro-based composite flour. The parameter ranged from 4.33 to 13.71% and 7.89 to 15.56% respectively. As the degree of inclusion of chickpea and sorghum increased, the foaming capacity increased due to richer protein constitutions in the taro-based composite flour and for the case with higher constitution of sorghum and chickpea flour. Foam stability refers to a protein's capacity to tolerate gravitational and mechanical stress. For the sweet potato-based composite flour, the WS was measured to alter as 11.80–18.25% (Table 2). For taro-based composite flour, the WS altered as 14.43–16.39%. WS mainly depends on the protein molecules. The increased percentage inclusion of sorghum and chickpea results in an increase in the water solubility. SC of flours mainly depends on the starch and amylose content. The swelling capacity of sweet potato-based composite flour mix and taro-based composite flour decreased significantly in the present investigation. The SC ranged from 4.36 to 13.30% in sweet potato-based composite flour and 5.43–9.76% in taro-based composite flour (Table 3). Protein and starch levels are usually associated to swelling capacity [17]. The decreasing trend may be attributed to the inclusion of sweet potato and taro flour in the CF. This is due to the reason that starch is one of the prevalent major macromolecules in sweet potato and taro. Both these swell up due to the imbibing of water. An increased protein content in flour may result in starch granules getting trapped inside a hard protein network. Thereby, this phenomenon restricts the starch's ability to absorb water and reduces its potential to swell [18].

Table 3 Functional parameters of taro-based composite flour

Treatments	WAC (mL/g)	OAC (mL/g)	FC (%)	FS (%)	WS (%)	SC (%)
TF1	3.17	1.04	7.89	1.66	14.43	9.76
TF2	3.22	1.34	8.03	2.44	14.76	8.66
TF3	3.28	2.08	9.07	3.62	15.17	8.50
TF4	3.63	2.17	12.07	3.82	15.77	6.63
TF5	3.85	2.49	15.56	4.42	16.39	5.43
S.Ed(\pm)	0.03	0.03	0.04	0.06	0.11	0.22
CD (5%)	0.08	0.06	0.10	0.14	0.25	0.49

3.3 Sensory Analysis of Cookies

The results of the organoleptic evaluation of cookies developed from sweet potato-based CF were found to be non-significant (Table 4). Color is an essential sensory property of any product as it influences acceptance. Foods cooked in an oven always have a brown color due to the Milliard reaction. The color scores of sweet potato-based CF cookies (ranging from 6.60 to 7.55) and taro-based CF (ranging from 7.05 to 7.60) suggested that cookies were liked moderately by the panel. This corroborates with the observation of Iwe, 2007 [19]. The mean scores of appearance, color, flavor and texture of taro-based composite cookies were non-significant. However, the taste and overall acceptability are statistically significant. All cookies samples have been rated to be acceptable by the panel.

The overall acceptance of the developed cookies was assessed based on quality ratings of appearance, color, flavor, texture and taste. The graph of the overall acceptance scores indicated for sweet potato-based composite flour are SP1 = 6.70, SP2 = 6.25, SP3 = 7.80, SP4 = 7.15 and SP5 = 7.30. For the taro-based composite flour, these are TF1 = 6.90, TF2 = 7.90, TF3 = 7.20, TF4 = 7.60 and TF5 = 7.10 (Fig. 5). From the sensory data of all the cookies, the overall acceptability score for cookies containing 60% sweet potato flour (treatment SP3) and 70% taro flour (treatment TF2) were opined to be the best by the judges. These findings support inferences given by Hegazy et al., 2009 [20]. The developed cookies' overall acceptability scores showed no significant mean difference. All samples were rated above average and overall cookies quality at the different levels of substitution was found to be acceptable as per the recommendations of the sensory panelists.

4 Conclusions

Locally available crops can be utilized to make gluten-free CF with organoleptic and nutritive properties equivalent to commercial wheat flours [21]. The utilization of composite flours may be advantageous in developing nations due to the existence of

Table 4 Sensory analysis of SP1-SP5 and TP1-TP5 formulations-based cookies

Sensory evaluation	Sweet potato-based CF					Taro-based CF								
	SP1	SP2	SP3	SP4	SP5	S.Ed(±)	CD (5%)	TF1	TF2	TF3	TF4	TF5	S.Ed(±)	CD (5%)
Appearance	6.80	6.60	7.60	7.50	7.00	0.41	0.23	7.50	7.40	7.30	7.20	7.10	0.41	NS
Color	6.55	6.60	7.55	7.15	7.05	0.40	0.93	7.25	7.60	7.20	7.05	7.50	0.39	NS
Flavor	6.35	6.85	7.50	6.50	7.10	0.40	0.93	7.10	7.70	6.80	7.20	7.30	0.37	NS
Texture	6.80	6.20	7.50	7.30	6.90	0.38	0.87	7.20	7.45	6.95	7.00	7.40	0.39	NS
Taste	6.65	6.40	7.75	7.20	7.00	0.39	0.90	7.40	7.20	6.50	7.50	7.90	0.44	1.01

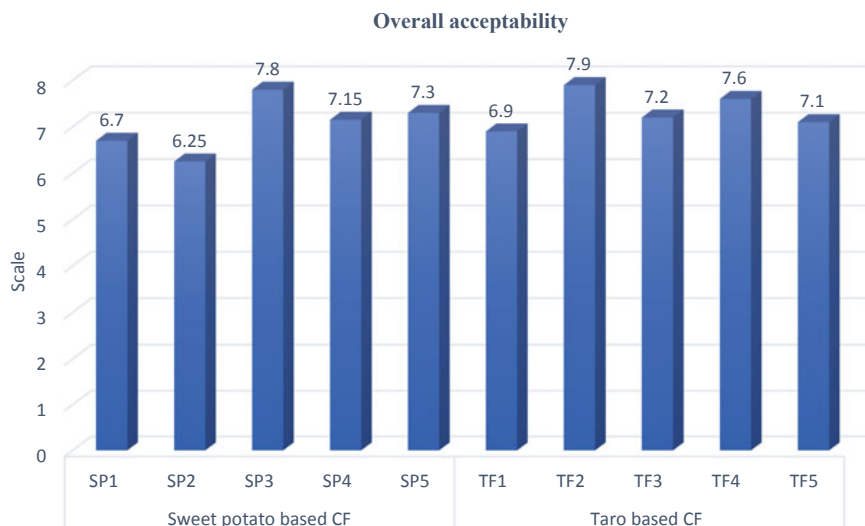


Fig. 5 Overall acceptability of SF1–SF5 and TF1–TF5 cookies

a large number of raw botanical sources. The creation of blends from locally grown crops may lead to increased usage of indigenous food crops in countries that import wheat to a significant quantity. Composite flour derived from tuber crops might be utilized as a novel base material in the production of bakery items. The developed cookies earned greater customer acceptability ratings in addition to being gluten-free. The developed tuber crops-based composite flour proved to be a good source of protein, fiber, carbohydrate and fat.

The moisture combined with other components provides good machining qualities to the dough and helps to regulate the expansion and spread of the dough during baking. From the conducted study, it is clear that the composite flour-based formulation SP3 (60% sweet potato flour, 20% sorghum flour, 15% chickpea flour and 5% flax seed) and TF2 (70% taro flour, 15% sorghum flour, 10% chickpea flour, 5% flax seed flour) were the best with very high overall acceptability scores in comparison to other formulations of cookies. The sensory attributes evaluated in sweet potato-based cookies showed significant difference. However, no significant difference ($p > 0.05$) existed in the appearance, color, flavor and texture of the taro-based CF cookies. The findings demonstrated that it is feasible to make nutritious and appealing cookies using tuber crop-based composite flour. These nutrient-dense cookies have the potential to significantly reduce malnutrition. Thus, commercial manufacturing of these cookies will provide excellent opportunities for entrepreneurial growth.

