



Characterization of extruded whole grain cereals enriched with Indian horse chestnut flour

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Abstract

This study explores the development of a fibre-rich breakfast cereal utilizing Indian Horse Chestnut Flour (IHCF) combined with traditional whole grains, including whole wheat flour, barley flour, and corn flour. The research focuses on optimising key extrusion process variables—feed moisture content (10–18%), barrel temperature (70–150 °C), screw speed (290–380 rpm), and IHCF concentration (1.75–4.75%) to enhance the cereal's quality attributes. Comprehensive measurements and analyses were conducted to determine the optimal conditions, focusing on water absorption capacity, bulk density, sectional expansion, water solubility index, porosity, rehydration ratio, hardness, and overall product acceptability. Extrudates rich in IHCF were also evaluated for rheological and Fourier Transform Infrared (FTIR) spectroscopy. Statistical analysis revealed significant impacts of these variables on the cereal's physical properties. Under optimal conditions (12% feed moisture, 130 °C barrel temperature, 380 rpm screw speed, and 2.5% IHCF), the breakfast cereal exhibited superior expansion, porosity, and rehydration ratios, along with desirable hardness, achieving an overall acceptability score of 77.1. Further, an increase in feed moisture content corresponded with improved pasting properties in the optimized extruded flours. The rheological assessment indicated that the storage modulus (G') of both extruded and native flours surpassed the loss modulus (G''), with samples at higher moisture content displaying the highest flow index but the lowest consistency coefficient. FTIR spectroscopy analysis underscored a reduction in peak intensity during extrusion, indicating notable structural modifications in the cereal constituents. This research presents significant advancements in the measurement and characterization of breakfast cereals, underscoring the potential of IHCF as a nutritious ingredient in the food industry.

Keywords Breakfast cereal · Horse chestnut flour · Whole grain flours · Extrusion cooking · Rheological property

Introduction

The change in lifestyle owing to increased working hours and limited free time has increased the demand for convenience food. Advanced processing technologies such as extrusion cooking, and high-temperature short time; have been used for the development of a wide range of extruded products such as breakfast cereals, snacks, porridge, puffed snacks, and fiber-rich products [1]. Extrusion cooking is a high throughput processing technology that combines shear force, high pressure and temperature causing distinctive changes in the structural and textural characteristics of the extrudates such as retaining nutritional quality, improving functional attributes, high expansion, and low density with uniform microstructure and crunchy texture [2]. Generally, extruded foods are prepared from processed and whole-grain cereals. Whole grain flours has been widely utilized as a functional ingredient and are rich in dietary fiber, vitamins,

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starch, protein, minerals and bioactive compounds that are linked with potential health benefits against chronic diseases [3]. In addition to whole grain flours, the incorporation of non-traditional ingredients like Indian horse chestnut flour (IHCF) may encourage a sustainable and healthy plant-based ingredient. Incorporating IHCF into breakfast cereals not only enhances the nutritional value of the product but also contributes to sustainable food production. By promoting the use of an underutilized resource, optimizing processing methods, and scaling up production sustainably, IHCF-enriched products can play a significant role in addressing the global demand for healthy and environmentally friendly food options.

Indian Horse chestnut (*Aesculus indica*) found in the Himalayan region of India, though nutritious, has remained underutilized at the commercial level [4]. It can be a boon for the food, and pharmaceutical industries if the inherent characteristics are explored. IHCF is a rich source of starch, essential fatty acids (oleic acid, and linoleic acid), and phytochemicals such as flavonoids, escin, epicatechin, kaempferol, tannins, and polyphenols. Due to these unique phytoconstituents, IHCF is used in the prevention and treatment of several disorders as venous congestion in leg ulcers, cardiovascular, bruises, fever, wound healing, piles, viral infections, diarrhea, dermatitis, cancers, arthritis, rheumatism, phlebitis [5–7]. There is a huge demand for healthy foods across the globe due to the enhanced prevalence of several chronic disorders. Therefore, the association between non-conventional seed flour and whole grain flours in expanded extruded cereals seems to be the best option for developing a novel product with improved overall nutritional, structural, functional, and mechanical characteristics of the developed product and an opportunity to contribute meaningful research to the cereal industry. It aligns with current consumer trends towards healthier, more diverse food options and addresses gaps in knowledge regarding alternative cereal ingredients. This study addresses significant gaps in the literature by exploring the use of IHCF in breakfast cereals, providing valuable data on its nutritional, functional, textural, and sensory properties, and optimizing the extrusion process for commercial production. The findings have the potential to promote IHCF as a sustainable and nutritious ingredient in the food industry, meeting the growing demand for healthy and convenient snack options.

Previous literature has reported the development of extrusion-based breakfast cereal to characterize their physicochemical, functional and mechanical characteristics using conventional (maize, barley, wheat) and non-conventional starch-rich ingredients (green banana, potato, water chestnut) [8], agricultural by-product (passion fruit peel) [9], corn flour and whole grain wheat flour [10]. The results from the recent studies showed higher bulk density, water absorption index, water solubility index, and acceptability score with

desirable low hardness and crispness. Some of the researchers have also investigated the effect of extrusion parameters (screw speed, moisture, and barrel temperature) on the properties (density, expansion index, color, flavor and hardness) of expanded snacks prepared with mixtures of cereals and legumes [11].

The practical applications and implications of incorporating IHCF into breakfast cereals are far-reaching for both the food industry and consumers. For the industry, it provides opportunities for product innovation, market differentiation, sustainable sourcing, process optimization, and enhanced nutritional labelling. For consumers, it offers enhanced nutrition, functional health benefits, convenience, taste, sustainability, and dietary diversity. By exploiting these advantages, IHCF-enriched cereals can meet the growing demand for nutritious, functional, and sustainable breakfast options, contributing to improved public health and well-being. Therefore, in the present work, the use of IHCF in the development of sugar-free breakfast cereal will be explored and the impact of IHCF and extrusion process conditions on the structural, functional, textural, and sensory attributes of developed breakfast cereal will be explored as well.

Materials and methods

Barley (PL 807), white corn (DT-2), and wheat (SW-2) were procured from Kargil, India, and SKUAST-K, Shalimar, J&K, respectively. Milling was done to prepare whole grain flours by grinding using a Buhler roller mill (MDDK250/1000, China), to the fineness that passed through a sieve of aperture size 1.5 mm. The whole grain flours were then packed in polyethylene bags and stored at 4 °C until analysis. Indian horse chestnut seeds (*Aesculus indica*) were collected manually in October (2020) from the local area of Shalimar, J&K (Harwan).

Preparation of Indian horse chestnut flour (IHCF)

Indian horse chestnut seeds were manually peeled. The kernels were sliced uniformly and boiled for 10 min. The slices were then dried at 60 °C and fed to a laboratory grinder (Usha, New Delhi) to obtain IHCF of fineness that passed through a sieve of aperture size 0.6 mm.

Experimental design

In all treatments, the percentage of whole wheat flour and whole barley flour was kept at 10% whereas in the control sample, the remainder 80% was made of whole corn flour. In the feed mixture, IHCF was used to replace the WCF. The central composite rotatable design (CCRD) was used to study the effect of four independent variables i.e., the concentration

of IHCF (X_1), feed moisture (X_2), barrel temperature (X_3), and screw speed (X_4). $X_1, X_2, X_3,$ and X_4 were at five levels each as shown in Table 1.

$$y_i = b_0 + \sum_{i=1}^4 b_i X_i + \sum_{i=1}^4 b_{ii} X_i^2 + \sum_{i=1}^4 \sum_{j=1}^4 b_{ij} X_i X_j$$

where y_i is the response variable, x_i ($i = 1, 2, 3, 4$) are independent variables (feed composition, feed moisture content, barrel temperature, and screw speed) and b_0, b_i, b_{ii}, b_{ij} are the coefficients of regression for constant, linear, quadratic and interaction regression terms respectively.

After numerical optimization, five different optimized extrudates were taken with constant feed composition (10% WWF, 10% WBF, 2.5% IHCF and 77.5% WCF) and process conditions (barrel temperature 130 °C and screw speed 380 rpm) but varying feed moisture content (12%, 13%, 14%, 15%, and 16%) to investigate the effect of feed moisture content on the pasting properties, rheological characteristics and FTIR spectroscopy.

Extrusion cooking

The breakfast cereals were prepared in a co-rotating twin-screw extruder (Basic Technology Pvt. Ltd., Kolkata, India) with a die diameter of 3.0 mm, and a length-to-diameter ratio of 8:1. Before being fed to the extruder, the composite flours were subjected to conditioning for adjustment of moisture content as per the experimental design. IHCF and process parameters of extrusion were varied as per Table 2. The extrudates were cooled at room temperature and packed in High-density polyethylene (HDPE) bags for further analysis.

Structural properties of extrudates

Bulk density (BD, g/mL)

The bulk density was determined according to the method of [10] using the following formula

$$\text{Bulk density (g/mL)} = \frac{\text{weight of sample (g)}}{\text{volume of sample after tapping (mL)}}$$

Apparent density (AD, g/mL)

Apparent density was calculated as per the method defined by [12] by using the following expression

$$\text{AP (g/mL)} = \frac{\text{Mass of ground extrudates}}{\text{Volume}} \times 100$$

Porosity (ϵ)

Porosity of the extruded samples was measured by following the method of [13] based on the bulk density (BD) and apparent density (AD) of samples and was expressed as a percentage using the following equation

$$\epsilon = 1 - \frac{\text{BD}}{\text{AD}} \times 100$$

Sectional expansion (SE)

Sectional expansion of the extrudates was determined as per the method adopted by [14]. It is calculated as the ratio of extrudate diameter to the die diameter (mm) and expressed in the following equation

$$\text{SE} = \left(\frac{D_e}{D_t} \right)^2$$

Rehydration rate (RR)

The Rehydration rate (RR) was measured by the method of [15]. A briefly small amount of sample (1.5 g) was transferred to a tea bag and sealed followed by dispersing in distilled water (100 mL) for soaking at room temperature (25 °C). The sample weight is continuously monitored until the samples reach equilibrium mass transfer. The RR ratio is expressed by the following equation

$$\text{RR} = \frac{M_t - M_o}{M_e - M_o}$$

Table 1 Coded levels and experimental ranges of the independent variables generated using CCRD

