



# Integrated Biorefinery Strategy for Valorization of Pineapple Processing Waste into High-Value Products

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## Abstract

**Purpose** The present study aims to recover value-added products from fresh pineapple processing wastes in an integrated biorefinery.

**Methods** Bromelain, a therapeutic protease, was extracted from pineapple by-products via an aqueous, low-temperature process. Bromelain-free biomass was rich in insoluble fibre and was further fractionated into hemicellulose, cellulose and lignin.

**Results** The highest content of active bromelain was obtained from pineapple core waste of the different varieties namely, Smooth Cayenne ( $\sim 1.9 \pm 0.05$  CDU/mg), Giant Kew ( $1.6 \pm 0.05$  CDU/mg) and MD2 ( $1.5 \pm 0.1$  CDU/mg). The activity of extracted bromelain was close to commercial stem bromelain ( $2.2 \pm 0.1$  CDU/mg, % purity of 95–96%). The fractionation of fibrous residue ( $97.2 \pm 0.5$  g/100 g of dry mass) was optimised, and maximum yield of hemicellulose ( $97.5 \pm 0.2\%$ ) was obtained with 5% (w/v) alkali at the end of 1.5 h. The hemicellulose and cellulose-rich residues were further valorised into xylooligosaccharides ( $26.1 \pm 0.4$  g/100 g of hemicellulose) and glucose ( $85.3 \pm 1.7$  g/100 g of cellulose-rich residue), respectively. From one ton of fresh pineapple processing waste,  $\sim 1$  kg bromelain,  $\sim 24$  kg xylooligosaccharides,  $\sim 88$  kg glucose and  $\sim 68$  kg residual hemicellulose could be obtained.

**Conclusion** The proposed biorefinery concept not only addresses the environmental issues but also creates an opportunity to generate wealth in the form of products required for food and pharmaceutical industries from processing waste. As demand for more natural products is rising and consumers choice are prompted by healthy foods, conversion of pineapple waste into above mentioned valuable products creates a niche in food and therapeutics industry.

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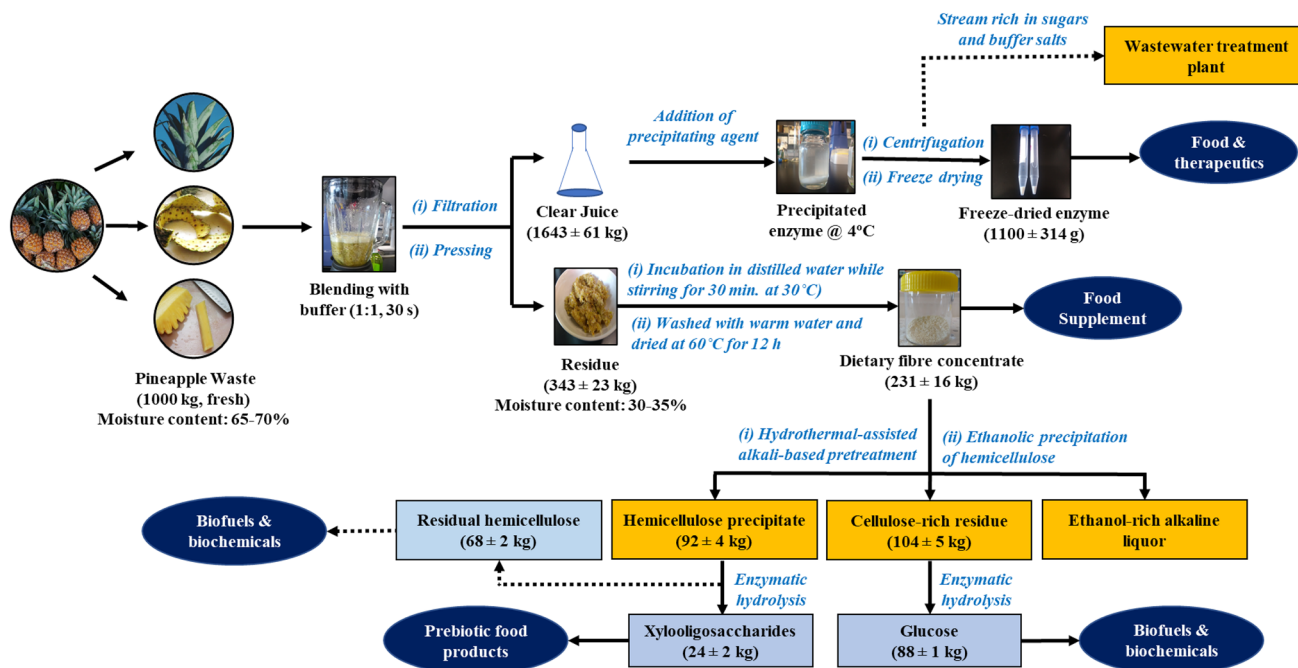
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## Graphic Abstract

Schematic for extraction of bromelain coupled with co-production of xylooligosaccharides and glucose from pineapple waste.



**Keywords** Pineapple waste · Biorefinery · Bromelain · Xylooligosaccharides · Hemicellulose

## Statement of Novelty

Pineapple is among the most widely consumed tropical fruits with a global production of 28.3 million metric tons. The fruit is widely processed as canned juice/slices and is known to generate a substantial amount of solid residues as high as 55–65% of the total weight of fresh fruit. In this study, pineapple processing waste has been demonstrated to be a valuable bioresource for the production of biofuels, biochemicals and bioproducts. This is the first attempt to propose a biorefinery approach from pineapple processing waste which offers a sustainable pathway towards the simultaneous recovery of high value products such as bromelain enzyme, xylooligosaccharides (a prebiotic) and glucose which can be further utilized as a substrate to produce high value chemicals.