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Formulation and Characterization of Squash Enriched Cookies



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Abstract For many people living in urban regions, fresh fruits and vegetables are practically a fantasy in the contemporary circumstances due to time constraints and the risks inherent in procuring them. To profoundly boost human nutritional value, the inclusion of abundantly produced healthy fruits and vegetables in the region and as ready-to-eat snacks is certainly desired. Squash or Chayote (*Sechium edule*) is prevalent in mountainous regions of India and can be found throughout the year. The people of Northeast India extensively use this vegetable in a variety of forms, despite the fact that the peel is nothing more than a waste product containing rich nutrients. Dried squash powder can be used to improve the nutritional value of a wide range of dishes. Cookies that contain little or no gluten are becoming increasingly popular. It is possible to bake gluten-free treats using sugar-snap cookies, which are heavy in fat and sugar. Squash pulp and peel powder can serve as a good substitute to wheat flour for those with gluten sensitivities. In the current study, rice flour and varied proportions of squash pulp powder were combined to achieve squash-based cookies after analysing their functional properties and proximate properties. In comparison to fresh samples, dried powdered samples were found to considerably improve the nutritional characteristics such as ash, crude fibre content, fat, carbohydrate, and protein. The squash pulp and peel powder can be utilized for baked goods. This is evident in their functional characteristics such the capacity to absorb water and oil, the lowest gelatinization temperature, and the lowest concentration of gelation. Cookies were analysed for their sensory properties by utilizing a 9-point hedonic scale and fuzzy logic analysis. It was observed that the squash-based cookies with a rice:wheat ratio of 3:1 and 30% squash pulp powder substitution were the most

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preferred. With greater substitution of squash pulp powder, the amount of crude fibre and vitamin C increased while the amount of fat lowered. With greater substitution of squash pulp powder, the TPC values and antioxidant activity also increased.

Keywords Fuzzy logic · Gluten-free cookies · Squash peel powder · Squash pulp powder

1 Introduction

Belonging to the *Cucurbitaceae* gourd family, the edible plant *Sechium edule* (commonly known as chayote), bears green, pear-shaped fruits and tough tendrils that distinguish the perennial herb from other climbers. The ideal conditions for Chayote to thrive are mild temperatures, high relative humidity, evenly distributed annual rainfall, and 12 h of daylight [1]. It is easy to grow squash (chayote) due to its excellent ability to acclimatize to an extensive choice of environmental surroundings. As one of the cheapest vegetables in India, the prominent home garden crop in Northeast India, is also widely cultivated for both commercial and personal consumption [2]. Squash is an excellent source of fibre, protein, fat, minerals, and vitamins, especially vitamins A and C [3]. Due to its fragile flesh's short shelf life and severe perishability, it has a substantial negative impact on productivity. In the recent years, the fruits of this tree, which are becoming increasingly popular in the world as a vegetable, have been exported.

Any perishable vegetable or fruit that is being exported can be dried to reduce its water content and enhance its shelf life. Additionally, drying prevents microbial attack-related breakdown, giving dried goods with an extended storage life. Such dried items can hence be available in the entire year without losing their nutritional composition. For such drying, a wide variety of drying methods can be deployed, including sun, tray, oven, shadow drying etc. As a thumb rule, shadow drying takes a long time, whereas sun drying is detrimental to the preservation of vegetable nutritional values. Heat treatment and drying environments can adversely affect several nutritional components. As a result, it's important to adapt a drying process that effectively preserves nutritional content of the fruit at a lower cost. For the North-east India, horticultural produce drying in a hot air oven or a tray dryer would be a feasible inexpensive option to reduce their water content. Dried vegetable powders can be used to serve as key constituents in several food products, including bakery and confectionary items.

Due to their high nutritional value and low cost, cookies are popular. Rapid population expansion, enhanced influence of the Western countries habitat in food consumer products, the emergence of a female working population, and changing eating patterns have all contributed to enhanced cookie and biscuit production. Due to a rise in health awareness of foods, a strong demand exists for healthier cookie products. In the recent years, consumer interest in wheat-free meals (*Triticum aestivum*) has steadily increased to reduce the risk of celiac disease and associated disorders

that is still largely unknown [4]. Since wheat flour contains gluten, patients with celiac disease cannot eat baked treats or other dishes cooked with it. A variety of flours can be used to supplement gluten, which significantly influence and determine the cookie's processability and end productivity.

Sensorial assessment process is a scientific strategy for eliciting, measuring, assessing, and interpreting the product's sensory reactions. The five sense organs of the human beings generally respond to the associated stimuli and reactions [5]. On a scale of 9 that corroborate to "like highly" and "dislike excessively," a hedonic scale rating test is often utilized to assess upon the degree of pleasurable and unpleasurable cookie consumption experience. For this purpose, evaluation form is provided to experienced panelists with a number ranking system that contained several sensory criteria and scoring options. In due course of evaluation, the panelist data is averaged and tabulated. Taste, texture, colour, appearance and general acceptability have all been considered in due course of the sensory analysis-based rating of the cookies. Also, basic fuzzy logic approaches can be as well followed to establish an experimenter's truthfulness using its fuzzy sets. In the recent research endeavours, fuzzy notions have been used along with hedonic scale-based sensory evaluation.

Existing research in this field has been devoted towards the nutritional analysis of squash fruit, squash pulp and peel flour and sensory analysis of squash cookies. The existing literature is fairly suggestive to this area of research. The moisture content of squash was found to be about 89–95 g/100 g by few authors [6]. Chayote always consists of good quantity of moisture (89.3–94.2%) but has lower protein constitution (0.77–1.05%). The extracted fruit juice has abundant constitution in vitamin C (22.3%). Additionally, the fruits of the *S. edule* plant have significant amounts of ash content (0.245–0.321%), crude fibre (4.88–5.89%), and carbohydrate (4.12–4.98%) [7]. The protein content varies from 0.82 to 1.74 g per 100 g, while the starch ranges from 0.20 to 1.56 g per 100 g, and the fat content varies as from 0.10 to 0.30 g per 100 g [8]. Also, ash content has been evaluated to be about 0.40–3.60% dry weight in the vegetables. Mineral content of the vegetable refers to 125–338 mg/100 g (potassium), 12–25 mg/100 g (calcium), 12–15.4 mg/100 g (magnesium) and 4–60 mg/100 g (phosphorus) [9]. The immature uncooked fruits of several squash varieties have high vitamin C (7.7–20 mg/100 g) on dry weight basis. The peel portion of the vegetable contains 8.31% (dry weight) ash, which predominantly constitutes 307 mg/100 g of calcium, 196 mg/100 g of phosphorus and iron (6.72 mg/100 g) [9]. Fruit, peel and leaves of the vegetable have ample quantity of vitamin C and carotenoids such as lutein and carotene (β).