Numerical variable	Symbol	Coded variable levels				
		-2	-1	0	1	2
Indian horse chestnut flour (%)	X_1	1.75	2.5	3.25	4	4.75
Feed moisture content (%)	X_2	10	12	14	16	18
Temperature (°C)	X_3	70	90	110	130	150
Screw speed (rpm)	X_4	290	320	350	380	410

Table 2 Effect of independent variables on structural, functional, sensory and mechanical characteristics of extrudates

Runs	Independent variables				Structural properties				Functional		Mechanical		Sensory	
	IHCF (%)	Feed moisture (%)	Barrel temperature (°C)	Screw speed (rpm)	BD (g/mL)	SE	ε	RR	WAI (g/g)	WSI (%)	Hardness (N)	OA		
1	2.5(-1)	12(-1)	90(-1)	320(-1)	6.5	2.67	0.84	4.74	5.72	31	268.89	8.15		
2	4(+1)	12(-1)	90(-1)	320(-1)	6.74	2.6	0.77	4.69	5.88	27	287.3	8.14		
3	2.5(-1)	16(+1)	90(-1)	320(-1)	6.88	1.42	0.74	4.31	7.51	26	424.65	8.22		
4	4(+1)	16(+1)	90(-1)	320(-1)	7.36	1.4	0.68	4.18	7.65	22	428.5	8.2		
5	2.5(-1)	12(-1)	130(+1)	320(-1)	5.4	2.77	0.88	5.33	7.24	42	252.13	8.3		
6	4(+1)	12(-1)	130(+1)	320(-1)	5.63	2.72	0.8	5.2	7.41	37	270	8.23		
7	2.5(-1)	16(+1)	130(+1)	320(-1)	6.34	2.11	0.79	4.58	8	40	314.68	8.35		
8	4(+1)	16(+1)	130(+1)	320(-1)	6.53	2.09	0.75	4.53	8.33	35	330.23	8.3		
9	2.5(-1)	12(-1)	90(-1)	380(+1)	5.95	3.14	0.89	5.44	5.54	44	226.52	8.2		
10	4(+1)	12(-1)	90(-1)	380(+1)	6.33	3.11	0.82	5.28	5.62	35	241.5	8.11		
11	2.5(-1)	16(+1)	90(-1)	380(+1)	5.65	2.52	0.83	4.89	6.88	40	310.2	8.29		
12	4(+1)	16(+1)	90(-1)	380(+1)	6.14	2.49	0.79	4.73	7	31	320.27	8.22		
13	2.5(-1)	12(-1)	130(+1)	380(+1)	4.95	2.84	0.92	5.65	6.73	48	199.29	8.33		
14	4(+1)	12(-1)	130(+1)	380(+1)	5.22	2.81	0.85	5.47	6.89	43	233.6	8.27		
15	2.5(-1)	16(+1)	130(+1)	380(+1)	5.24	2.7	0.87	4.93	7.76	46	282.81	8.4		
16	4(+1)	16(+1)	130(+1)	380(+1)	5.4	2.67	0.8	4.78	7.97	42	300.03	8.3		
17	1.75(-2)	14(0)	110(0)	350(0)	4.68	2.77	0.85	5.13	6.66	38	250.34	8.33		
18	4.75(+2)	14(0)	110(0)	350(0)	5.61	2.52	0.77	5	6.85	29	277.34	8.2		
19	3.25(0)	10(-2)	110(0)	350(0)	5.93	3	0.9	5.97	6.13	34	222.81	8.19		
20	3.25(0)	18(+2)	110(0)	350(0)	6.87	2.13	0.68	4.78	8.87	28	366.05	8.28		
21	3.25(0)	14(0)	70(-2)	350(0)	6.96	1.51	0.76	4.42	6.33	27	350	8.12		
22	3.25(0)	14(0)	150(+2)	350(0)	5	2.03	0.82	4.98	8.73	48	220.9	8.33		
23	3.25(0)	14(0)	110(0)	290(-2)	7.1	2.33	0.73	4.34	6.18	34	368.4	8.25		
24	3.25(0)	14(0)	110(0)	410(+2)	5.74	3.66	0.91	5.15	5.44	50	300.37	8.3		
25	3.25(0)	14(0)	110(0)	350(0)	6.63	2.1	0.8	4.87	6.5	42	346.99	8.29		
26	3.25(0)	14(0)	110(0)	350(0)	6.58	2.11	0.83	4.85	6.61	43	365.53	8.29		
27	3.25(0)	14(0)	110(0)	350(0)	6.64	2.13	0.86	4.8	6.66	40	376.41	8.32		
28	3.25(0)	14(0)	110(0)	350(0)	6.44	2.15	0.8	4.82	6.57	39	369.18	8.33		
29	3.25(0)	14(0)	110(0)	350(0)	6.47	2.09	0.75	4.9	6.72	40	376.16	8.36		
30	3.25(0)	14(0)	110(0)	350(0)	6.36	1.99	0.82	4.86	6.59	41	385.78	8.3		

IHCF Indian horse chestnut flour; coded values are in parenthesis; BD bulk density (g/mL); SE sectional expansion index; ε porosity; RR rehydration ratio; WAI water absorption index; WSI water solubility index; OA overall acceptability

where M_0 represents initial water content, M_t water content at time t (min) and M_e represents water content at equilibrium.

Data obtained from the rehydration rate was used to determine the water diffusion coefficient using Fick's law equation as given below.

$$X_w = X_e + (X_0 - X_e) \frac{8}{\pi^2} e^{-\frac{D_{\text{eff}} \pi^2 t}{4L^2}},$$

where X_w , X_e , X_0 is the moisture content of the sample at time t , equilibrium, and initial respectively. L = half thickness of the extrudates, t = rehydration time, D_{eff} is the effective diffusion coefficient (m^2/s).

Functional attributes of extrudates

Water absorption indices (WAI) and water solubility index (WSI)

Both water absorption indices (WAI) and water solubility index (WSI) were calculated by [16] method with slight modifications. Around 2 g of extrudates were mixed with distilled water (25 mL) in a pre-weighed centrifuge tube for 60 min at ambient temperature (22 °C) with periodic stirring, and then centrifuged at $3000 \times g$ for 10 min. The supernatant collected was subjected to oven drying at 70 °C till constant weight was achieved. The WAI and WSI are expressed under

$$\text{WAI(g/g)} = \frac{\text{Weight of sediment (wet basis) (g)}}{\text{weight of sample (g)}}$$

$$\text{WSI(\%)} = \frac{\text{weight of solids dissolved in the supernatants(dry basis)}}{\text{weight of sample}} \times 100$$

Mechanical property of extrudates

A texture analyzer (TA-HD Plus, Stable Micro System, Godalming, Surrey, UK) attached with 5 blade kramer shear cell was used to measure the hardness of breakfast cereals. The test was carried out at 1.0 mm/s of pre-test speed, 2 mm/s test speed and 10.00 mm/s of post-test speed [10].

Sensory evaluation

The panel of thirty consumers carried out the sensory evaluation of breakfast cereals on a 9-point Hedonic scale (9—like very much and 1—dislike very much). Samples were coded in a 3-digit manner and served to panelists randomly for the evaluation of different attributes such as taste, crispiness and overall quality). The panelists were well known for the rating technique. The overall acceptability of breakfast

cereals was evaluated as the average score of sensory attributes determined.

Optimal point and validation

To validate the developed models, independent variables were optimized using the highest desirability function i.e., a comparison of predicted and actual values. The ideal conditions considered for numerical optimization were to minimize the BD, and hardness, and to maximize the SE, porosity, RR, WAI, WSI and overall acceptability. The developed breakfast cereals were evaluated for all the chosen parameters and % prediction error was measured as follows [17].

$$\text{Prediction error(\%)} = \frac{\text{Actual value} - \text{predicted value}}{\text{Predicted value}} \times 100$$

Pasting properties

Pasting properties of native whole grain flours, IHCF, their blends, and five different optimized extruded flours [same feed composition (10% WWF, 10% WBF, 2.5% IHCF, and 77.5% WCF) and process conditions (barrel temperature 130 °C and screw speed 380 rpm) but varying feed moisture content (12%, 13%, 14%, 15%, and 16%)] were determined using a Rheometer Physica MCR 101 (Anton Paar, Ostfildern, Austria) with a standard procedure as reported by [18] with slight modifications. 3 g of weighed flour was put directly in the canister and 25 mL of distilled water was added. A heating and cooling profile were programmed starting at 50 °C for 1 min followed by heating up to 95 °C

for 12 °C/min, then held for 2.5 min at 95 °C and cooled from 95 to 50 °C at a rate of 12 °C/min and lastly held for 2 min at 50 °C. The parameters recorded were peak viscosity (PV), breakdown viscosity (BV), final viscosity (FV), peak time (PT), setback viscosity (SV), trough viscosity (TV), and pasting temperature (PaT).

Rheological characteristics

A paste of native samples, their blend, and extruded flour was prepared by blending 3 g of flour with 30 mL of distilled water, and stirred thoroughly on a magnetic stirrer. All the samples were allowed to rest for 5 min as equilibration time according to the method adopted by [19]. A parallel plate geometry (50 mm plate diameter) was used for the steady shear rate ramp test. Rheometer Physica MCR 101 (Anton Paar, Ostfildern, Austria) with a Peltier

temperature-controlling system was used. The top plate was slowly lowered until the final sample thickness was 1 mm. All the experiments were conducted at 25 °C. The flow behavior of the slurry i.e., shear rate versus shear stress was studied at a constant shear rate from 0.1 to 150/s. The data obtained on shear rate ($\dot{\gamma}$) vs shear stress (σ) were subjected to the power-law model using the below equation:

$$\sigma = k\dot{\gamma}^n$$

where k and n are the consistency coefficient and flow behavior index respectively.

Rheological properties were determined by using the same stress-controlled rheometer as mentioned above equipped with a controlled temperature of 25 °C. The appropriate amount of slurry was placed on the lower plate and the upper plate was dropped until the gap between the two parallel plates reached 1 mm. The excess sample was trimmed, and the exposed edges of the suspension were applied with a thin layer of oil to prevent moisture loss during experimentation. For dynamic viscoelastic properties, a fixed strain of 2% was maintained within the linear viscoelastic region with a shear rate of 0.1–150/s at a constant frequency of 1 Hz to measure storage or elastic modulus (G'), loss or viscous modulus (G'') derived from the equation given below and loss tangent ($\tan \delta = G''/G'$).

$$G' = K' \cdot \omega^{n'}$$

$$G'' = K'' \cdot \omega^{n''},$$

where ω is the angular frequency (rad/s), K' , K'' , n' and n'' represents the consistency coefficients in (Pa.sⁿ) i.e., viscoelastic nature and the flow behavior index of corresponding values of G' and G'' .