## Introduction

Solid and liquid waste generated by food processing industries is among the major contributors to environmental pollution. Efficient disposal of this waste is one large problem faced by the food processing industries [1, 2]. Large amounts of such wastes, especially from the vegetable and fruit

processing industries are diverted as animal feed or as natural composts/fertilizers [3]. The strategies such as (a) reduction of the waste and (b) developing sustainable solutions to manage the residual waste could implement a circular economy in this industrial sector [4]. Waste valorization is often described as an approach of converting waste into value-added products and as a waste management strategy [4–7]. According to the European Commission [8], the bio-based economy utilizes bio-based renewable resources obtained from both land and sea, processing them into materials and energy for consumption. A fully functional bio-based economy is one of the many routes identified to achieve a circular economy. The principles of circular economy and bio-based economy work together in terms of the goal of attaining a sustainable technological and socio-economic development [9]. Food waste, mainly fruit and vegetable residues, have been demonstrated to be valuable bioresources that can find potential application in obtaining biofuels, biochemicals and bioproducts [4, 10]. The growth in demand for processed and packaged food has led to increased processing of fruits and vegetables, resulting in the generation of organic waste. The generated waste could be in the form of peels, kernels, and leaves as well as edible components that are discarded as wrong size off-cuts. The recent developments in the field of bioprocess engineering and the increasing quantum of food

by-products have also increased the interest of stakeholders in the valorization of such wastes [11–13]. The potential of various fruit wastes, such as citrus, mango, pomegranate and avocado, have been explored incorporating the biorefinery concept [14–17].

Pineapple is among the most widely consumed tropical fruits with a global production of 28.3 million metric tons in 2018 [18]. India is the sixth-largest producer of pineapples with an annual production of 1.7 million metric tons in 2018 [19] while Australia reported annual production of 0.076 million metric tons in 2018 [20]. The fruit is widely processed as canned juice/slices and is known to generate a substantial amount of solid residues as high as 55–65% of the total weight of fresh fruit [1, 11, 21]. The core and peels are a good source of a proteolytic enzyme, bromelain, which shows promising applications in the food and therapeutic sectors [22]. Pineapple waste, particularly peels and crown, are a rich source of hemicellulose. The valorization of hemicellulose in an integrated biorefinery approach could generate valuable co-products from the lignocellulosic biomass in addition to cellulosic ethanol as the major product [23]. Such an integrated process can turn agro-industrial wastes into multiple value-added products and improve the overall economics of the process. Specific valorization strategies have been proposed for pineapple waste for its integral valorization in a biorefinery approach. Gil et al. 2018 have proposed the extraction of bromelain from pineapple wastes followed by co-production of bioethanol with the remaining residue [24]. Similarly, Sepulveda et al., demonstrated the effective recovery of glycosides and polyphenols from pineapple wastes via autohydrolysis [25]. They further proposed the utilization of such value-added molecules in food, cosmetics and health products. Pineapple by-products have also been investigated as low-cost substrates for production of organic acids such as citric, ferulic and lactic acids using fermentation technology due to the commercial value of these products in the food and pharmaceutical sectors [26, 27]. Campos et al. 2020 have recently reported a green approach for valorization of pineapple by-products (peels and stem) [28]. Liquid and solid fractions were obtained from both, peels and stem. The liquid fraction was rich in two ingredients, namely, a enzymatic bromelain-rich fraction and another fraction rich in polyphenols and soluble dietary fibre [28]. In another valorization strategy, solid-state fermentation of pineapple peel biomass with *Trichoderma viride* produced high protein fungal biomass which could be recommended as suitable animal feed [29]. Casabar et al. 2019 evaluated the effects of alkaline pretreatment and microbial hydrolysis through *Trichoderma harzianum* of pineapple fruit peel [30]. Bioethanol yield of  $5.98 \pm 1.01$  g/L from pineapple fruit peel was successfully produced at 48 h of fermentation. In another study, de Ramos et al. 2020 recovered bioactive compounds and sugars from pineapple waste extract using

spray-drying technology [31]. Teixeira et al. 2021 isolated filamentous fungi compatible-consortia from pineapple wastes for cellulose-degrading enzymes production using pineapple crown waste in solid-state cultivation. Further, the potential of the cocktail enzymes was evaluated for saccharification of pineapple crown waste [32]. Along the same line, the current work focuses upon the utilization of industrial fresh pineapple processing waste in an integrated biorefinery approach. This is the first study to best of our knowledge that explores bromelain, xylooligosaccharides (XOS) and glucose as the major biorefinery products from pineapple processing wastes. Three commonly processed pineapple varieties; Smooth Cayenne (Australian), MD2 (Australian) and Giant Kew (Indian) were used in the study to compare the yields of products and impact of geographical differences on the composition of pineapple waste.

## Materials and Methods

### Raw Material

Different varieties of pineapples (Smooth Cayenne, MD2 and Giant Kew) were procured from local markets in India and Australia. The pineapples were processed by removing the crown, peeling off the skin and separating the pulp from the core. The waste parts were then frozen at  $-20^{\circ}\text{C}$  until further use.

### Chemicals

All the analytical grade chemicals were used as received. Sodium hydroxide, glacial acetic acid, the total dietary fibre assay kit and other chemicals were procured from Sigma Aldrich. Millipore water was used for preparing all other reagents. The enzymes were procured from Megazyme.

### Extraction of Bromelain

The method for extraction of bromelain was adapted from the previous study conducted by the same group [33]. Briefly, fresh pineapple processing waste (peels, core and crown) was blended with cold phosphate buffer (50 mM, pH 7.0) in the ratio 1:1 in a laboratory blender for 30 s. The liquid and the solid residue were separated using a cheese-cloth. The solid residue was diverted to another valorization stream of the biorefinery. The liquid extract was centrifuged at  $4^{\circ}\text{C}$  for 20 min at 3260 g to remove the suspended solids. The final supernatant obtained was the enzyme-rich extract and was later tested for its activity. Different precipitants (acetone, acetonitrile, ammonium sulphate, ethanol) were evaluated for precipitation efficiency. The precipitant was

added to the crude extract in the ratio 1:5 and kept for overnight precipitation of bromelain. The precipitated enzyme was centrifuged at 4 °C and freeze-dried for further characterization and activity test.