Till date, only one article devoted towards the development of squash enriched wheat-based cookies [10]. However, researchers investigated the nutritional and functional aspects of several squash pulp powders [11]. The water, fat, protein, total carbohydrate and energy contents of squash powder were found to be in the range of 10.65–12.5%, 0.8–2.19%, 2.92–4.76%, 73.76–77.39%, and 324.87–333.75 kcal respectively for the chayote flour-based biscuits. The micronutrient constitution in terms of total carotenoid, vitamin C, phosphorus, calcium, iron, potassium, zinc levels have been 6.38–23.30 mg/100 g, 14.58–20.54 mg/100 g, 13.75–26.45 mg/100 g, 39.60–60.98 mg/100 g, 0.55–0.97 mg/100 g, 102.56–119.65 mg/100 g, 0.34–0.56 mg/100 g

respectively [12]. These findings demonstrate that the nutrition-based analysis of the average proximate parameters (carbohydrate, fat, protein, water, and inorganic mineral or ash content) have been as 57.66, 19.39, 16.99, 6.68, and 5.28 g/100 g respectively. In the vegetable, the maximum vitamin C content has been 13.97%, followed by vitamin E (0.11%) and vitamin A (0.041%). Highest mineral content corresponded to 3,902 mg/100 g for potassium, followed by sodium (861.0 mg/100 g) and calcium (665.6 mg/100 g) [11]. Water absorption, water retention, and oil absorption capabilities of squash powder have been estimated to vary as 7.50–9.13, 3.35–6.05 and 1.02–2.04 respectively [11]. The thermal properties and the particle size analysis of squash pulp and peel powder have not been analysed. Till date, the nutritional parameters of squash peel powder have not been investigated.

Cookies with 90:10 wheat:squash constitution have been reported to have 75% acceptance by a set of panellists. Similarly, the researchers investigated squash-based culinary items [10]. The authors targeted variant squash constitution in the prepared items. The best sensory scores formulations for making squash cookies use various quantity of squash. There were 7.58, 7.69, 7.62, 7.46, 7.67 and 7.60 for taste, colour, aroma, texture, appearance and weighted mean score respectively. Upon storage in airtight jars, the squash-based cookies possessed a shelf life of nine days [10]. Fuzzy logic has been a useful technique for analysing unclear and ambiguous data and drawing critical conclusions about food approval, refusal, rating negative and positive features. Language variables (for example not satisfactory, acceptable, exceptional etc.) are often used in fuzzy clustering to build correlations between independent (such as appearance, aroma, flavour, texture, accessibility, etc.) and dependent (such as acceptability, denial, rating, good and bad food qualities) variables [13]. Fuzzy sets can be used to analyse sensory data instead of using the average scores approach to compare the qualities of the samples. This is due to the reason that they are not constrained to a predetermined value and are advantageous in sensory evaluation as human expressions on how we feel about food are fuzzy rather than predictable [13]. For judging and ranking food products, the created fuzzy computing models do an impressive job [14]. In fuzzy logic control, a subject can be represented by a series of elements and the degrees to which they are members of the two sets without membership [15]. The fuzzy sets have been proven to provide mathematical approaches for the analysis of the complexity of human perception [16]. These methods have studied well with mango beverages [17], dahi [18] and instant green tea powder [19]. Cookies made with squash were made in accordance with the earlier study [10], although without the correct composition modification. Not many attempts were reported to determine the sensory qualities of the various squash pulp flours. Cookies made with squash have not yet been determined to have any nutritional benefits. Therefore, the current research focused on the developing squash-based cookies with the best sensory properties (for both 9 point hedonic and fuzzy logic scale). In order to accomplish this, the nutritional, functional, and physical attributes of squash pulp and peel flours were assessed and utilised in the development of the cookies.

2 Materials and Methods

2.1 Procurement of Raw Materials and Sample Preparation

From Kamrup (Shingimari area), Assam, India, raw squash vegetables were bought (26.2205 °N; 91.6241 °E). During transportation, polyethylene bags were used. Thereafter, the vegetables were washed several times with running water to get rid of any dirt or debris that could have been on them. Later, the squash was peeled. The peeled squash was then sliced into 0.5 mm thick slices with a slicer and the uneven pieces were removed. The vegetable samples were dried on trays in a laboratory-scale oven (Make: REICO) for five hours at 60 °C.

2.2 Preparation and Baking of Cookies

The cookies were developed with several ingredients such as wheat flour, sugar, butter, baking powder, vanilla essence, rice flour, and squash pulp flour. To do so, firstly, squash pulp powder was substituted in 10, 30, and 50% weight proportions in the flour constitution. For the rice and wheat based flour constitution, the squash pulp was kept at 30% and rice was varied from 1 to 3 in proportions (dry weight basis) and with reducing wheat flour proportion in the corresponding formulation. Also, same rice: wheat flour formulations were used to bake few cookies without squash constitution. The proportions of sugar, butter, and baking powder were kept constant and as 19, 34 and 2% respectively. The cookies were cut using a cookie cutter of diameter 6 cm and height of 1 cm. The baking temperature and time were varied as 150–170 °C and 15–20 min respectively. Thereby samples (A–J) with the altered constituents were obtained.

2.3 Chemicals

Anthrone, DPPH (extra pure), indophenol sodium salt and sodium bicarbonate were acquired from the manufacturing company SRL Pvt. Ltd., India; Anthrone, Anthrone extra pure, sodium bicarbonate were purchased from Rankem and Sigma Aldrich, India respectively. Merck India supplied pellets of sodium hydroxide, oxalic acid (dehydrate), petroleum ether, 98% sulfuric acid, dextrose, methanol (absolute).

2.4 Determination of Proximate Characteristics

Moisture content (Moc): The moisture content of the squash was calculated using a prior art-based technique (AOAC, 2010). For this, squash sample was kept at 105 ± 5 °C for overnight. Thereafter, the sample was allowed to reach room temperature without absorbing moisture and was evaluated by weighing. The following equation was used to assess Moc (AOAC, 2010) of the sample:

$$\text{Moc}(\%) = \frac{w_1 - w_2}{w_1} \times 100 \quad (1)$$

where Moc = moisture content, w_1 and w_2 are taken as the weights of the squash specimen and the squash (bone dried) sample respectively.

Ash-content: The AOAC, 2010 method was employed to measure the ash-content. For this, in duplicate, a constant amount of squash sample was placed inside a muffle furnace for 6 h at 550 °C. The ash residue was weighed after desiccated storage. The ash content has been calculated with the expression:

$$\text{Ash}(\%) = \frac{w_3}{w_4} \times 100 \quad (2)$$

where w_3 and w_4 are taken as the weight of ash and squash (dehydrated) taken respectively.

Crude-fibre: The crude-fibre content was determined using the method delineated by Sadasivam and Manikam's (1992) method [14]. For this, firstly, 1 g dried sample with 1.25% H_2SO_4 was stirred with a setup of magnetic plate for 30 min. The acid was subsequently removed from the sample by filtering it through a cotton cloth and washing it in hot water at boiling stage. Then the residual portion was exposed to 1.25% NaOH and filtered. The residue was rinsed with H_2SO_4 (1.25%), along with water (50 mL) and alcohol (25 mL). The residual portion was washed thoroughly and a crucible was used for keeping the residue for 2 h at 130 °C. The sample was burnt in a muffle furnace for 30 min at 600 °C after cooling and weighed it. The sample was subsequently weighed after cooling to ascertain its quantity of crude fibre using the following formula.

$$\text{Crude fibre}(\%) = \frac{w_5}{w_6} \times 100 \quad (3)$$

where w_5 and w_6 are the weight loss at 600 °C (during ignition in a muffle furnace) and the sample taken initially respectively.