FTIR spectroscopy

FTIR spectra of the native and extruded flours were gathered as an average of 40 scans using spectrum-2 LiTa spectrometer L1 600300 (Perkin Elmer, Buckinghamshire, UK) in the range of 4000–650 cm⁻¹ for native flours and 4000–700 cm⁻¹ for extruded flours with 4 cm⁻¹ resolution at 25 °C temperature. The change in functional groups was identified by observing the changes in absorbance of the peaks of hydroxyl groups (OH) at a resolution of 3000 to 3600 cm⁻¹ and the analysis for the sample was repeated thrice.

Statistical analysis

The experimental data were analyzed using central composite rotatable design (CCRD) in response surface methodology using Statistical Software Design-Expert-12 (Stat-Ease

Inc., Minneapolis, MN, USA). Data were fitted to a second-order polynomial model to obtain regression analysis and coefficients of the model with p -values ≤ 0.05 . The significance of regression ($p \leq 0.05$) and lack of fit (> 0.05) was evaluated by analysis of variance (ANOVA).

Economics of the developed optimized product

The cost of developed extrudates was optimized by achieving the highest desirability and the highest score achieved during sensory evaluation. The cost of ingredients, packaging materials and other processing charges (including fuel, water, electricity and machinery depreciation), profit (20%) and Goods and Service Tax (GST) were taken into consideration for the development of the product.

Results and discussion

Structural characteristics

Bulk density and apparent density

Density is an important variable in the postproduction of extrudates. Low extrudate density is regarded as a desirable quality. Bulk density (BD) and apparent density (AD) of the extrudates were found to vary from 4.86–7.36 g/mL and 0.4–2.35 g/mL respectively. Due to the high bulk density, there is less air between the particles, hence these are more resistant to oxidative degradation and have improved stability during storage [20] also reported BD of 6.25 g/mL for extruded products developed from whole wheat flour. Table 3 shows the model for BD where X_1 , X_2 , X_3 , and X_4 represent the concentration of IHCF, feed moisture content, barrel temperature, and screw speed respectively.

Table 3 showed a significant positive linear effect while X_3 and X_4 showed a negative linear effect. The quadratic terms in Table 3 showed negative effects on BD. Among all the four independent variables, barrel temperature (X_3) had the main impact on BD, which is parallel with the findings of [21]. Apparent density does not consider voids between the particles and corresponds to true density. The results for apparent density (AD) are graphically presented in Fig. 1a–d. To determine the effect of independent variables on AD, one independent factor was varied, keeping other factors constant. BD (Table 2) and AD (Fig. 1a–d) increased with increasing IHCF and feed moisture content whereas, elevation in screw speed and barrel temperature, decreased in BD and AD, as reported by [22]. The increase in density is due to the increased percentage of fiber content in IHCF than whole-grain flours, suggesting for maximum dispersibility of flours and food products [23]. Fibre typically causes the cell walls to rupture before the formation of bubbles,

Table 3 ANOVA for the fit of data to response surface models

Dependent variables	Models	R ²	Adjusted R ²	Predicted R ²	Adequate precision	C.V (%)	p-value	Lack of fit
BD (g/mL)	6.52 + 0.17X ₁ + 0.19X ₂ - 0.44X ₃ - 0.38X ₄ + 0.1125X ₂ X ₃ - 0.1788X ₂ X ₄ - 0.33X ₁ ² - 0.13X ₃ ²	0.98	0.97	0.94	32.22	1.83	<0.05	NS
SE (mm)	2.10 - 0.29X ₂ + 0.10X ₃ + 0.29X ₄ + 0.132X ₂ X ₃ + 0.138X ₂ X ₄ - 0.115 X ₃ X ₄ + 0.139X ₁ ² + 0.319X ₂ ² - 0.079X ₃ ² + 0.226X ₄ ²	0.98	0.97	0.92	34.77	3.71	<0.05	NS
ε	0.8100 - 0.0275X ₁ - 0.0400X ₂ + 0.0175X ₃ + 0.0367X ₄	0.85	0.83	0.80	23.68	3.11	<0.05	NS
RR	4.85 - 0.0529X ₁ - 0.3021X ₂ + 0.1387X ₃ + 0.2179X ₄ - 0.0494X ₂ X ₃ - 0.0769X ₃ X ₄ + 0.0453X ₁ ² + 0.1228X ₂ ² - 0.0459X ₃ ² - 0.0347X ₄ ²	0.99	0.98	0.96	51.48	0.95	<0.05	NS
WAI (g/g)	6.61 + 0.0729 X ₁ + 0.6479 X ₂ + 0.5554 X ₃ - 0.2013 X ₄ - 0.1556 X ₂ X ₃ + 0.0549 X ₁ ² + 0.241 X ₂ ² + 0.2486 X ₃ ² - 0.1814 X ₄ ²	0.98	0.97	0.94	37.97	1.87	<0.05	NS
WSI (%)	40.83 - 2.63 X ₁ - 1.54 X ₂ + 4.96 X ₃ + 4.21X ₄ - 1.19 X ₃ X ₄ - 1.70 X ₁ ² - 2.32 X ₂ ² - 0.697X ₃ ²	0.98	0.96	0.93	32.18	3.48	<0.05	NS
Hardness (N)	370.01 + 7.76 X ₁ + 42.44 X ₂ - 24.30 X ₃ - 2.43 X ₄ - 11.67 X ₂ X ₃ + 9.97 X ₃ X ₄ - 26.77 X ₁ ² - 19.12 X ₂ ² - 21.37 X ₃ ² - 9.13 X ₄ ²	0.96	0.92	0.80	16.63	5.69	<0.05	NS
OA	8.26 - 0.0304 X ₁ + 0.0304X ₂ + 0.0571 X ₃ + 0.0367 X ₄	0.79	0.76	0.73	17.72	0.44	<0.05	NS

BD bulk density (g/mL); SE sectional expansion index; ε porosity; RR rehydration ratio; WAI water absorption index; WSI water solubility index; OA overall acceptability; C.V coefficient of variance; NS non significant

limiting the total expansion of the extruded product and developing a product with a higher density and less porous structure [9]. Increment in screw speed (320–380 rpm) and barrel temperature (90–130 °C) reduces the BD and AD of extrudates due to the dextrinization of starch and disintegration of macromolecular structures of starch and protein that subsequently weakens the structure under increased shear rate [12]. Figure 1e and f showed the positive interaction effect of X₂X₃ and the negative interaction effect of X₂X₄ on BD.

Sectional expansion (SE)

Consumers mostly prefer light and puffed extrudates. SE of the developed extrudates ranged from 1.4 to 3.66 mm (Table 2). The fitted regression model for SE is shown in Table 3. The results of regression (Table 3) showed the non-significant effect of IHCF on SE. Feed moisture content, barrel temperature, and screw speed showed a significant effect on SE (p < 0.01) (Fig. 2a–c). Table 2 shows that SE increased from 1.51 to 2.03 mm as the barrel temperature increased from 70 to 150 °C. Protein cooking and starch gelatinization may increase the plasticity in turn enhancing the expansion of extrudates with increasing temperature [21]. However, cooking temperature (X₃²) had a significantly negative quadratic effect on SE. The influence of prolonged high cooking temperature reduces the heat stability and disrupts the starch granules due to hydrolysis resulting in slight expansion of extrudates developed at higher barrel temperature [24]. Although the positive quadratic effect of feed moisture content was more dominant than its negative

linear effect. Therefore, the increment in the moisture content shows a low expansion coefficient of products, as the high dietary fiber content of IHCF and whole-grain flours that were added with other starchy ingredients resulted in the rupture of cell walls and prevented air bubbles from expanding [25]. Furthermore, Table 3 shows a significant positive linear and quadratic effect of screw speed (X₄²) on SE that varied from 2.33 to 3.66 mm. Higher screw speed produces more energy to the mixture resulting in quick evaporation of moisture at the exit of the die due to sudden pressure drop and thus increases expansion [26].

Porosity and rehydration rate

Porosity (ε) and rehydration rate (RR) are positively correlated and extrudates with high porosity can also elicit a high rehydration rate with a higher diffusion coefficient [12]. In this study, ε and RR of developed extrudates ranged from 0.68 to 0.92 and 4.18 to 5.97 respectively (Table 2). ε and RR showed linear and quadratic regression models as depicted in Table 3. Regression models (Table 3) demonstrated a negative linear effect of IHCF and feed moisture content on ε and RR. This reduction can be ascribed to the high-water retention inside the extrudates as feed moisture plays multiple roles as a plasticizer, blowing agent, and modification. The rheological characteristic of the feed also decreases, lowering the friction of the rotating screw, resulting in low torque and less mechanical energy input. This negatively affects the structure of melt and consequently the porosity of extrudates [27]. Barrel temperature showed a significant