### Assay to Measure Bromelain Activity

The enzymatic activity of bromelain was expressed in terms of casein digestion unit (CDU) according to the method by Murachi with casein (0.65%, w/v) as the substrate [34]. The principle of the colorimetric assay was based on enzymatic hydrolysis of the peptide bonds in casein which results in the release of a free amino acid (L-tyrosine). Trichloroacetic acid (TCA) was used to quench the hydrolysis reaction and precipitate any unhydrolyzed casein. The precipitate was then removed through centrifugation followed by filtration with 0.45 µm syringe filters. The clear filtrate was used to record absorbance value at 280 nm on a 96-well plate reader. A blank was prepared with the reaction quenching agent (TCA) and casein solution followed by the addition of the enzyme extract. The blank sample contains unreacted casein which was precipitated before the addition of enzyme. The enzyme activity was expressed by the equation described below:

$$\text{Activity (CDU/mL)} = \frac{\mu \text{ moles of tyrosine released} * \text{total reaction volume (mL)}}{\text{time (min)} * \text{volume analysed (mL)}}$$

where one casein digestion unit (CDU) is defined as 1 µg of L-tyrosine liberated per min per mL of the sample when casein is hydrolysed for 10 min at 37 °C and pH 7.0. Experiments were conducted in triplicates, and calculations were performed with the average activity value.

### Characterization of Extracted Bromelain

#### Attenuated Total Reflection Infrared Spectroscopy (ATR-IR)

The functional features of the enzyme were determined using attenuated total reflection infrared spectroscopy. The analysis was undertaken with an FT-IR Spectrophotometer model Cary 640, provided with a universal attenuated total reflectance unit. Spectra of the extracted and commercial bromelain samples were recorded from 4000 to 600 cm<sup>-1</sup> at 64 scans, with a spectral resolution of 2 cm<sup>-1</sup> with a blank window for the background.

#### Molecular Weight Determination

The molecular weight of the enzyme samples was determined with Liquid Chromatography-Electrospray Ionization-Mass

Spectrometry (LC-ESI-MS) using MicroTOFq, a quadrupole TOF mass spectrometer (Bruker Daltonics, Bremen, Germany) coupled online with a 1200 series capillary HPLC (Agilent Technologies, Santa Clara, CA, USA). The enzyme samples were injected onto a MabPac SEC-1 5 µm, 300 Å, 50×4 mm (Thermo Scientific) column. The mobile phase constituted of 50% acetonitrile 0.05% trifluoroacetic acid, 0.05% formic acid was adjusted at a flow rate of 50 µL/min. The sample was eluted over a 20 min run-time monitored by UV detection at 254 nm. The Bruker electrospray source with a capillary voltage of 4500 V dry gas was used to nebulize and ionize the eluant at 180 °C, flow rate of 4 L/min, and nebulizer gas pressure at 300 mbar. After 20 min the flow path is switched to infuse low concentration tune mix (Agilent technologies, Santa Clara, CA, USA) to calibrate the spectrum post-acquisition. The spectra were extracted and deconvoluted using Data explorer software version 3.4 build 192 (Bruker Daltonics, Bremen, Germany).

### Extraction of Dietary Fibre Concentrate (DFC)

After the extraction of bromelain and removal of adhering free sugars, the residual material was washed thrice with

warm water (30 °C) 1:1 (v/w) followed by drying at 60 °C for 12 h in hot air oven before grinding them to a particle size of 100–500 µm. The loss of soluble fibre components as well as bioactive compounds, was minimized by washing the material with water under mild conditions.

### Chemical Analysis of DFC

Moisture, fat, protein and ash content of the DFC were determined by AOAC methods [35]. Briefly, moisture (g water/100 g sample) was determined by drying 5 g of the sample at 105 °C until a constant weight was achieved. Ash content (g ash/100 g dry matter) was determined by heating the sample in a muffle furnace at 525 ± 25 °C for 6 h. Protein (g protein/100 g dry matter) was calculated by determining the total N content of the sample on a dry basis. Fat (g fat/100 g dry matter) was determined by weight loss after a 10-cycle extraction with hexane in a Soxhlet apparatus. Total dietary fibre (TDF) and insoluble dietary fibre (IDF) were determined by the TDF assay kit based upon the enzymatic gravimetric method [36]. Soluble dietary fibre (SDF) was calculated by subtracting the IDF content from the TDF. Each assay was carried out in triplicate.

## Functional Properties of DFCs

Functional properties namely, water-holding capacity (WHC), swelling capacity (SWC) and oil-holding capacity (OHC) were investigated according to the method described by Martínez et al. with few modifications [37]. Briefly, 25 mL of phosphate buffer (1 M, pH 6.3) or commercial cooking oil (canola oil in this case) were added to 0.25 g of dry sample, stirred and left at room temperature for 1 h. The residue was weighed after centrifugation. WHC was calculated as g of water held by 1 g of sample, while OHC was articulated as g of oil held by 1 g of sample. Swelling capacity was estimated by accurately weighing 0.2 g of dry sample in a graduated measuring cylinder and adding 10 mL of phosphate buffer (1 M, pH 6.3) to it. The final volume attained by the fibre was measured after the mixture was hydrated for a period of 18 h. All the estimations were carried out in triplicate.

## Extraction of Hemicellulose from Pineapple Pomace Fibre

The cellulose, hemicellulose and lignin content of the pineapple pomace fibre were estimated by the Van Soest and NREL protocols [38–40]. Hemicellulose along with cellulose-rich residue was extracted from pineapple pomace by the hydrothermal-assisted alkali-based method [23]. Briefly, the extraction was carried out at 121 °C (without any previous incubation) with varying concentrations of sodium hydroxide (5, 10, 15% w/v) and solid to liquid ratio of 1:10 for 30, 60 and 90 min. Dried pineapple pomace powder was mixed with alkali solution of different concentrations at 121 °C in the hydrothermal reactor at 15 psi pressure. After the desired reaction time, the reactor was depressurized, and the solution was filtered. The residual matter was washed with hot water and acetone to remove the adhering sugars and alkali. The pH of the liquor was set to pH 5 with glacial acetic acid followed by overnight precipitation of hemicellulose using 95% (v/v) ice-cold ethanol [23].

## Scanning Electron Microscopy (SEM)

Morphological features of the residual biomass left after extraction of hemicellulose were analyzed using a Hitachi SU8030 Scanning Electron Microscope. Powdered samples were coated over carbon tape mounted over the sample holder. The cellulosic residue left after pre-treatment was sputtered with gold plasma to improve SEM images by reducing electrical charging of the samples.