Fat content: The AOAC (2010) method was used to determine the amount of fat in the vegetable sample. A specific amount of the dried material had to be put into a thimble before being extracted with petroleum ether in a Soxhlet apparatus. After evaporating petroleum ether, the fat content was determined using the formula below:

$$\text{Fat (\%)} = \frac{w_7}{w_8} \times 100 \quad (4)$$

where w_7 and w_8 correspond to the weight of treated and initial sample respectively.

Soluble-protein: To quantify the amount of soluble-protein in the squash sample, Bradford's method was applied [15]. For this, firstly 100 mL of distilled water was used to dissolve about 100 mg of dried material. After performing the extraction, 5 mL of Bradford's reagent was added. The absorbance of the mixture was measured at 595 nm using a BSA standard and a UV-Visible spectrophotometer (Model No. UV-2600, Make: Shimadzu, Singapore).

Carbohydrate-content: The Clegg anthrone technique was used to determine the carbohydrate content [16]. The following is a summary of the involved steps. First, 10 mL of distilled water and 1 g of the substance were well combined. After adding 62% of perchloric acid (13 mL), the mixture was agitated for 20 min. Subsequently, the liquid was diluted to 250 mL and filtration was performed. With 100 mL of distilled water and 5 mL anthrone reagent solution, the filtrate was diluted. With 5 mL of anthrone reagent and 1 mL of distilled water, a blank sample was created. Following that, both samples were kept at 100 °C for 12 min. At 630 nm, the samples' absorbance was finally measured. Applying the following formula, its carbohydrate content was determined [16].

$$\text{Carbohydrate (\%)} = \frac{A_1}{A_2} \times 100 \quad (5)$$

where A_1 and A_2 are the diluted sample and standard (blank) absorbances respectively.

2.5 Bioactive Compounds

Vitamin C content: The method outlined by Sadasivam and Manickam (1992) was used to calculate the sample's vitamin C. To begin with, dried sample of 0.1 g were extracted with 4% oxalic acid (10 mL). Centrifugation and subsequent harvesting of the sample in another media was the next step. 10 mL of oxalic acid (4% concentration by volume) and the supernatant of 5 mL were added together. The indophenol dye (2,6-dichlorophenol) solution was added to titrate the mixture. As a result, vitamin C was calculated using the following equation:

$$\text{Vitamin C} \left(\frac{\text{mg}}{100 \text{ g}} \right) = \frac{0.5 \times v_3 \times 10 \times 100}{v_4 \times 9 \times w_9} \quad (6)$$

where v_3 and v_4 are the volume of standard (ascorbic acid) and sample extract consumed and w_9 corresponds to the sample weight.

TPC and TFC: The FCR method was used to estimate the TPC [18]. The TPC content of the samples was expressed as mg of gallic acid equivalents (GAE)/g weight of the corresponding samples (mg/g sample) using a calibration curve made with gallic acid. The TFC of the samples was evaluated using the AlCl_3 technique [18]. The TFC of the sample was calculated as mg of QE/g weight of the sample. The absorbance of the samples was measured at 517 nm for TFC and 750 nm for TPC.

Antioxidant activity: The 2,2-diphenyl-1-picrylhydrazyl (DPPH) assay methods described by the authors [19] with methanol were taken to assess the activity of antioxidants of the vegetables. The samples were then incubated in the dark for half an hour. Finally, at 517 nm, the absorbance of the samples was measured with a UV spectrophotometer. The antioxidant activity of the samples was evaluated using the expression utilising measured absorbance values and the measured absorbance values [19].

$$\text{AnO} = \frac{A_3 - A_4}{A_3} \times 100 \quad (7)$$

where AnO is the Anti-oxidant activity and A_4 and A_3 resemble to the absorbance of sample and control case respectively.

2.6 Functional Attributes of Flours

Water absorption capacity (WAC, %), oil absorption capacity (OAC, %), temp. of gelatinization (GT, °C), least concentration of gelatinization (LGC, %), and bulk density (g/cc) were used to analyse the functional qualities of flours.

Water absorption capacity (WAC): The WAC of the flours was determined by the prior art method [20]. For this, 1 g of sample and 10 mL of distilled water were combined with and kept to rest for 30 min at room temperature. The mixture was then exposed to centrifugation for 30 min at 2000 g or 3000 rpm. In terms of g of water bound per g of flour, the WAC was measured.

Oil absorption capacity (OAC): OAC was calculated using the prior art methods [20]. For this, 10 mL of soybean oil with a specific gravity of 0.9092 was combined with 1 g of the sample and left to stand for 30 min at room temperature. The mixture was then centrifuged for 30 min at 2000 g or 3000 rpm. In terms of g of oil bound per g of flour, oil absorption was measured.

Least-gelation concentration (LGC): LGC was assessed with minimal adjustments and with the prior art reported approach [21]. In 5 mL of distilled water, flour dispersions with variation from 2 to 30% (w/v) samples were made. They were then cooked in a water bath at 90 °C for one hour. The samples were then cooled using tap water and maintained at 10 °C for 2 h. The concentration that corresponds to the sample that did not fall from an inverted tube was chosen as the one with the least amount of gelation.

Least-gelatinization temperature: Gelatinization temperature was determined as per the reported prior art method [22]. In order to do so, a 1 g quantity of flour was precisely weighed in triplicate and placed into 20 mL screw-capped tubes. Then, each sample received 10 mL of water. The samples were then gradually cooked in a water bath until they solidified. Thereby, the respective temperature for complete gel formation was noted and reported as gelatinization temperature.

Packed Bulk density: To evaluate the parameter, 100 g of flour was weighed into a 250 mL measuring cylinder. Thereafter, the cylinder was tapped on a wooden plank until there was no discernible volume loss. The apparent (bulk) density was then computed using the weight and volume [23].

2.7 Flour-Thermal Properties

Using a differential scanning calorimeter, the squash samples' thermal characteristics were examined (Make: DSC Sirious). In order to do so, 5–7 mg of the sample were placed on the DSC pan for thermal analysis at temperature range and heating rate of 30–350 °C and 10 °C/min, respectively.

2.8 Average Particle Size and Poly-diserspity Index

The particle size (average) and PDI of the squash samples were evaluated using particle analyser [Make: Beckman Coulter Model: Delsa Nano C] and with dynamic light scattering method. To do so, 0.1 g sample and 20 mL water was mixed properly with 10 min sonication and was thereafter transferred into a cuvette for analysis at 25 °C and 170° scattering angle. For these experiments, the deployed diluent water possessed a refractive index of 1.3328 and viscosity 0.8818 cP respectively.

2.9 Functional Group Analysis Using Fourier Transform Infrared Spectroscopy

Fourier transform-infrared spectrophotometry (FTIR; Shimadzu; Japan; IR Affinity1) (4000–400 cm^{-1} wavenumber range) was conducted to determine the existence of functional groups in raw and dried squash peel and pulp. Thereby, relevant interactions have been analysed.

2.10 *Physical Properties of Cookies*

The average of the highest and minimum values of the measured thickness was used to calculate the thickness of a cookie. The ratio of the baked round diameter to thickness was used to compute the spread factor. The weight reduction was calculated as a percentage of the cookie's final weight to its initial weight [24]. Such measurements were based on six repetitions.