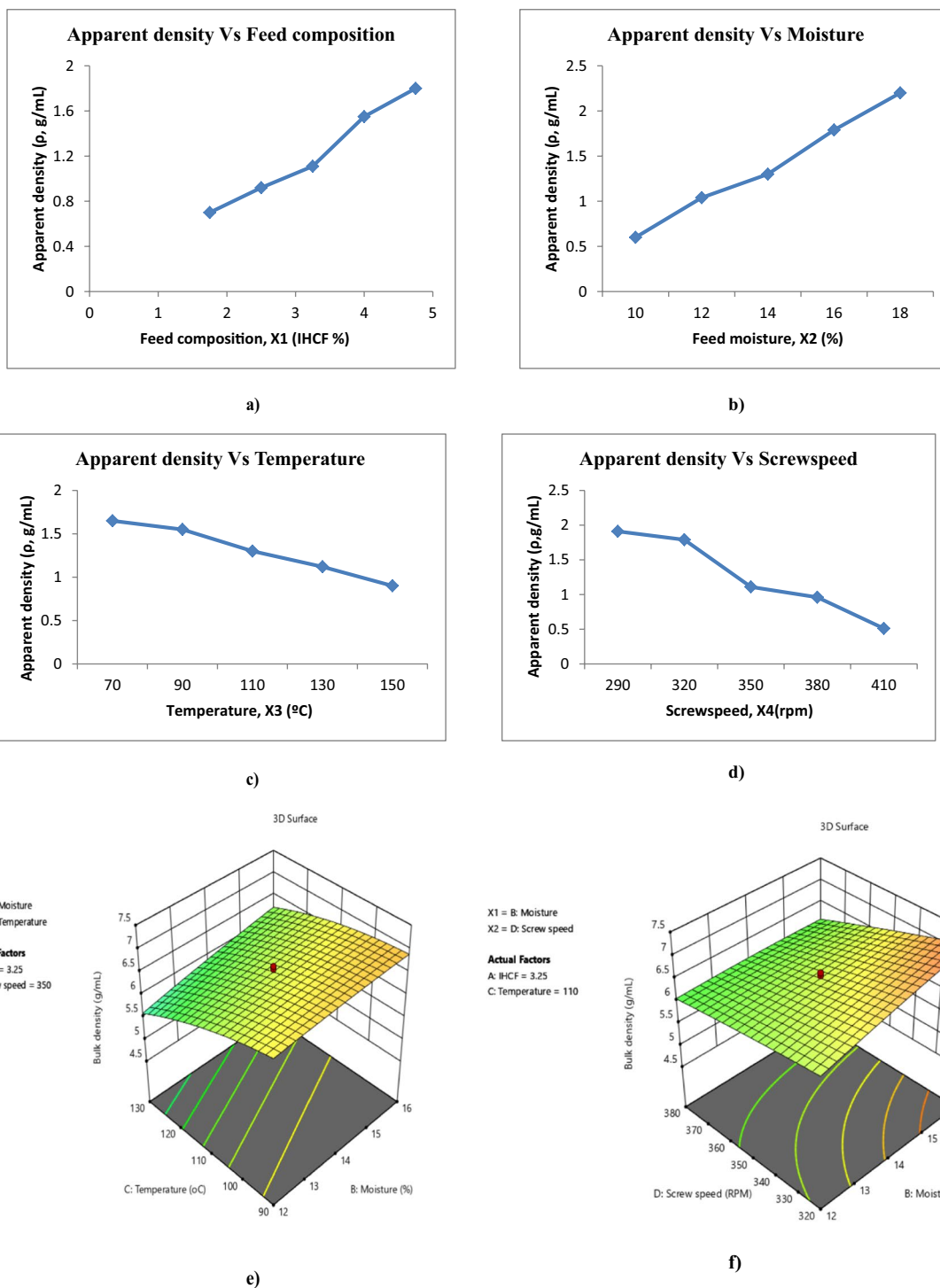


Fig. 1 Effect of **a** feed composition (IHCF); **b** feed moisture content; **c** barrel temperature and **d** screw speed on the apparent density of extrudates. Effect of **e** moisture and temperature on BD and **f** moisture and screw speed on BD

positive effect on both ϵ and RR. Such a trend has been reported for various extrudates developed by several researchers [28, 29]. High temperature creates more air cells and opens voids in the structure of extrudates by producing high thermal energy resulting in enhanced

levels of superheated steam during extrusion cooking. So, the rehydrated extruded products imbibe more water and subsequently increase the rehydration rate. Additionally, the gases released during expansion get entrapped into the matrix leading to a less dense structure with higher

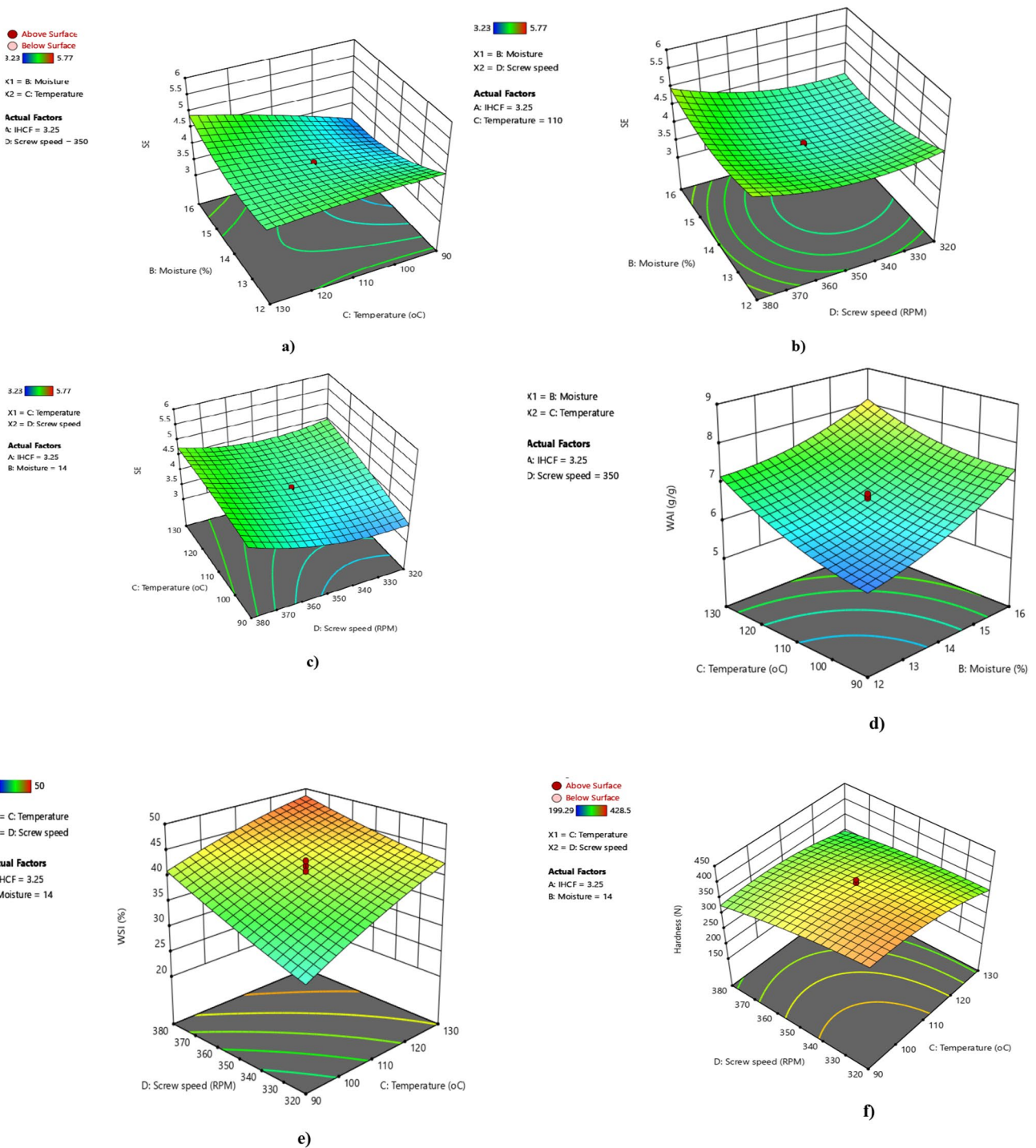


Fig. 2 Effect of **a** moisture and temperature on SE; **b** moisture and screw speed on SE and **c** screw speed and temperature on SE. **d** Effect of temperature and moisture on WAI; **e** effect of temperature

and screw speed on WSI and **f** Effect of temperature and screw speed content on hardness

expansion indicating increased porosity of the samples. Screw speed also exhibited positive relationships with both porosity and rehydration rate. ϵ increased from 0.73 to 0.91 and RR from 4.34 to 5.15 as the screw speed

increased from 290 to 410 rpm (Table 2). When the melt exits through the die, more vapours are formed due to sudden pressure drop, leading to higher porosity and puffing of dough with reduced density [12].

Functional characteristics

Water absorption index (WAI) and water solubility index (WSI)

The WAI of extrudates varied from 5.44 to 8.87 (g/g) and 22 to 50% respectively (Table 2). The fitted regression model for the water absorption index and water solubility index is given in Table 3. The results for the analysis of variance are depicted in Table 3, where a quadratic model is significant. The regression analysis indicated that all four variables had a significant effect on WAI. The coefficients of IHCF, barrel temperature, and feed moisture content showed a positive linear effect while a negative linear effect of screw speed and interaction effect of moisture and temperature (Fig. 2d) was evidenced in Table 2. IHCF contains high starch content that might have a higher rate of WAI of extruded snacks due to the availability of hydrophilic sites of starch to water molecules, thus better penetration of moisture into the porous structure of extrudates [30, 31]. The quadratic of barrel temperature and feed moisture content can be related to the modification of starch granules, where the starch molecules are disrupted, and more water remains bound to starch granules leading to enhanced WAI [21]. Moreover, higher screw speed results in an increased shear rate that disintegrates the molecules of flour which produces a higher solubility index and lower WAI of the extruded snacks [16]. This partly is due to the increased shearing of starch under severe operating conditions.

The progressive IHCF addition and the increase of feed moisture content produced significantly negative effects ($p < 0.10$) on WSI. WSI is a useful indicator of starch transformation as well as the intensity of extrusion conditions. Native starches do not absorb water, whereas extruded starches absorb water rapidly and form the gel. In this study, increasing moisture content (18%) of raw material resulted in decreased WSI. The positive relationship of WSI with barrel temperature (70–150 °C) and negative with feed moisture is a consequence of increased mechanical energy consumption of the extruder. At lower water content, extrusion cooking possibly increases starch degradation, which enhances the degree of formation of water-soluble molecules. High moisture content reduces WSI as it increases the degree of gelatinization and retards the denaturation of protein and disintegration of starch molecules [32]. Barrel temperature and screw speed (Fig. 2e) were found to be the most significant factor that exerts the greatest influence on gelatinization and melting of starch during extrusion and thus increase WSI [33].

Mechanical properties

Hardness

Hardness is an important textural characteristic of extrudates and is correlated with the cell structure and expansion of the product. A positive effect of IHCF and feed moisture on hardness was observed in Table 3 and consequently has a reverse trend with an expansion of the extrudates [10]. Changes in the characteristics of extruded products containing more fiber are due to the cell structure weakening and decreased elasticity caused by interaction between starch-fibre, resulting in a harder structure [34]. Excess water has a plasticizing effect reducing the rheological characteristics and capacity of mechanical energy dissipated in the extruder, resulting in the compressed, hard, and dense structure of extrudates with lower crispiness. Further, increasing the screw speed and barrel temperature (Fig. 2f) reduces the hardness of extrudates as less moisture is available during extrusion cooking favours expansion and results in softer extrudates [35].