## Enzymatic Production of XOS

The hemicellulose extracted from pineapple pomace was enzymatically hydrolyzed using endo-1,4- $\beta$ -Xylanase M1 obtained from *Trichoderma viride* (Megazyme, U.S.A.). The optimized enzymatic method (50 °C, pH 5.0 and 15 U enzyme dose) for production of XOS was adapted from Banerjee et al., 2019. The XOS (xylobiose and xylotriose) and sugar monomers produced by enzymatic hydrolysis were analyzed using HPLC (Agilent Technologies 1200 Infinity series). The sugar oligomers and monomers were eluted using 5 mM sulfuric acid in HPLC grade water as mobile phase at a flow rate of 0.7 mL/min. The samples were analyzed using Hi-Plex H column (300  $\times$  7.7 mm) attached to a refractive index detector (RID), operating at 65 °C and 50 °C, respectively.

## Enzymatic Production of Glucose

The dried cellulose-rich residue was enzymatically hydrolyzed with cellulase from *Trichoderma reesei*. The optimized conditions for enzymatic hydrolysis were obtained from previous studies with slight modifications [41, 42]. Briefly, 60 U enzyme/g substrate was incubated with 10% (w/v) solid loading of substrate at pH 5.5, 50 °C, 120 rpm and 72 h to obtain glucose. The enzyme was inactivated by heating the hydrolysate at 95 °C followed by centrifugation at 2840 g for 10 min to settle down the enzyme and the unreacted cellulose. The supernatant was filtered using 0.2  $\mu$ m pore size filter followed by determination of glucose concentration using high performance liquid chromatography.

## Statistical Analysis

All the experiments in the study were carried out in triplicates and results have been expressed as mean  $\pm$  standard deviation. One way Analysis of variance (ANOVA) was conducted to determine the statistical significance of the response ( $p < 0.05$ ). The difference between bromelain content (and enzymatic activity) present in different pineapple by-products (crown, peels, core) of the three different varieties and commercial stem bromelain was evaluated using a one-way ANOVA. The compositional difference among pineapple by-products (core, pomace and peels) for the three varieties was evaluated using two-way ANOVA. Means were compared using Tukey's test at 95% confidence interval. Minitab Statistical software version 18 (Pennsylvania State University, USA) was used for analyzing the data.

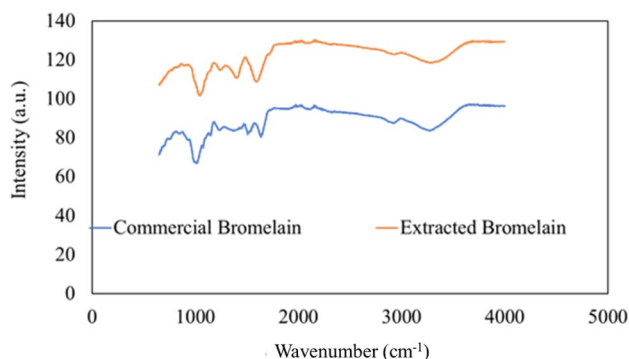


**Table 1** Bromelain content in different waste parts of the pineapple

Variety	Sample	Bromelain content (mg/mL)	Activity (CDU/mL)	Activity (CDU/mg)
Giant Kew (Indian)	Crown	1.20 ± 0.03 <sup>e</sup>	0.70 ± 0.05	0.60 ± 0.01 <sup>e</sup>
	Peels	2.0 ± 0.1 <sup>b,c</sup>	2.20 ± 0.01	1.10 ± 0.03 <sup>d</sup>
	Core	1.70 ± 0.01 <sup>c,d</sup>	2.7 ± 0.1	1.60 ± 0.05 <sup>e</sup>
Smooth Cayenne (Australian variety)	Crown	3.00 ± 0.02 <sup>a</sup>	0.90 ± 0.02	0.30 ± 0.01 <sup>f</sup>
	Peels	2.30 ± 0.01 <sup>b</sup>	1.50 ± 0.01	0.60 ± 0.01 <sup>e</sup>
	Core	1.5 ± 0.1 <sup>d,e</sup>	2.9 ± 0.1	1.90 ± 0.05 <sup>b</sup>
MD2 (Australian variety)	Crown	0.60 ± 0.02 <sup>f</sup>	0.30 ± 0.01	0.50 ± 0.01 <sup>e,f</sup>
	Peels	2.1 ± 0.1 <sup>b</sup>	1.9 ± 0.1	0.90 ± 0.01 <sup>d</sup>
	Core	2.20 ± 0.03 <sup>b</sup>	3.3 ± 0.2	1.5 ± 0.1 <sup>c</sup>
Commercial stem bromelain		0.50 ± 0.01 <sup>f</sup>	1.20 ± 0.03	2.2 ± 0.1 <sup>a</sup>

Means that do not share a letter in the same column are significantly different ( $p < 0.05$ )

CDU means Casein Digestion Unit



**Fig. 1** ATR-IR spectra of commercial and extracted bromelain

## Results and Discussion

### Effect of Precipitant on the Activity of Bromelain

The efficacy of the precipitant (acetone, acetonitrile, ammonium sulphate, ethanol) was determined by evaluating the activity of the precipitated enzyme (Figure ES1). Saturated ammonium sulphate (60% w/v) and ethanol (95%, v/v) were among the best precipitants for bromelain. Ethanol, being a green solvent, was then chosen as the precipitating agent for further experiments. Ethanolic precipitation was chosen as the purification method for its ease to scale up [43, 44]. All the by-products, namely, crown, peels and core from pineapple processing, were found to contain bromelain (Table 1). However, the highest content of active bromelain was obtained from pineapple core waste (Smooth Cayenne variety) ( $\sim 1.9 \pm 0.1$  CDU/mg) which is at par with the activity of commercial pineapple stem bromelain ( $2.2 \pm 0.1$  CDU/mg). Similarly, core waste contains the maximum amount

of active bromelain in case of other varieties, namely, Giant Kew ( $1.6 \pm 0.1$  CDU/mg) and MD2 ( $1.5 \pm 0.1$  CDU/mg). The enzymatic activity is lower than that of commercial stem bromelain but is better than the bromelain obtained from pineapple peels and crown waste.