2.11 *Sensory Analysis*

A panel of sixteen semi-trained panellists (senior research scholars) evaluated the sensory features of cookies. The samples had been coded with alphabets ranging from A to J for this purpose. Prior to organoleptic evaluation, the panellists were instructed on the hedonic 9-point scale and fuzzy scale-based rating. The attributes being evaluated were flavour, colour, texture, breakability, taste, crunchiness, after taste and overall acceptance. A 9-point hedonic scale was utilised, with 1 representing dislike strongly, 5 representing neither like nor dislike, and 9 representing like extremely. Samples being analysed with 70% of the scores in the 'like range' (6–9) of the hedonic region for various attributes were considered to be acceptable. Panellists were asked to provide ratings in the form of NS (Not Satisfactory), F (Fair), Medium (M), Good (G), and Excellent (E) for samples, and NI (Not Important), SI (Somewhat Important), I (Important), HI (Highly Important), and EI (Extremely Important) for the entire product for fuzzy logic analysis. These were recorded with a fuzzy linguistic score sheet [25]. Excel 2016 (Microsoft Inc. 2016, United States) was applied in this study to analyse sensory assessment data of squash-based cookies using a fuzzy logic technique. The individuals were given water to rinse their mouths between evaluations. Thereby, they were allowed comfort if they did not wish to swallow the samples.

3 Results and Discussion

3.1 *Nutritional Characteristics*

Moisture content: Fresh squash pulp had a moisture content of 96% while squash peel had a moisture content of 94.41%. Similarly, the authors [6] found that the moisture content of fresh squash fruit was in the range of 89–95%. The dried squash pulp powder has a moisture % of 8.68 whereas for dried squash peel powder, it was found to be 12.24%. Similarly, the prior art reported 10.65–12.5% moisture content in squash pulp powder [11]. In cookies, the range of moisture was determined to be about 0.9–8.48%. Among various samples, the cookies with 1:1 rice:wheat formulation

possessed the lowest moisture content (0.9%) and control cookies possessed the highest moisture content (8.48%). The moisture content for cookies with 3:1 rice:wheat and 30% squash constitution was 2.68%. Similar results were reported by a research group [12] for chayote powder-based biscuits (6.68% moisture content).

Ash content: For fresh samples, it was observed that the squash pulp's ash concentration was higher (0.075%) than that of the squash peel (0.013%). Similarly, researchers [7] reported that the ash content varied as 0.245–0.321% for the chayote fruits. For dried powdered samples, the ash content was comparatively higher for squash peel (3.415%) than for the squash pulp (1.345%). The ash content of papaya powder has been reported to be 4.42%. The cookies' ash content ranged from 1.015 to 1.956%. The highest ash content was for the cookies with no substitution and for 1:1 rice:wheat formulation, the value was assessed to be lowest. The ash content of chayote based biscuits (5.28%) was reported in the relevant literature [12].

Crude fibre: For fresh samples, the crude fibre content was found to vary as 1.7–1.8%. The literature confirmed that the crude fibre content of the chayote fruit varied as 4.88–5.89% [7]. For powdered samples, the crude fibre content has been in the range of 4.4–9.7% and the highest value has been for the squash peel. For papaya peel powder, a crude fibre content of 0.65% has been reported [26]. The crude fibre content for cookies has been in the range of 0.5–1.8%. The highest fibre content of 1.8% was obtained for the formulation with 30% squash flour and 3: 1 rice to wheat ratio while the lowest was for the control cookies (0.5%). However, for the control sample, crude fibre content is found to be lowest. The crude fibre content of the papaya pulp based cookies was reported to be 3.48% [27].

Fat content: The fat content of fresh squash pulp and fresh squash peel were 0.1 and 0.048% respectively. Few authors, reported that the fat content in fresh squash fruit varied as 0.1–0.3% [17]. The squash pulp powder and squash peel powder possessed fat content of 3.3–5.47%. For squash pulp powder, the fat content of 0.8–2.19% has been reported by few authors [11]. For cookies, the fat content was in the range of 31–38.44%. For the control cookie samples, the fat content of 38.44% was obtained. Comparatively, for the 30% squash constituted 3:1 rice to wheat formulated cookies, the fat content was 31%. The fat content of chayote-based cookies was reported to be 19.39% [12].

Carbohydrate: For fresh samples, the carbohydrate content was 5.09 and 5.39% for squash pulp and peel respectively. Similarly, in the literature, researchers reported carbohydrate content of the chayote fruit to vary as 4.12–4.98% [7]. The carbohydrate content of squash pulp and squash peel powder have been in the range of 41.1–52.6%. Among both, the squash peel had lower carbohydrate content. In the literature, few authors found that the carbohydrate content of squash pulp powder varied as 73.76–77.39% [11]. For cookies, the carbohydrate content was 51.91% for control samples and was 54.4% cookies with 30% squash constitution and 3:1 rice to wheat ratio. For the literature reported cookies, the carbohydrate content was about 56.6% [12].

Vitamin C content: The vitamin C content of fresh squash pulp and fresh squash peel was found to be 7.142 and 14.28 mg/100 g respectively. Similarly, the literature reported vitamin C content of the chayote fruit varied as 7.7–20 mg/100 g [9]. The squash pulp powder and squash peel powder possessed vitamin C content in the

range of 71.42–142.8 mg/100 g. The vitamin C content of squash powder as per literature varied as 14.58–20.54 mg/100 g [11]. For control cookie sample, it varied as the vitamin C content was nil and was 142.85 mg/100 g for 1: 1 rice to wheat constituted cookies with 30% squash content. For chayote powder, the literature reported a vitamin C content of 139.7 mg/100 g [12].

Soluble protein: The protein content of fresh squash pulp and squash peel samples have been in the range of 0.7–1.5%. The literature reported the protein content of squash fruit to vary as 0.7–1.05% [7]. The protein content of squash pulp powder varied as 5.454–5.677%. For the squash peel powder it varied as 6.171–6.430%. The literature confirmed that the protein content of squash powder to vary from 2.92 to 4.76% [11]. The cookies possessed protein content in the range of 1.77–1.98%. The literature reported protein content of cookies was about 16.99% [12].

Total Phenolic Content (TPC) and Total Flavonoid Content (TFC): For fresh squash pulp and fresh squash peel samples, the TPC were 1.89 and 1.31 mg GAE/g respectively. The fresh pulp and fresh squash peel samples possessed TFC of 4 mg of QE/g and 4.05 mg of QE/g respectively [28]. The TPC values for squash fruits have been reported in the literature to vary as 1.02–6.18 mg GAE/g [9]. Corresponding TFC varied as 1.5–60.1 mg QE/g [9]. The dried samples of squash pulp and squash peel possessed TPC of 0.912 and 1.18 mg GAE/g respectively. Corresponding TFC values for the samples were 3.85 and 6.2 mg QE/g respectively. For pumpkin slice powder, the reported TPC in the literature was 11.13 mg GAE/g [29]. For butternut squash powder, the reported TFC was 1.32 mg GAE/g [30]. The cookies with 3:1 rice to wheat and 30% squash constitution had highest TPC of 0.97 mg GAE/g. On the other hand, TPC was lowest for 1:1 rice to wheat ratio cookies and was 0.27 mg GAE/g. The control sample possessed a TPC of 0.66 mg GAE/g. With respect to the TFC, the control samples had the highest value (5.5 mg QE/g) and the lowest was for cookies with 1:1 rice to wheat ratio (3 mg QE/g). However, the TFC value for cookies with 3:1 rice to wheat ratio and with 30% substitution of squash was 5 mg QE/g.

Antioxidant activity: The antioxidant activity of fresh squash pulp and squash peel samples were found to be about 7.68 and 43.19% respectively. The dried samples reported an antioxidant activity of 18.83% for squash pulp powder and 35.5% for squash peel powder. For the cookies, the highest antioxidant value has been obtained for samples prepared with 3:1 rice to wheat ratio and 30% squash constitution (62.84%) and the lowest was for the control samples (15.24%). Incidentally, the antioxidant activity of the cookies with 1:1 rice to wheat constitution was 18.51%.