Sensory evaluation

Overall acceptability (OA) of extrudates prepared from different ingredients varied from 8.11 to 8.4 (Table 2). The fitted linear model for OA is presented in Table 3, showing that OA enhanced significantly with an increase in feed moisture content, barrel temperature, and screw speed. With an increase in feed moisture content from 10 to 18%, barrel temperature from 70 to 150 °C, and screw speed from 290 to 410 rpm, OA increased from 8.19 to 8.4, 8.12 to 8.33 and 8.25 to 8.3, respectively. This is ascribed to an increase in feed composition which decreases the OA due to its unsavory taste at higher addition levels.

Optimization of concentration of IHCF and extrusion conditions

Numerical optimization was done to obtain the optimum criteria as given in Table 4. Based on the highest desirability value (0.77) (Fig. 3), the optimal criteria suggested that feed moisture content of 12%, barrel temperature of 130 °C, screw speed of 380 rpm, and IHCF concentration of 2.5% were optimum values of independent variables to produce good quality wholegrain-based sugar-free extrudates. At these optimized conditions the predicted values for bulk density (5.11), section expansion (2.66), rehydration ratio (4.9), porosity (0.81), WAI (7.63), WSI (46.8) and hardness (242) were recorded for extruded sample. The predicted

values of responses were almost similar to the actual values of 5.23, 2.74, 4.92, 0.84, 7.68, 47.3, and 242.5 recorded for BD, SE, RR, porosity, WAI, WSI and hardness respectively,

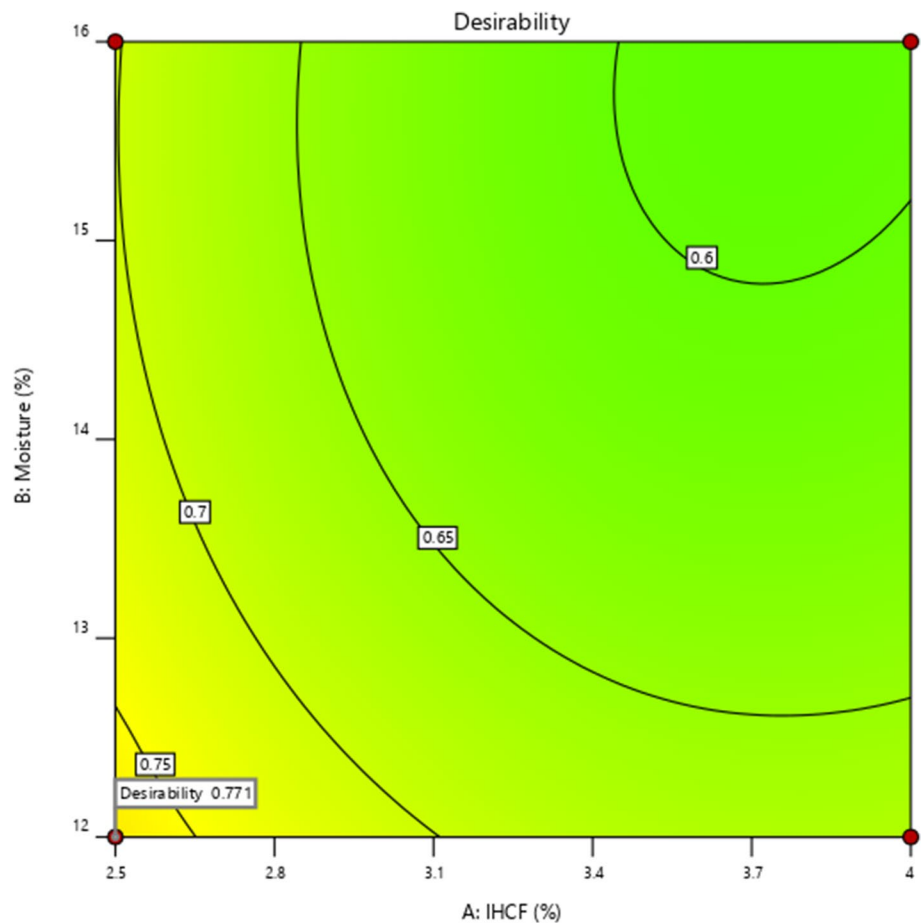
after following the optimal criteria of independent variables with a variation of less than 3.57 (Table 4).

Table 4 Numerical optimization

Variable	Goal	Range		Importance	Values		Variation level (%)
		Lower limit	Upper limit		Actual	Predicted	
IHCF	Is in range	2.5	4	3	–	–	
Feed moisture	Is in range	12	16	3	–	–	
Barrel temperature	Is in range	90	130	3	–	–	
Screw speed	Is in range	320	380	3	–	–	
Responses							
BD	Minimize	4.68	7.36	3	5.23	5.11	2.29
SE	Maximize	1.4	3.66	3	2.74	2.66	2.9
ε	Maximize	0.68	0.92	3	0.84	0.81	3.57
RR	Maximize	4.18	5.97	3	4.92	4.9	0.4
WAI	Maximize	5.44	8.87	3	7.68	7.63	0.65
WSI	Maximize	22	50	3	47.3	46.8	1.05
Hardness	Minimize	199.29	428.5	3	242.5	242.0	0.2
OA	Maximize	8.11	8.4	3	8.33	8.3	0.36

IHCF Indian horse chestnut flour; *BD* bulk density (g/mL); *SE* sectional expansion index; *ε* porosity; *RR* rehydration ratio; *WAI* water absorption index; *WSI* water solubility index; *OA* overall acceptability

Fig. 3 Response surface plot showing desirability



Moisture diffusion

Extrudates were rehydrated to investigate the influence of process variables on the uptake of water during rehydration. Extrudates were rehydrated before consumption like in a breakfast cereal. Rehydration occurred at a higher rate at the beginning of the process. The uptake of water at the early stage of the rehydration process is higher as the water fills up the surface pores of the extrudates. Subsequently, in the second stage, the diffusion coefficient shows the intermediate rehydration rate, representing the filling of remaining cells with water with a reduced rate of absorption, and finally, the process ceases as the rehydration rate approaches equilibrium. A similar trend for water absorption has been reported by [36].

Figure 4a showed the maximum water uptake capacity for breakfast cereals prepared at 10% moisture content, 110 °C barrel temperature, and 350 rpm screw speed followed by 12% feed moisture, 130 °C barrel temperature, and screw speed of 380 rpm. High feed moisture (16%), low barrel temperature (90 °C), and screw speed (320 rpm) caused a reduction in rehydration rate and consequently the reduction of porosity. The higher moisture diffusivity has a direct relation with the porosity [37], larger pores and bigger void spaces cause higher water transfer inside the extrudates. Moreover, the porous structure and texture may probably open the cells of the extrudates, resulting in higher moisture diffusivity in less time.

The diffusion coefficients were plotted against the porosity of the extruded product as shown in Fig. 4b. The values for diffusion coefficient varied from 1.06×10^{-8} to 6.64×10^{-8} . From Fig. 4c–f diffusion coefficients were reduced with IHCF and moisture content while screw speed and barrel temperature showed a reverse trend. Figure 4c–f was plotted by varying one independent parameter and keeping the rest of the parameters constant (as per optimization criteria, Table 5). Lower feed moisture means lesser water content inside the extrudates, increasing the rehydration rate and, in turn increasing the porosity and hence diffusion coefficient [12]. The extrudates processed at high barrel temperatures exhibit gelatinization of starch. Gelatinized starch has a higher water absorption capacity rather than raw starch, thus enhancing the diffusivity [38].

Pasting properties

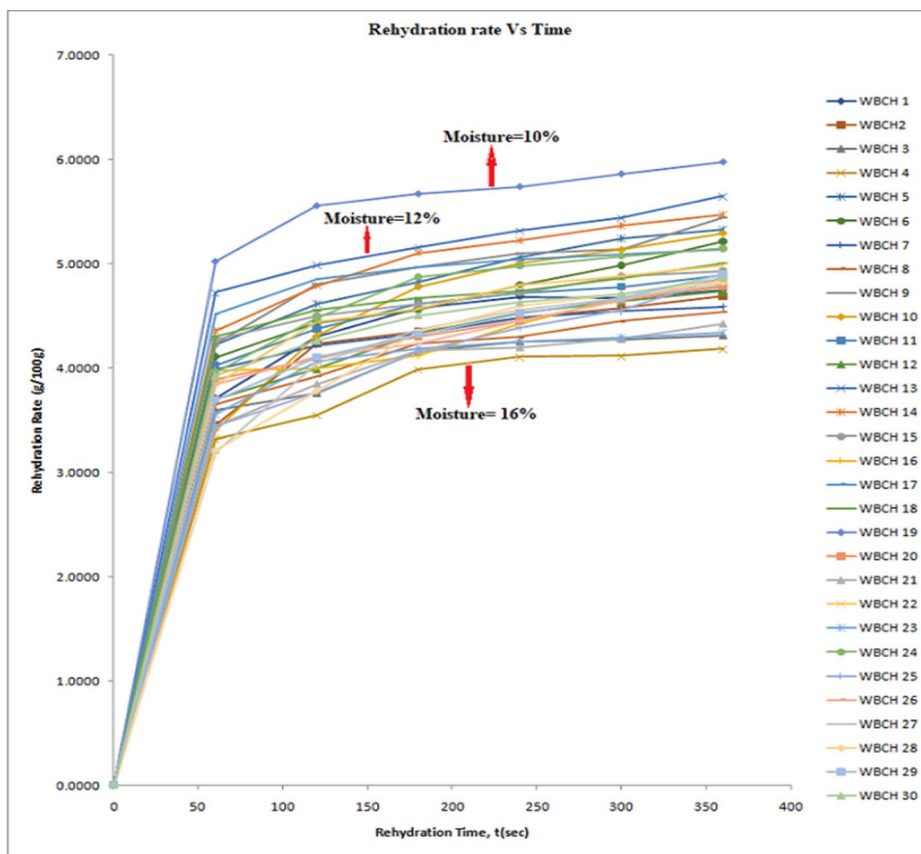
The pasting properties of native WWF, WCF, WBF, IHCF, and their blends, and extruded flour are presented in Table 6. Statistically significant variations in the pasting properties were observed among all the samples. Viscosity values [Peak viscosity (PV), breakdown viscosity (BV), final viscosity (FV), setback viscosity (SV), and trough viscosity (TV)] for the native flours were higher than those for the

extruded flour. Pasting time was higher for the native flours and decreased significantly with the extrusion process. The higher PV is due to the increased percentage of starch in flours [39]. Extruded flours had higher initial velocities than native flours samples, indicating the pregelatinized behavior of extruded flours that could absorb water and form viscous paste at ambient temperature. The increment in viscosity is mainly attributed to the degree of starch gelatinization, and the extent of the disintegration of the molecular structure during the extrusion process [40]. In this way, flour loses the tendency to swell during the thermal process and also affects the amylose leaching and amylose–lipid complex formation, resulting in reduced viscosity in comparison to the native flours. Moreover, the PV, BV, FV, and SV of the extruded flour enhanced with an increment in feed moisture content. E-16 had higher viscosity values as compared to E-12 as high moisture content gelatinize the starch partially and some ungelatinized starch was still found in the extruded flour causing higher viscosity during the thermal process. This explains that the sample with high feed moisture content reduced the stress during extrusion on disruption of the starch granule. The higher values of setback viscosity showed higher retrogradation of native flours than extruded flours owing to the reassociation of amylose during cooling. Our results are in agreement with those of [41].