### Characterization of Extracted Bromelain

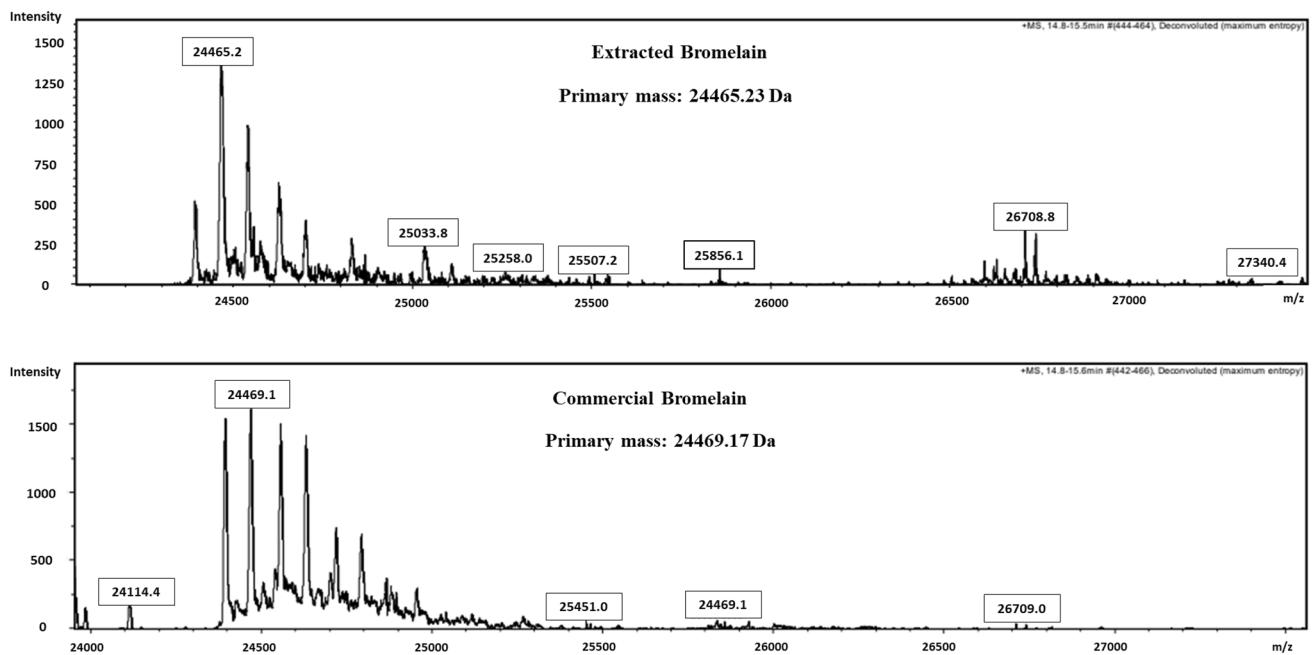
The bromelain extracted from pineapple core was then characterized using ATR-IR and molecular weight determination to ascertain its similarity with commercial stem bromelain.

### Attenuated Total Reflection Infrared Spectroscopy (ATR-IR)

Typical broad bands at  $3350\text{--}3400\text{ cm}^{-1}$  were observed for both commercial and extracted bromelain. These bands represent O–H stretching vibrations arising due to hydrogen bonds or a contribution from an N–H stretching mode. The bands between  $3000$  and  $2800\text{ cm}^{-1}$  contributed to C–H stretching vibrations. The most signature spectral region for protein structure was observed between  $1800$  and  $1500\text{ cm}^{-1}$  and corresponds to amide I and amide II (Fig. 1). The characteristic absorption bands at  $\sim 3380\text{ cm}^{-1}$  and  $\sim 1600\text{ cm}^{-1}$  contributed to the  $\text{--NH}_2$  and  $\text{--COO}$  group in the enzyme [45].

### Molecular Weight Determination

The molecular weight of commercial and extracted bromelain was estimated with Liquid Chromatography-Electrospray Ionization-Mass Spectrometry (LC-ESI-MS). The protein in the samples was detected and is seen as multiple species with a delta mass of approx. 75 Da and 88 Da (a mass difference of an unknown modification). Both samples



**Fig. 2** LC-ESI-MS spectra of extracted and commercial bromelain sample

**Table 2** Chemical composition of fibrous residue left after extraction of bromelain

Pineapple by-product	Variety	Protein (g/100 g d.m.)	Ash	Fats	Extractives
Core	Giant Kew	0.9 ± 0.1 <sup>a,b</sup>	3.2 ± 0.5 <sup>b</sup>	0.20 ± 0.03 <sup>e</sup>	22.8 ± 2.4 <sup>a,b</sup>
	Smooth Cayenne	1.5 ± 0.2 <sup>a</sup>	3.7 ± 0.2 <sup>b</sup>	1.5 ± 0.1 <sup>a</sup>	20.4 ± 1.5 <sup>a,b</sup>
	MD2	1.2 ± 0.3 <sup>a,b</sup>	2.7 ± 0.2 <sup>b</sup>	1.0 ± 0.1 <sup>a,b,c</sup>	19.9 ± 1.6 <sup>b</sup>
Pomace	Giant Kew	0.4 ± 0.2 <sup>a,b</sup>	3.7 ± 0.8 <sup>b</sup>	0.3 ± 0.1 <sup>d,e</sup>	23.5 ± 1.1 <sup>a,b</sup>
	Smooth Cayenne	1.3 ± 0.4 <sup>a,b</sup>	3.0 ± 1.0 <sup>b</sup>	1.3 ± 0.2 <sup>a,b,c</sup>	20.7 ± 1.2 <sup>a,b</sup>
	MD2	0.8 ± 0.1 <sup>a,b</sup>	3.1 ± 0.1 <sup>b</sup>	0.8 ± 0.1 <sup>b,c,d,e</sup>	19.8 ± 0.5 <sup>b</sup>
Peels	Giant Kew	0.5 ± 0.1 <sup>a,b</sup>	9.3 ± 4.2 <sup>a</sup>	0.7 ± 0.1 <sup>c,d,e</sup>	26.2 ± 1.2 <sup>a,b</sup>
	Smooth Cayenne	0.3 ± 0.1 <sup>b</sup>	10.6 ± 0.9 <sup>a</sup>	1.4 ± 0.1 <sup>a,b</sup>	29.1 ± 1.5 <sup>a</sup>
	MD2	0.5 ± 0.2 <sup>a,b</sup>	8.1 ± 1.6 <sup>a</sup>	0.9 ± 0.1 <sup>a,b,c,d</sup>	28.1 ± 2.5 <sup>a,b</sup>

Means that do not share a letter in the same column are significantly different ( $p < 0.05$ )

*d.m.* means dry matter

contained this spread of masses in the 24 kDa region (Fig. 2) [46]. The main difference between the two samples is that a species around 26 kDa was observed in the extracted bromelain but it was negligible in the commercial bromelain sample. The difference could be attributed to residual impurity in the extracted bromelain.