3.2 Functional Properties

Water absorption capacity and oil absorption capacity: While water absorption capacity was maximum for the squash peel fine powder (7.98 g water/g sample), it was lowest for the squash pulp finer powder (4.8 g water/g sample). The coarser squash pulp powder had a water absorption capacity of 6.12 g water/g sample. The

Table 1 Functional properties of squash flour samples

Properties	Squash pulp powder (coarse)	Squash pulp powder (fine)	Squash peel powder
Water absorption capacity (g water/g sample)	6.12 ± 0.73	4.8 ± 0.55	7.98 ± 0.86
Oil absorption capacity (g oil/g sample)	1.23 ± 0.23	1.26 ± 0.07	1.44 ± 0.34
Least gelation concentration (%)	–	12 ± 1.08	14 ± 0.93
Least gelatinization temperature (°C)	–	97 ± 1.2	94 ± 1.6
Bulk density (g/mL)	–	0.606 ± 0.03	0.59 ± 0.06

oil absorption capacity of squash pulp fine powder, squash pulp coarse powder and squash peel fine powder were 1.26, 1.23 and 1.44 oil/g sample respectively. In the literature, the reported water absorption and oil absorption capacities varied as 7.50–9.13 g water/g sample and (1.02–2.04 g water/g sample) respectively [11]. These have been summarized in Table 1.

Least gelatinization temperature and least gelation concentration: The least gelatinization temperature for squash pulp powder and squash peel powder were 97 and 94 °C respectively. The least gelation concentration of squash pulp powder and squash peel powder were 12 and 14% respectively.

Bulk density: The packed bulk density of squash pulp powder and (g/mL) squash peel powder were 0.606 and 0.59 g/mL respectively.

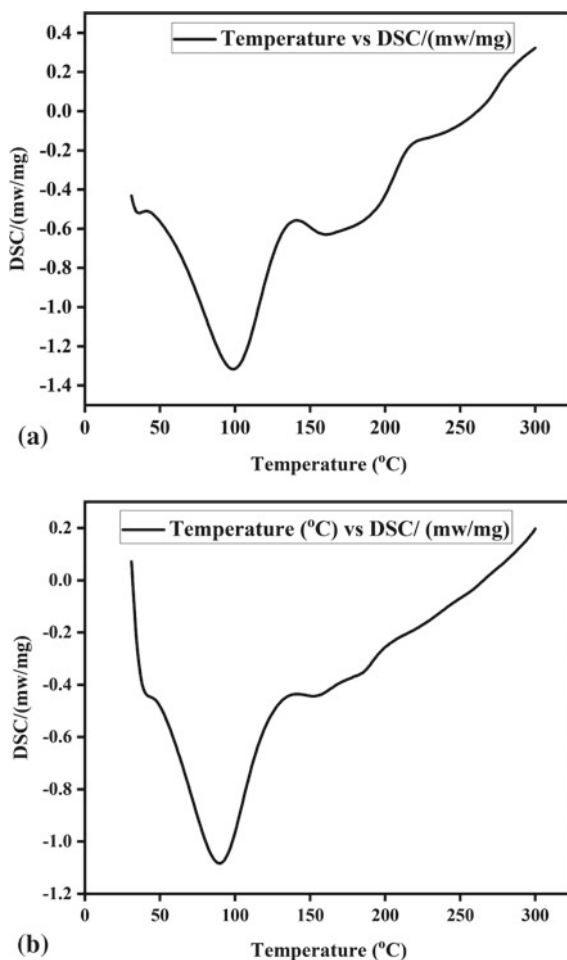
3.3 Thermal Properties

For squash pulp powder, the glass transition onset temperature was 56.2 °C and the end temperature was 182.0 °C. The peak glass transition temperature has been 98.6 °C and the specific heat (C_p) was (0.622 J/(g °K)). For squash peel powder, the glass transition onset temperature was 63.2 °C, end temperature was 201.1 °C and peak temperature was 89.3 °C. The specific heat capacity (C_p) was 0.475 J/(g °K) for the squash peel powder (Fig. 1).

3.4 Average Particle Size

The average particle size of fine (F) squash pulp powder was 3747.1 nm and its polydispersity index was 0.946 (Fig. 2). The coarse (C) squash pulp powder sample

Fig. 1 DSC plots of squash
a pulp and **b** peel powder

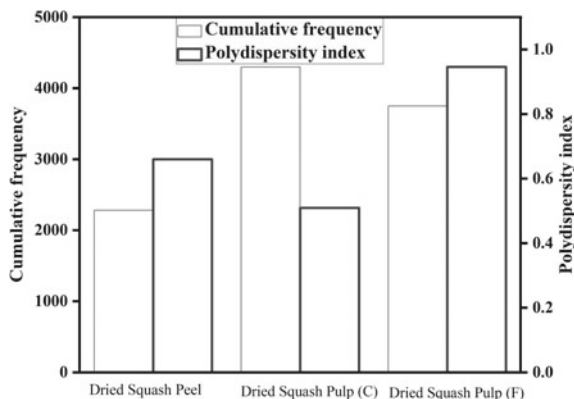


had an average particle size of 4295.6 nm and polydispersity index of 0.509. For the squash peel powder sample, the average particle size was 2282.2 nm and the polydispersity index was found to be 0.660 respectively (Table 2).

3.5 Fourier Transform Infrared Spectroscopic Analysis

The raw pulp spectra exhibited hydrogen bonded normal polymeric OH stretch (peak at 3307.33 cm^{-1}), Alkenyl C=C stretch (peak at 1636.99 cm^{-1}), methyl asymmetric bend (peak at 1440.84 cm^{-1}), CN stretch (primary amine) with (at 1029.71 cm^{-1}), aliphatic C-I stretch Iodo compounds peak at 564.19 cm^{-1} . Apart from these functional groups in raw squash pulp, the squash peel spectra confirmed the aliphatic

Fig. 2 Particle size analysis plot of dried squash peel and pulp



bromo compound with a peak at 608.96 cm^{-1} . For the coarse squash pulp powder sample, most mentioned functional groups of the fresh pulp existed. Along with these, a methylene C–H asymmetric stretch related peak was observed at 2926.81 cm^{-1} . However, the methyl asymmetric bend was absent and a carboxylate group can be confirmed with a peak at 1406.45 cm^{-1} . For the fine squash pulp powder, aliphatic chloro compounds with a peak at 774.7 cm^{-1} and aryl group having C–H 1,4 di substitution (para) peak at 818.59 cm^{-1} can be analysed in addition to the other groups being observed for the coarse squash pulp powder. The squash peel powder spectra confirmed similar functional groups that exist in the coarse squash pulp powder sample (Figs. 3 and 4).

3.6 Physical Properties of Cookies

For the control cookie sample, the spread ratio was 9.375. Similarly, for the squash enriched cookies with 10, 30 and 50% squash constitution, the spread ratio varied as 10.41–10.71. For cookies with 1:1, 1:2 and 1:3 rice to wheat ratio, the spread ratio was 14.5, 17.6 and 27.14 respectively. Thus, the spread ratio increased with rice inclusion in the wheat flour. The squash enriched cookies along with rice and wheat flour having 30% squash substitution and rice: wheat in ratio (1:1), (2:1) and (3:1) spread ratio possessed values of 14.11, 11.18 and 13.84 respectively. The squash inclusion reduced the spread ratio of cookies with rice and wheat flour. The weight reduction for control (only wheat) and wheat cookies with 10, 30 and 50% squash constitution was 11.66, 11.11, 10.67 and 10.1% respectively. For cookies with rice to wheat ratio of 1:1, 2:1 and 3:1, these were 10.13, 10.23 and 10.9% respectively. The squash enriched cookies with 30% substitution and rice to wheat ratio of 1:1, 2:1 and 3:1 possessed a spread ratio of 8.03, 8.56 and 9.1% respectively.