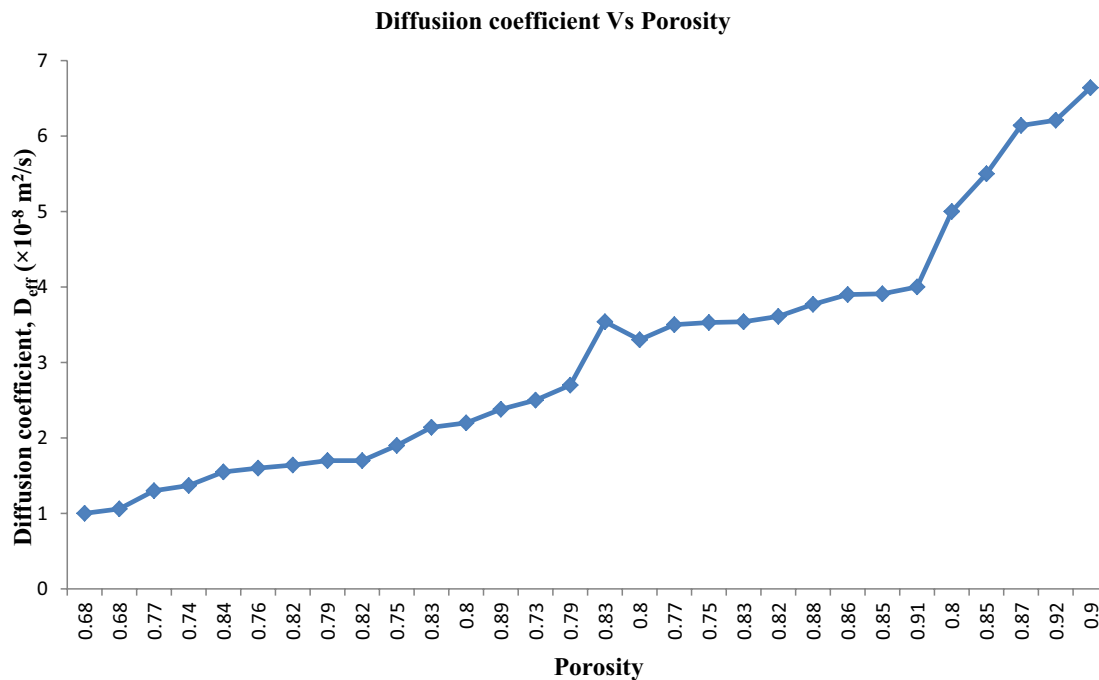
Rheological characterization of native and extruded flour

Flow characteristics

The flow behaviors of native whole grain flours (WWF, WCF, WBF and IHCF), their combination (10% WWF, 10% WBF, 2.5% IHCF and 77.5% WCF) and optimized extrudates (E-12, E-13, E-14, E-15 and E-16) under steady shear conditions at 25 °C were presented in Fig. 5a, b. The flow characterization of whole-grain flours varied significantly depending on the flour type (Table 7). All the samples in experimentation showed a shear-thinning behavior i.e., with an escalation in shear stress, the shear rate increases while as apparent viscosity reduces (Fig. 5c, d) [42]. The shear-thinning behavior is related to the decrease in the strength of the molecular network in batter due to the applied shear rate. Also, Power-law parameters were fitted to the flow curves. From Table 7, the regression coefficient, R^2 ranged from 0.981 to 0.999, indicating that flow curves fitted well with the power-law model. The closeness to Newtonian fluid was reflected by the flow behavior index (n), where n values are less than 1 showing pseudoplastic property. The consistency coefficient values for native whole grain flours ranged from 13.32 to 1.894 (Pa·sⁿ) (Table 7) with the highest value in WWF followed by IHCF, WCF, and WBF. This could be ascribed by the alignment of flour particles with the



(a) Rehydration rate of extrudates with increase in rehydration time



(b) Correlation of Porosity with diffusion coefficient

Fig. 4 **a** Rehydration rate of extrudates with increase in rehydration time. **b** Correlation of porosity with diffusion coefficient. Effect of **c** material concentration (IHCF); **d** feed moisture content; **e** barrel temperature and **f** screw speed on the diffusion coefficient of extrudates

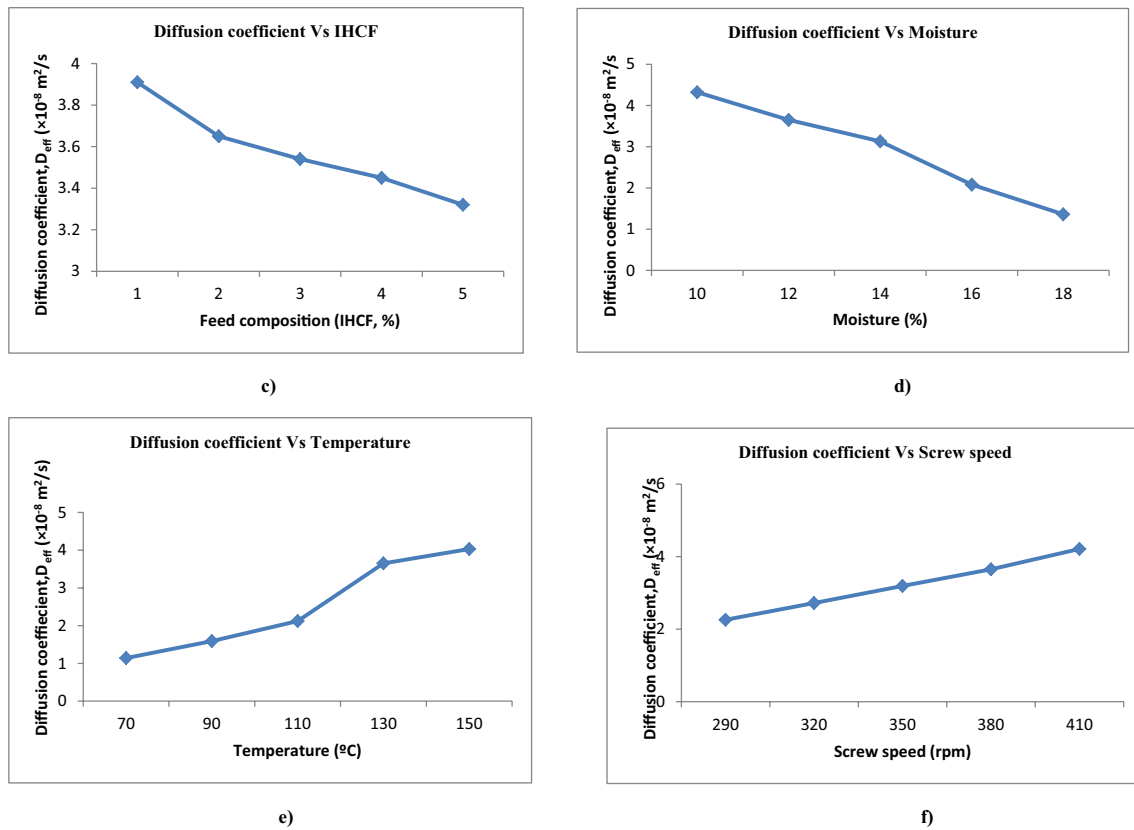


Fig. 4 (continued)

flow direction and disruption of agglomerated particles and their interaction into small molecules under the influence of shear flow, resulting in enhanced fluidity [43]. Also, starch molecules, their solubility, and the structure of amylose and amylopectin have a significant impact on the flow properties of flours [18]. Due to the pseudoplastic nature of whole-grain flours, they can easily be extruded [44]. Initially, at low shear rates, these materials have high viscosity, which helps maintain shape and structure. However, as the shear rate increases (such as during extrusion), viscosity decreases significantly. This property is advantageous in extrusion processes because it facilitates the flow of the material through the extruder die, ensuring smoother processing and shaping of the product.

Apparent viscosity, which is a measure of the consistency coefficient (K) at a shear rate of 12/s, increased with the change in treatment conditions of extruder flour. Figure 5b shows the influence of moisture content (12%, 13%, 14%, 15%, and 16%) on the variation of shear stress versus the shear rate of the extruded flour made with the same proportion of composite flour (optimized criteria) and are owed to the optimized fixed parameters of temperature (130 $^{\circ}\text{C}$) and screw speed (380 rpm) in the extrusion cooking process.

Even though the flours extruded at the same screw speed and temperature caused severe changes in the protein and starch contents of the extruded flour. The power-law model parameters illustrate that the samples developed from whole grain extrusion-modified flour provide a higher consistency coefficient (K) of batter and decreased flow behavior index (n), indicating a higher shear thinning behavior. This could be due to starch gelatinization where the particles swell and offer higher viscosity. However, the moisture content of flours during extrusion cooking had a significant effect on the flow characteristics. A higher 'n' value was found for the samples extruded at low feed moisture content. In addition, the flow behavior index (n) was reduced in all samples of extruded flour resulting in the unfolding of protein molecules which could have a positive impact on water absorption and shows increased shear-thinning behavior than native composite flour samples. Moreover, when the product exits from the extruder die, some of the moisture evaporates resulting in the closing of some pores due to volume shrinkage. This explains that E-16 differs by approximately 20% from E-12. Thus, extruded flour samples were more prone to particle alignment and disruption of their molecular structure under the influence of shear stress [19].