## Extraction of Dietary Fibre Concentrate

### Chemical Composition of Dietary Fibre Concentrates

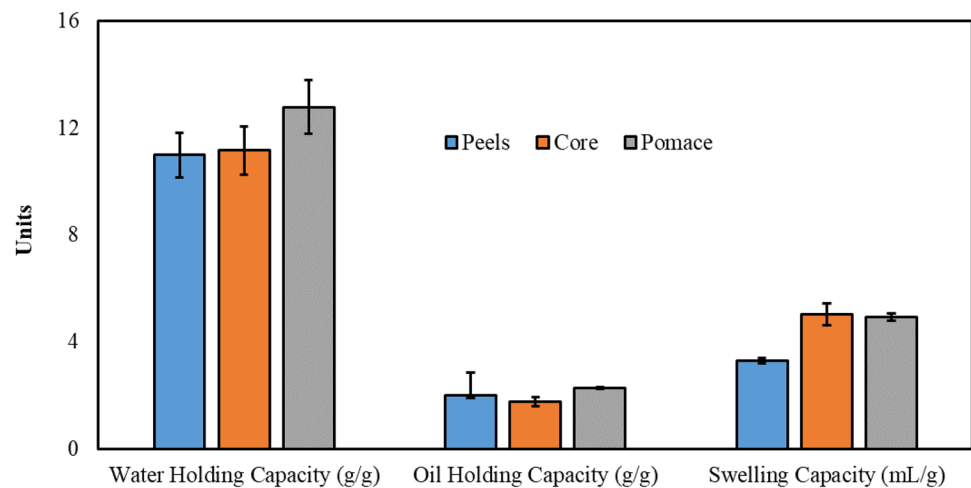
Table 2 shows the proximate composition of the residual fibrous material left after extraction of bromelain from

pineapple wastes. For all the three varieties of pineapple considered in this study, the core and pomace fibres are reported to have lower ash content in comparison to the peel. Table 3 shows the quantification of TDF, IDF and SDF in the dietary fibre concentrate. A large amount of IDF present in pineapple waste is an indicator of the significant amounts of lignocellulosic matter present in it. The soluble dietary fibre content is relatively low (~2 g per 100 g of DFC). Hu & Zhao have reported that a maximum of 8.76% of soluble dietary fibre can be obtained from pineapple pomace by shear homogenization-assisted extraction [47]. A high proportion of IDF in the dietary fibre concentrate could be considered an advantage because IDF could be used in the food industry

**Table 3** Dietary fibre content of fibrous residue left after extraction of bromelain

Variety	Part	Total dietary fibre (g/100 g d.m.)	Insoluble dietary fibre (g/100 g d.m.)	Soluble dietary fibre (g/100 g d.m.)
Giant Kew	Pomace	69.7 ± 0.5	67.8 ± 1.1	1.6 ± 0.3
	Core	70.2 ± 2.9	68.2 ± 3.6	1.8 ± 0.3
	Peels	62.0 ± 5.9	60.2 ± 6.5	1.5 ± 0.3
Smooth Cayenne	Pomace	67.2 ± 6.9	65.4 ± 7.4	1.1 ± 0.1
	Core	74.6 ± 1.2	73.5 ± 0.9	1.2 ± 0.1
	Peels	56.7 ± 3.3	55.8 ± 3.1	0.8 ± 0.1
MD2	Pomace	61.5 ± 5.7	60.3 ± 5.1	0.9 ± 0.1
	Core	63.2 ± 4.3	61.9 ± 3.3	1.1 ± 0.1
	Peels	48.5 ± 4.9	47.5 ± 4.9	0.7 ± 0.1

*d.m.* means dry matter

**Fig. 3** Functional properties of fibre concentrate from pineapple by-products

to increase the bulking effect of the food [48–50]. Besides, a higher IDF content could have beneficial health effects such as increase in satiety, increase in the volume and weight of the faecal mass, promoting better functioning of the digestive system.

### Functional Properties of Dietary Fibre Concentrate

WHC is a very significant functional property of the dietary fibre concentrate. It depicts the capacity of a moist substrate to hold water when subjected to an external centrifugal gravity force or compression [50]. WHC technically correlates with the quantity of insoluble fibre present in the dietary fibre concentrate. Among the different pineapple by-products, the fibre concentrates from pomace waste were found to possess the highest WHC i.e.  $14.4 \pm 1.5$  g water/g sample (Fig. 3). Selani et al. reported lower WHC of pineapple pomace i.e.  $5.3 \pm 0.6$  g water/g sample [50]. This variation in the values of WHC could be attributed to the differences in the material used for the analysis. The present study evaluated the dietary fibre concentrate extracted from the pineapple

and core while Selani et al. evaluated the pomace as a whole. Prakongpan et al. reported the WHC of dietary fibres extracted from pineapple pomace to be in the range 10.3–12.2 g water/g sample [51]. Since the dietary fibres from pineapple pomace and core were rich in the insoluble fibre content, their WHC value was found to be higher than orange peel (1.6 g water/g sample), lemon peel (1.7–1.8 g water/g sample) and apple pomace (1.6–1.8 g water/g sample) [52]. Swelling capacity is another significant hydration property correlating the cellulosic components present in the fibre. The dietary fibres from core ( $5.8 \pm 1.2$  mL/g) and pomace ( $5.5 \pm 0.7$  mL/g) have reported higher swelling capacity when compared to those from the peels ( $3.6 \pm 0.6$  mL/g).