Table 2 Characteristics of fresh and dried (powdered) squash pulp and peel samples and targeted cookie precursors and precursor mixtures

Properties	Fresh squash pulp	Fresh squash peel	Dried squash pulp powder	Dried squash peel powder	Control (wheat)	Rice:wheat (1:1)	Rice:wheat (3:1) with 30% squash pulp powder
Moisture content (%)	96 ± 4.55	94 ± 2.74	8.68 ± 0.26	12.24 ± 0.53	8.48 ± 0.14	0.9 ± 0.22	2.68 ± 0.48
Ash content (%)	0.08 ± 0.04	0.013 ± 0.05	1.34 ± 0.05	3.41 ± 0.04	1.95 ± 0.08	1.01 ± 0.03	1.55 ± 0.1
Crude fibre (%)	1.70 ± 0.12	1.8 ± 0.43	4.4 ± 0.28	9.7 ± 0.33	0.5 ± 0.07	0.3 ± 0.02	1.8 ± 0.15
Fat (%)	0.10 ± 0.03	0.048 ± 0.01	3.3 ± 0.18	5.47 ± 0.59	36.44 ± 0.48	38.44 ± 0.37	31 ± 0.81
Carbohydrate (%)	5.09 ± 0.66	5.39 ± 0.24	52.6 ± 0.65	41.1 ± 0.95	51.91 ± 0.73	53.44 ± 0.42	54.4 ± 0.88
Soluble protein (%)	0.70 ± 0.05	1.52 ± 0.02	5.56 ± 0.13	6.30 ± 0.32	1.87 ± 0.46	1.77 ± 0.49	1.98 ± 0.60
Vitamin C (mg/100 g)	7.14 ± 0.1	14.28 ± 0.15	71.42 ± 0.23	142.8 ± 0.28	0	0	142.85 ± 0.36
TPC (mg GAE/g)	1.89 ± 0.77	1.31 ± 0.06	0.91 ± 0.05	1.18 ± 0.30	0.66 ± 0.06	0.27 ± 0.01	0.97 ± 0.01
TFC (mg QE/g)	4.00 ± 0.35	4.05 ± 0.44	3.85 ± 0.62	6.2 ± 0.96	5.5 ± 0.81	3 ± 0.16	5 ± 0.27
Antioxidant activity (%)	7.68 ± 0.87	43.19 ± 1.02	18.83 ± 1.46	35.5 ± 2.07	15.24 ± 1.82	18.51 ± 0.25	62.84 ± 0.67
Peak glass transition temperature (°C)	-	-	182	201.1	-	-	-
Specific heat Cp (J/g °K)	-	-	0.622	0.475	-	-	-
Average particle size (nm)	-	-	3747.1 ± 10.06 (F) 4295.6 ± 14.72 (C)	2282.2 ± 18.54	-	-	-
Polydispersity index	-	-	0.95 (F) 0.51 (C)	0.66	-	-	-

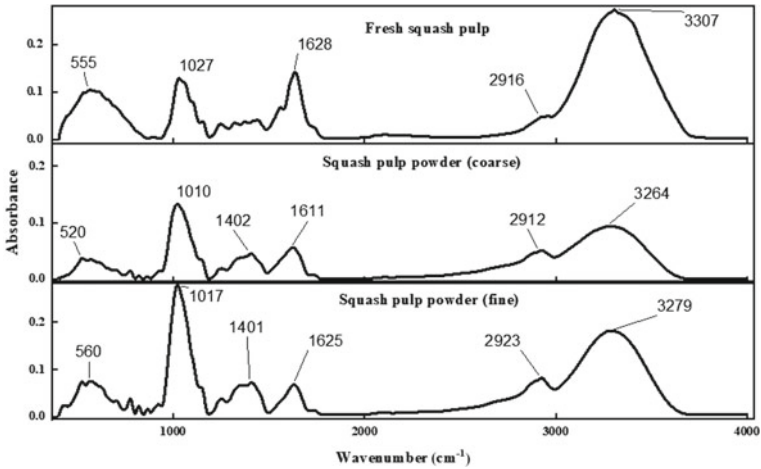


Fig. 3 FTIR spectra of fresh, coarse and fine squash pulp flour

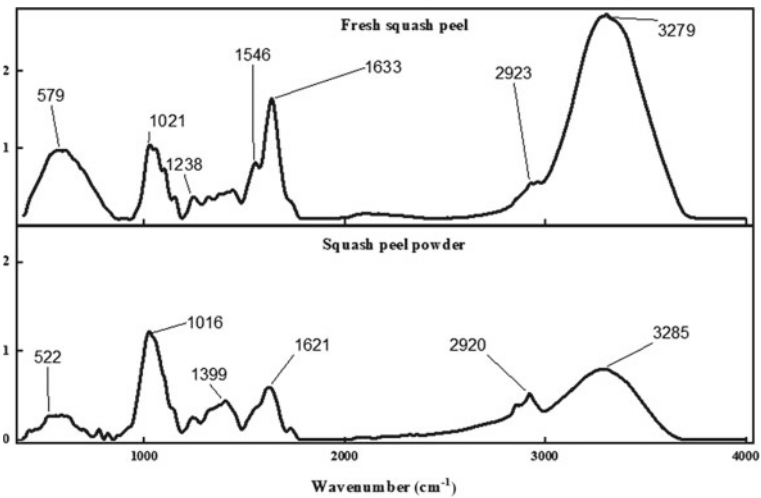


Fig. 4 FTIR plots for fresh squash peel and squash peel flour

3.7 Sensory Analysis

The overall sensory score based on 9-point hedonic scale for control cookie samples was 8.45. However, the score reduced with the addition of squash pulp powder 10, 30 and 50 proportions and as 7.88, 6.81 and 6.46 respectively. However, the score was above 6 even for 50% squash substituted samples and were hence considered to be acceptable. For sample with rice to wheat ratio of 1:1, 2:1 and 3:1, the overall sensory scores were 7.91, 7.79 and 7.59 respectively. It is evident that with an increase in rice

constitution, the sensory score reduced. The cookies with 30% squash constitution and rice to wheat ratio of 1:1, 2:1 and 3:1 constitution possessed as overall sensory scores of 6.63, 6.81 and 7.51 respectively (Table 3). These scores convey that with an increase in rice constitution along with squash pulp powder, the overall sensory score increased for the cookies. The quality features of the cookies were graded based on fuzzy logic in the order of decreasing importance as taste, flavour, after taste, crunchiness, overall acceptability, texture, breakability and colour. According to fuzzy logic sensory scale, the control cookies with only wheat flour have been rated to be very good (0.79) followed with wheat cookies substituted with 10% squash (also rated to be very good with 0.74 sensory score). The wheat cookies with 30% and 50% squash constitution were rated as good (0.7) and satisfactory (0.75). For control cookie samples with rice to wheat ratio of 1:1, 2:1 and 3:1, these sensory ratings were very good (0.71), very good (0.69) and good (0.69) respectively. The cookies with 30% squash pulp powder constitution and with rice to wheat ratio 1:1, 2:1 and 3:1 possessed these ratings as good (0.74), good (0.72) and very good (0.71).