Table 5 Diffusion coefficient of optimized representative extrudates

IHCF	Moisture	Temperature	Screw speed	D_{eff} ($\times 10^{-8}$ m^2/s)
1.75	12	130	380	3.91
2.5	12	130	380	3.65
3.25	12	130	380	3.54
4	12	130	380	3.45
4.75	12	130	380	3.32
2.5	10	130	380	4.32
2.5	12	130	380	3.65
2.5	14	130	380	3.13
2.5	16	130	380	2.08
2.5	18	130	380	1.36
2.5	12	70	380	1.14
2.5	12	90	380	1.59
2.5	12	110	380	2.12
2.5	12	130	380	3.65
2.5	12	150	380	4.03
2.5	12	130	290	2.26
2.5	12	130	320	2.72
2.5	12	130	350	3.19
2.5	12	130	380	3.65
2.5	12	130	410	4.21

IHCF Indian horse chestnut flour; D_{eff} diffusion coefficient

Dynamic oscillatory properties

The dynamic oscillatory frequency-dependent curves were drawn to evaluate the viscoelastic characteristics of native

and extrusion-modified whole grain flours at varied levels of feed moisture content (Fig. 6a, b). For all the samples, the storage modulus (G') was greater than the loss modulus (G'') indicating an elastic behavior. Also, both moduli exhibit an increment with the angular frequency, indicating an overall increase in the chain mobility within the structure. A higher frequency leads to the quick mobility of solid particles [45]. In the case of native flours, with an increase in angular frequency, IHCF showed higher values of G' and G'' indicating a more elastic and viscous nature followed by WWF, WCF, and WBF (Fig. 6). The variation in G' and G'' among different flours could be due to the differences in the supramolecular network structure of flour [46]. Besides, among the flour modified at a different feed moisture content at the same barrel temperature (130 °C) and screw speed (380 rpm) shows a regular trend of increase in G' and G'' . The extrusion moisture significantly affected the G' and G'' of all the modified samples (Fig. 6). The highest frequency dependency curve of G' and G'' were observed in sample E-12 i.e. at the lowest feed moisture content (12%). This is in agreement with other studies that have reported that an increase in the moisture content reduces G' and G'' [47], as water may reduce the granule–granule interaction, and sample rigidity and enhance the sample deformation and granule hydration; these all changes could occur inside the extruder barrel [48]. Furthermore, the increased moisture content of the modified flour prevented the viscous energy dissipation in the extruder due to less melt viscosity. It indicates that the crystalline structure of protein and starch might be broken by swelling, inhibiting the formation of new structures and thus the sample with a higher water

Table 6 Pasting properties of extruded and unextruded flours

Treatments	Peak viscosity (Cp)	Breakdown viscosity (Cp)	Final viscosity (Cp)	Peak time (min)	Set back viscosity (Cp)	Trough viscosity (Cp)	Pasting temp (°C)
WWF	470 ± 1.14 ^c	289 ± 1.98 ^c	2163 ± 2.65 ^a	8.5 ± 0.06 ^a	1502 ± 1.54 ^a	384 ± 0.09 ^a	66.79 ± 0.08 ^f
WCF	509 ± 0.07 ^a	290 ± 1.43 ^b	665 ± 1.22 ^c	8.5 ± 1.13 ^a	156 ± 1.77 ^d	189 ± 1.65 ^e	69.15 ± 0.75 ^e
WBF	310 ± 0.23 ^c	130 ± 1.77 ^e	644 ± 1.34 ^d	8.5 ± 2.45 ^{ab}	356 ± 1.54 ^b	300 ± 0.34 ^d	66.98 ± 0.32 ^c
IHCF	450 ± 2.4 ^d	137 ± 1.34 ^d	398 ± 2.3 ^e	8.5 ± 1.15 ^a	102 ± 2.45 ^e	312 ± 0.22 ^b	61.78 ± 1.34 ⁱ
Blend	480 ± 3.3 ^b	325 ± 2.11 ^a	786 ± 2.44 ^b	8.16 ± 1.7 ^c	226 ± 1.55 ^c	302 ± 0.24 ^c	74.59 ± 2.3 ^a
Optimized extrudates							
E-12	122.8 ± 0.11 ^j	84.05 ± 0.24 ^j	68.19 ± 0.17 ^j	6.63 ± 0.09 ^f	44.6 ± 0.32 ^j	84.97 ± 0.13 ^f	66.51 ± 0.07 ^g
E-13	127.7 ± 0.24 ⁱ	84.27 ± 0.12 ⁱ	74.55 ± 0.12 ⁱ	6.85 ± 0.11 ^d	54.2 ± 1.54 ⁱ	44.27 ± 1.53 ^g	72.93 ± 0.04 ^b
E-14	132.6 ± 0.26 ^h	93.52 ± 0.05 ^h	82.59 ± 0.22 ^h	6.52 ± 0.18 ^g	60.34 ± 1.23 ^h	41.52 ± 0.11 ^h	66.02 ± 0.32 ^h
E-15	140.1 ± 0.09 ^g	100.0 ± 0.13 ^g	93.69 ± 0.04 ^g	6.76 ± 0.15 ^e	69.67 ± 1.11 ^g	39 ± 0.23 ⁱ	50.18 ± 0.12
E-16	146.2 ± 0.11 ^f	105.4 ± 0.04 ^f	104.3 ± 0.07 ^f	6.49 ± 0.34 ^h	77.41 ± 1.44 ^f	33 ± 2.34 ^j	66.93 ± 0.32 ^d

Values are mean ± SD for n=3 followed by superscripts. Values with same superscripts in a row do not differ significantly

WWF whole wheat flour; WCF whole corn flour; WBF whole barley flour; IHCF Indian horse chestnut flour; Blend (10%WWF, 10% WBF, 2.5% IHCF and 77.5% CF); E-12 extrudates with 12% feed moisture; E-13 extrudates with 13% feed moisture; E-14 extrudates with 14% feed moisture; E-15 extrudates with 15% feed moisture; E-16 extrudates with 16% feed moisture

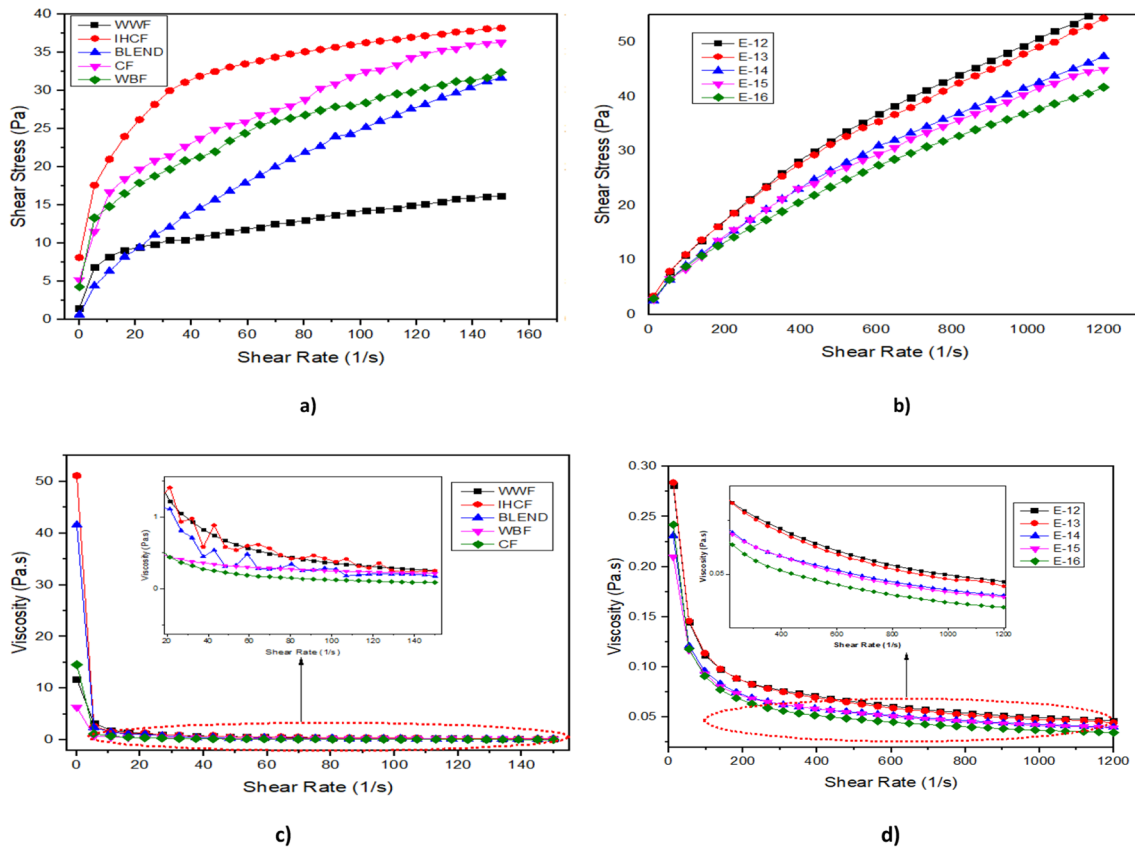


Fig. 5 Shear stress versus shear rate curves of **a** native whole grain flours: WWF, WCF, WBF, IHCF, their blend and **b** extrudates at its optimized conditions. Viscosity versus shear rate curve of **c** native

whole grain flours: WWF, CF, WBF, IHCF, their blend and **d** extrudates at its optimized conditions

Table 7 Tan δ , Power law model and statistical parameters of flow characterization of extruded and unextruded flour

Samples	Consistency (K, Pa·S ⁿ)	n	R ²	tan δ
Whole grain flours				
WWF	13.32	0.218	0.987	0.42
IHCF	8.557	0.280	0.981	2.5
WCF	3.454	0.309	0.984	0.18
WBF	1.894	0.553	0.994	0.26
Blend	7.843	0.676	0.98	0.31
Optimized extrudates				
E-12	0.692	0.596	0.996	0.63
E-13	0.650	0.613	0.998	0.71
E-14	0.594	0.627	0.997	0.8
E-15	0.530	0.62	0.997	0.87
E-16	0.5	0.641	0.999	0.92

WWF whole wheat flour; WCF corn flour; WBF whole barley flour; IHCF Indian horse chestnut flour; Blend (10% WWF, 10% WBF, 2.5% IHCF and 77.5% CF); n flow index behavior, R² coefficient of determination; E-12 optimized extrudates with 12% feed moisture; E-13 optimized extrudates with 13% feed moisture; E-14 optimized extrudates with 14% feed moisture; E-15 optimized extrudates with 15% feed moisture; E-16 optimized extrudates with 16% feed moisture

content showed comparatively less viscoelastic behavior [49]. Table 7 showed that increased G' and G'' at higher frequency resulted in reduced tan δ values. The tan δ of extruded flours decreased with a decrease in moisture content, resulting in an increment in elasticity that could be ascribed to the plasticizing effect caused by extrusion on the interactions formed between new binding zones among polymers [50].