OHC is another functional property related to the chemical structure of the plant polysaccharide. However, the OHC values for the fibre concentrate is quite low when compared to other fibrous materials such as coconut fibre (5.3 g oil/g fibre) [53]. Hence, the foods supplemented with these co-products will not be capable of retaining high amounts of oil. Hence, the specific use of dietary fibre in food products is largely determined by its functional properties.



**Table 4** Percentage hemicellulose recovered from pineapple pomace at different alkali concentrations and hydrothermal pretreatment conditions

Process	Alkali concentration (%)	Absolute recovery of hemicellulose (%)	Relative recovery of hemicellulose (%)
Hydrothermal (0.5 h) 121 °C	5	26.7 ± 1.1 <sup>d</sup>	55.1 ± 2.3 <sup>d</sup>
	10	30.3 ± 1.4 <sup>c</sup>	62.6 ± 2.9 <sup>c</sup>
	15	32.5 ± 0.6 <sup>c</sup>	67.2 ± 1.3 <sup>c</sup>
Hydrothermal (1.0 h) 121 °C	5	43.2 ± 0.9 <sup>b</sup>	89.3 ± 1.8 <sup>b</sup>
	10	44.4 ± 0.6 <sup>a,b</sup>	91.8 ± 1.3 <sup>a,b</sup>
	15	46.1 ± 1.3 <sup>a,b</sup>	95.1 ± 2.7 <sup>a,b</sup>
Hydrothermal (1.5 h) 121 °C	5	47.2 ± 0.1 <sup>a</sup>	97.5 ± 0.2 <sup>a</sup>
	10	47.8 ± 0.4 <sup>a</sup>	98.7 ± 0.8 <sup>a</sup>
	15	46.5 ± 0.6 <sup>a,b</sup>	96.2 ± 1.3 <sup>a,b</sup>

Means that do not share a letter in the same column are significantly different (p < 0.05)

**Table 5** Selected parameters and their levels for the general linear model of experiments

Factor	Levels	Values
Time (h)	3	0.5 1.0 1.5
Alkali conc. (%)	3	5 10 15

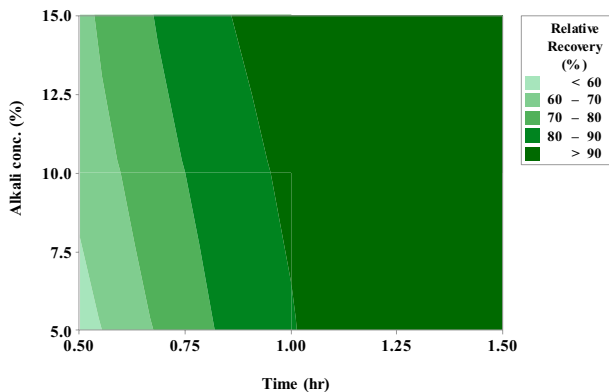
### Pineapple Pomace as a Source of Hemicellulose, XOS and Glucose

The pineapple pomace was found to be rich in hemicellulose content (48.4 ± 1.6 g per 100 g of dried extractive-free pomace) with low lignin content (2.1 ± 0.6 g per 100 g of dried extractive-free pomace), hence it was explored as a source

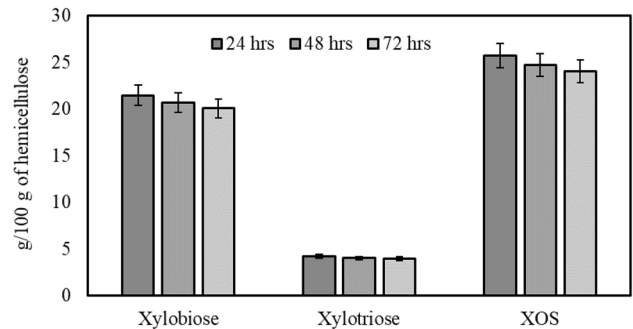
**Table 6** Analysis of variance (ANOVA) test for an experimental response for extraction temperature and alkali concentrations for recovery of hemicellulose

Source	df	Sum of squares	Mean square	F value	p-value
Time(h)	2	4476.09	2238.05	642.41	0.000
Alkali conc. (%)	2	94.79	47.39	13.6	0.002
Time (h)*alkali conc. (%)	4	94.27	23.57	6.76	0.008
Error	9	31.35	3.48		
Total	17	4696.50			

R<sup>2</sup> = 99.3%; Adjusted R<sup>2</sup> = 98.7%; Predicted R<sup>2</sup> = 97.3%

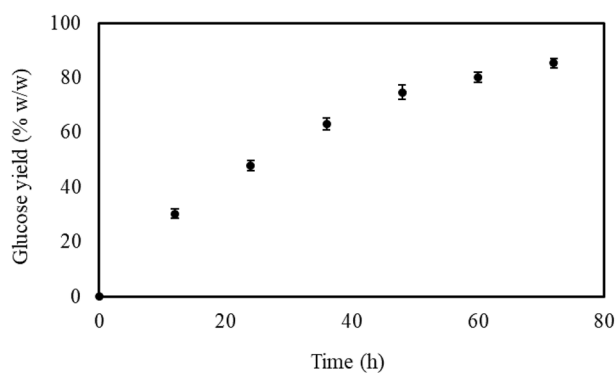


**Fig. 4** Contour plot showing the influence of alkali concentration and extraction time on relative recovery of hemicellulose from pineapple pomace