4 Conclusions

Since squash pulp flour and squash peel flour possessed significant amount of WAC constitution, they can be inferred to be useful for various bakery products such as cookies. Similar to the polysaccharides, good water absorption characteristics confirm upon the existence of compounds with higher presence of more hydrophilic groups. In baked products, oil holding capacity is an essential property to influence the texture-flavour-taste qualities of the product. Due to their improved absorption of oil in comparison with wheat, the squash pulp and peel can be used as alternate the gluten-free flour in the formulation of cookies. An increment in oil absorption capacity of squash flour has been probably due to the binding of non-polar side chain of the flour with the hydrocarbon side chain of the oil. Compared to wheat (56.22 °C), least gelatinization temperatures have been high for squash pulp and peel flours and they indicate lower constitution of starch. Least gelation concentration (LGC) defines lowest protein concentration that can be used as an index of gelation capacity. Wheat flour has lower gelation concentration (LGC) of about 8 g/100 mL in comparison to squash pulp and peel flours that exhibit an LGC of 12–14 g/100 mL [31].

Compared to the fresh samples, the nutritional parameters such as ash, crude fibre, fat, carbohydrate and protein have been found to significantly higher in dried powdered samples. This infers that such dried powders can be applied to enhance the nutritional qualities of various food materials. Vitamin C content was found in greater constitution in dry powders such as 71.42 mg/100 g for squash pulp powder and 142.85 mg/100 g for squash peel powder in comparison with the fresh pulp and peel. A similar trend existed for the followed for the antioxidant activity. It was analysed that the antioxidant activity of dried powders was 18.83% for squash pulp and 35.5% for squash peel powder and these were greater than the values obtained for fresh squash pulp and fresh squash peel. From thermal stability perspective, both squash

Table 3 Sensory analysis data (9-point hedonic scale) of grain and squash-based cookies

	Flavour	Colour	Texture	Breakability	Taste	After taste	Crunchiness	Overall acceptability	Overall sensory score
Control	7.94 ± 0.80	8.33 ± 0.41	8.5 ± 0.87	8.61 ± 1.34	8.56 ± 0.44	8.28 ± 0.59	8.22 ± 0.46	8.72 ± 0.41	8.45 ± 0.67
A (10%)	7.67 ± 0.67	7.67 ± 0.22	8.0 ± 0.91	8.11 ± 1.22	7.89 ± 0.37	7.61 ± 1.07	8.17 ± 0.15	7.94 ± 0.95	7.88 ± 0.70
B (30%)	6.83 ± 0.71	6.05 ± 0.54	6.94 ± 1.02	7.22 ± 0.95	7.06 ± 0.38	5.89 ± 0.06	7.44 ± 0.66	6.83 ± 0.27	6.81 ± 0.57
C (50%)	5.83 ± 0.54	5.38 ± 0.84	7.38 ± 0.96	7.89 ± 0.07	5.38 ± 1.05	5.11 ± 0.28	7.33 ± 0.74	5.61 ± 0.55	6.46 ± 0.63
D (R:W 1:1)	8.12 ± 0.58	7.87 ± 0.43	7.59 ± 0.47	8.12 ± 0.49	8.15 ± 1.48	7.46 ± 0.44	7.93 ± 1.43	8.0 ± 0.81	7.91 ± 0.77
E (R:W 2:1)	8.0 ± 0.32	7.78 ± 0.33	7.65 ± 0.66	8.28 ± 0.88	7.46 ± 0.78	7.37 ± 0.65	7.75 ± 0.64	8.03 ± 0.26	7.79 ± 0.57
F (R:W 3:1)	7.46 ± 0.69	7.65 ± 0.65	7.5 ± 0.65	8.03 ± 0.69	7.56 ± 0.36	7.25 ± 0.43	7.5 ± 1.08	7.78 ± 1.58	7.59 ± 0.77
G (R:W 1:1 30%)	6.81 ± 0.47	6.59 ± 0.51	6.90 ± 0.75	7.28 ± 1.25	6.31 ± 1.02	5.65 ± 0.79	6.93 ± 0.92	6.59 ± 2.04	6.63 ± 0.97
H (R:W 2:1 30%)	6.75 ± 0.84	6.93 ± 0.77	6.90 ± 1.07	7.53 ± 0.53	6.56 ± 1.46	6.18 ± 0.12	6.75 ± 0.35	6.87 ± 0.33	6.81 ± 0.68
J (R:W 3:1 30%)	7.40 ± 0.52	7.40 ± 0.55	7.65 ± 1.33	7.62 ± 0.47	7.5 ± 0.27	7.5 ± 0.72	7.43 ± 0.83	7.54 ± 0.62	7.50 ± 0.66

flours (pulp and peel) were found to be stable for baking temperatures upto 182–201 °C. The average particle size of the squash pulp and peel (2282.2–4295.6 nm) was comparable to that of the wheat flour. Additional methylene C–H asymmetric stretch (at 2926.81 cm^{-1} peak) and carboxylate group (peak at 1406.45 cm^{-1}) did exist in case of powdered squash pulp and peel in comparison with the fresh squash pulp and peel. Only control and squash substituted wheat cookies (10–50%) did not confirm upon any significant change in terms of spread ratio. However, only for wheat and rice constituted cookies without squash flour, a huge variation in spread ratio was observed. This confirms that the rice flour has a significant influence on the parameter. According to the findings of the current study, incorporating squash greatly decreased the spread ratio and increased the thickness of the cookies to a desired level. Spread ratio and thickness of cookies are often inversely proportional to one another and do have a crucial role in consumer acceptability. In general aspect, cookies with lower thickness and higher spread ratio are less preferred by the consumers.

The 9-point hedonic scale-based sensory evaluation confirmed the highest overall sensory score for wheat based samples control (8.45) followed by rice:wheat 1:1 rice to wheat ratio cookies, wheat flour substituted with 10% squash pulp flour and rice to wheat 3:1 ratio and with 30% squash pulp powder. The corresponding overall sensory scores were 7.91, 7.88 and 7.51 respectively. According to fuzzy logic-based sensory evaluation, all three samples (control, 1:1 rice to wheat ratio sample and sample with 3:1 rice to wheat ratio and 30% squash pulp flour) confirmed very good score. The quality attributes of cookies were classified on the basis of fuzzy logic and in the order of reducing significance of taste, flavour, after taste, crunchiness, overall acceptability, texture, breakability and colour. The substitution of squash pulp powder did enhance the nutritional parameters of the cookies. The fibre content (crude) of the samples (1.8%) was higher than that of the only wheat-based cookies. The fat content of squash based cookies with 30% squash content and 3:1 rice to wheat ratio was lesser than that of the control cookie samples. Marginal enhancement in carbohydrate content (about 3%) was observed in squash-based cookies. For the squash-based cookies, the vitamin C content enhanced to 142.85 mg/100 g sample from 0 mg/100 g in control cookies. The TPC values of squash-based cookies were higher (0.97 mg GAE/g) than the TPC values of control wheat cookies. The antioxidant activity of squash-based cookies (58.94–66.75%) was higher in conjunction with the control cookies with only wheat. Henceforth, the incorporation of squash peel powder and pulp in wheat and wheat and rice-based cookies will be an important choice for the development of various bakery products. The enhancement of squash pulp flour in cookies through an alteration in the suggested formulations can further promote the application of squash as a gluten-free alternative in bakery products.

Declarations

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