Fourier transform infrared spectroscopy (FTIR)

FTIR spectroscopy is a non-destructive identification tool for the detection of the presence of functional groups in organic macromolecules as well as for understanding the binding interactions between these chemical compounds. FTIR spectra of native whole grain flours (WWF, CF, and WBF), IHCF, their blends, and the extrudates at optimized processing conditions (temperature and screw speed) but at varying feed moisture levels (12%, 13%, 14%, 15%, and 16%) were recorded using an FTIR spectrometer with a wavelength ranging from 4000 to 650 cm⁻¹. WWF, CF, WBF, IHCF,

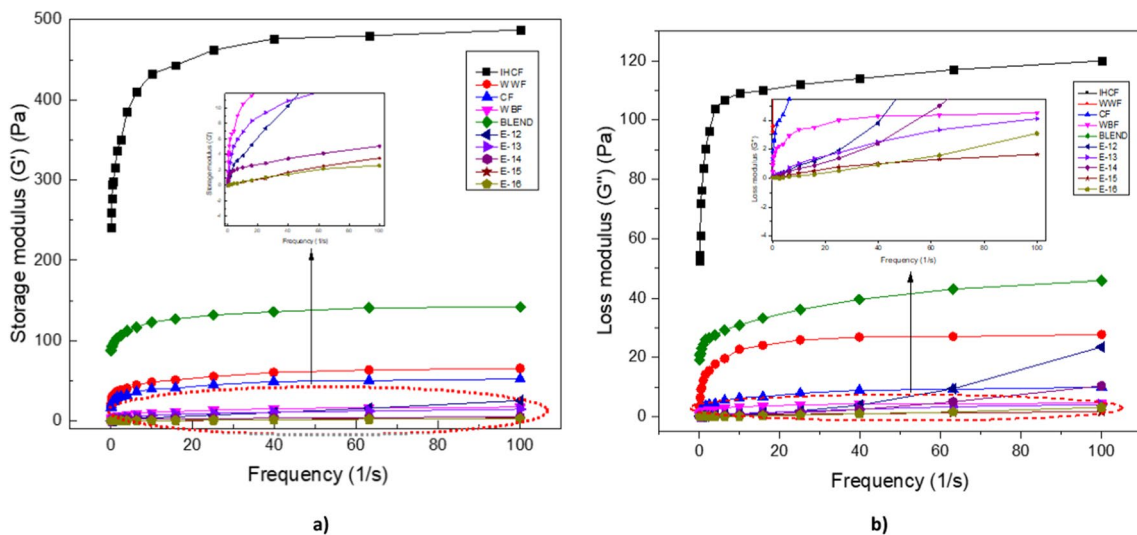


Fig. 6 a Storage modulus (G') of native whole grain flours: WWF, WCF, WBF, IHCF, their blend and extrudates at its optimized conditions and b loss modulus (G'') of native whole grain flours: WWF, WCF, WBF, IHCF, their blend and extrudates at its optimized conditions

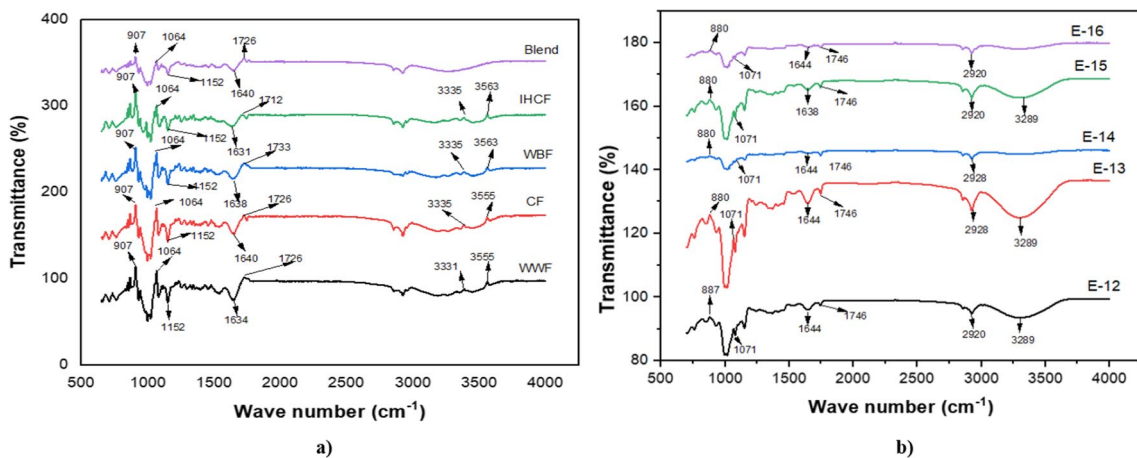


Fig. 7 FTIR spectrum of a native whole grain flours: WWF, CF, WBF, IHCF, and their blend and b Extrudates at its optimized conditions

and their blends showed similar spectral features (Fig. 7), with major peaks observed at 1700 cm^{-1} (protein region) and $1200\text{--}900\text{ cm}^{-1}$ (carbohydrate region). Figure 7a and b show the comparison between the spectra of native flours, their blends, and extruded blended flours. Common peaks for native whole grain flours, IHCF, and their blend was observed at $907\text{--}1064\text{ cm}^{-1}$ (C–O bending vibration, due to presence of cellulose and hemicellulose), 1152 cm^{-1} (coupling of C–O–O–H and C–C linkage), $1634\text{--}1726\text{ cm}^{-1}$ (H–O–H vibrations), $2852\text{--}2982\text{ cm}^{-1}$ (C–H stretch) and $3331\text{--}3555\text{ cm}^{-1}$ (O–H stretch). Similar results were shown by [51] for whole wheat flour; [52] for maize flour, [53] for barley flour; and [54] for horse chestnut. After extrusion, the

intensity of the peaks is reduced which is an indication of loss of moisture occurring during cooking as can be seen in the regions between $880\text{ and }887\text{ cm}^{-1}$ and –OH region. Figure 7b presented the spectra of different extrudates varying in their moisture content. It can be seen that all the samples showed similar spectra, as can be interpreted in the –OH stretching region. It could be because of the wide contribution from –NH bonds overlaying on the –OH band. The peaks associated with the carbohydrate region ($2820\text{ and }2920\text{ cm}^{-1}$) show both symmetric and asymmetric stretching of CH_3 . One of the most prominent modifications in FTIR spectra is the disappearance of the absorption band in a range of $1710\text{--}1740\text{ cm}^{-1}$ during the extrusion process and

the appearance of a 1746 cm^{-1} absorption band after extrusion. This indicates the presence of ester carbonyl groups of lipids (CO stretching of triglycerides) which interact with other compounds i.e., carbohydrates or proteins producing changes through the extrusion cooking process by modifying the frequencies. Also, the region near band 1644 cm^{-1} exhibited an amide I functional group from protein that shows a slightly broader peak after the extrusion, including a reduction in the absorption intensity that leads to denaturation of protein [55]. Such changes associated with denaturation can result in interactional modification between other molecules, with enhanced accessibility of the peptide bond to H–H exchange that shifts or reduces the frequency [52]. The band occurring at 1071 cm^{-1} is associated with the C–O stretching groups of the fiber.

Economics

The cost of production of sugar free whole grain based breakfast cereal enriched with IHCF (10% WWF, 10% WBF, 77.5% WCF and 2.5% IHCF) extruded at optimized process conditions (12% moisture content, $130\text{ }^{\circ}\text{C}$ temperature and 380 rpm screw speed) comes to be 12.7 per 100 g which is cheaper in comparison to commercially available breakfast cereals in the market having cost of 40 per 100 g.

Conclusion

It was observed that IHCF can be used to develop breakfast cereals with improved expansion volume, density, hardness, and water absorption index under optimum extrusion cooking conditions. RSM was found as an effective tool for the development of quality extruded breakfast cereals. The independent variables (concentration of IHCF, feed moisture content, barrel temperature, and screw speed) significantly affected the structural and functional characteristics of extrudates. IHCF of 2.5%, feed moisture content of 12%, barrel temperature of $130\text{ }^{\circ}\text{C}$, and screw speed of 380 rpm were the most appropriate conditions to prepare good quality whole grain breakfast cereal. After optimization, five different extrudates were taken with varying feed moisture content (12–16%). The feed moisture content significantly influenced the pasting and rheological characteristics of extrudates. Peak viscosity of extrudates rich in IHCF and whole grain flours having different moisture content exhibited a low tendency to retrograde. All these observations could be due to the high dietary fiber present in whole grains and IHCF. Whole corn flour exhibited high peak viscosity, probably attributed to the high lipid content present in the flour. The FTIR spectroscopy of native flours, their blends, and extrudates showed different peaks, and the intensity of

peaks was reduced during extrusion cooking due to the loss of moisture content. During extrusion, the disappearance ($1710\text{--}1740\text{ cm}^{-1}$) and the appearance (1746 cm^{-1}) of the absorption band take place, indicating the presence of ester carbonyl groups of lipids interacting with carbohydrates or proteins that produce changes by modifying the frequencies.

The overall acceptability for optimized breakfast cereal was recorded to be 8.4 which shows that the extrudates can have better acceptance. Future research should focus on expanding the understanding and application of non-conventional flours like IHCF in the food industry. By exploring alternative ingredients, optimizing processing techniques, assessing long-term health impacts, and conducting comprehensive environmental and economic analyses, the potential of IHCF-enriched cereals and similar products can be fully realized. These efforts will contribute to the development of nutritious, sustainable, and consumer-friendly food products that meet the growing demand for healthy eating options.

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Declarations

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