**Fig. 5** Production of XOS under optimal experimental conditions

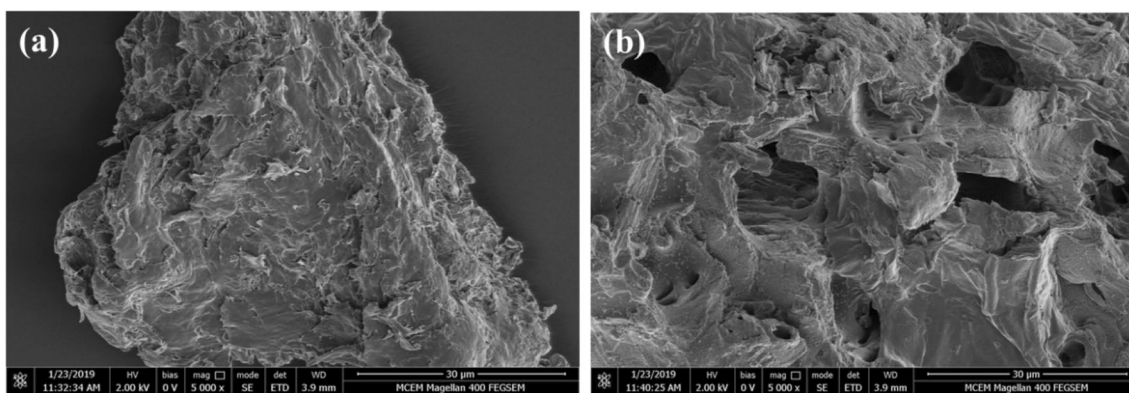
of hemicellulose for production of XOS. The maximum relative recovery of  $98.7 \pm 0.8\%$  of hemicellulose was obtained with 10% (w/v) alkali at the end of 1.5 h of hydrothermal extraction (121 °C and 15 psi pressure). A significant yield of hemicellulose ( $97.5 \pm 0.2\%$ ) was obtained with 5% (w/v) alkali at the end of 1.5 h (Table 4). The statistical model for recovery of hemicellulose contains two main effect terms, such as time (h) and alkali concentration (%) along with one interaction term. Statistical results of the model showed that the individual terms and interaction terms were significant (Table 5, 6). The adjusted  $R^2$  for this model was 98.7% which indicates a good fit of the model. The contour plot (Fig. 4) shows that the extraction time is the most influential factor in the recovery of hemicellulose from pineapple pomace waste. Above 1 h of extraction time, more than 90% of the hemicellulose could be recovered with all concentrations of alkali. No significant increase was observed in the % recovery of hemicellulose when the alkali concentration was increased at extraction times more than 1 h. This could be attributed to the synergistic effect of time-alkali concentration combination on the % recovery of hemicellulose. Higher alkali concentrations are often associated with higher costs and downstream challenges. In addition to this, another disadvantage associated with higher alkali dosage is the significant loss of polysaccharides [23]. Therefore, in the present study, a combination of 5% (w/v) alkali and 1.5 h duration was selected to pretreat pineapple pomace for hemicellulose recovery for further processing into XOS. Under the optimal experimental conditions of 50 °C, pH 5.0 and enzyme dosage 15 U/mg, a maximum of  $26.1 \pm 0.4$  g of XOS was obtained per 100 g of hemicellulose at the end of 24 h (Fig. 5). The total XOS consists of xylobiose and xylotriose which are known for their maximum prebiotic potential [23, 54]. The residual hemicellulose can further be converted into a wide range of value-added chemicals such as butanol, furfural derivatives, xylitol and others



**Fig. 7** Glucose yield during enzymatic hydrolysis of cellulose-rich pineapple residue left after extraction of hemicellulose

[55–62]. Furfurals further find potential application in the pharmaceutical, plastics and agro-chemicals industries [55]. Conversion of pentose sugars into ethanol is another widely studied approach for valorizing hemicellulose in the lignocellulosic biorefineries [63–66].

The pomace powder was analysed by scanning electron microscopy for its morphological changes after the extraction of hemicellulose. The SEM micrographs (Fig. 6) show that the structure of cellulosic pomace residue has opened up after the hydrothermal-assisted alkali treatment and is more prone to enzymatic degradation for its easy conversion into glucose which can further be valorized to obtain other value-added chemicals. The enzymatic hydrolysis of cellulose led to a recovery of  $85.3 \pm 1.7$  g of glucose per 100 g of cellulose-rich residue at the end of 72 h (Fig. 7) which is at par with the recovery of glucose reported in literature. Singh et al. 2017, reported the production of  $71.5 \pm 1.9$  g of glucose per 100 g of pre-treated arecanut husk biomass via enzymatic hydrolysis [67].



**Fig. 6** Scanning Electron Micrographs of pineapple pomace **a** before and **b** after hydrothermal-assisted alkali-based extraction of hemicellulose

## Conclusion

In this study, the valorization approach was focussed upon the generation of multiple products in an integrated biorefinery using fresh pineapple processing waste as the feedstock. Bromelain was obtained as a crude enzyme extract. Further purification was done by precipitating the enzyme using ethanol. The highest content of active bromelain was obtained from pineapple core waste (Smooth Cayenne variety) ( $\sim 1.9 \pm 0.1$  CDU/mg) which is close to the activity of commercial pineapple stem bromelain ( $2.2 \pm 0.1$  CDU/mg, % purity of 95–96%). The core waste from other varieties of pineapple, i.e. Giant Kew ( $1.6 \pm 0.1$  CDU/mg) and MD2 ( $1.5 \pm 0.1$  CDU/mg), also produced bromelain with similar enzymatic activity. The residue left after extraction of bromelain was found to be rich in insoluble fibre content and was analysed for its functional properties. Among the different pineapple by-products, the fibre concentrates from pomace were found to possess the highest water-holding capacity i.e.  $14.4 \pm 1.5$  g water/g sample followed by peels ( $11.2 \pm 1.1$  g water/g sample) and core ( $11.6 \pm 1.3$  g water/g sample). Thus, pomace can be blended with soluble dietary fibres such as apple pomace (rich in pectin) to meet the requirements for both soluble and insoluble fibres. Being low in lignin content ( $2.1 \pm 0.6$  g per 100 g of dried extractive-free sample), pomace was further fractionated into hemicellulose and cellulose-rich residues which were valorized into XOS ( $26.1 \pm 0.4$  g/100 g of hemicellulose) and glucose ( $85.3 \pm 1.7$  g/100 g of cellulose in pomace), respectively. The glucose can further be converted into potential biofuels and biochemicals in the biorefinery portfolio. The products (bromelain, insoluble dietary fibre, XOS and glucose) have an existing market demand in the food and therapeutic sector. Therefore, the biorefinery approach described in this work might help in the overall utilization of fresh pineapple processing waste. Methods for complete utilization of by-products resulting from pineapple processing should be developed on a large scale and at affordable levels. Life-cycle analysis and techno-economic feasibility studies must be conducted to evaluate the commercial viability of the proposed biorefinery.

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