

Chapter 2

**To study different extraction methods on dietary fiber from queen
pineapple waste of Northeast India and its process optimization**

2.1 Introduction

The pineapple (*Ananas comosus*), a tropical fruit belonging to the Bromeliaceae family, is well-known around the world for its high nutritional value and medicinal characteristics, which include antioxidant, anticancer, and hypoglycemic capabilities (Sepulveda et al., 2018). In 2019, over 26 million tonnes of pineapple were globally produced according to FAO reports (FAO, Rome 2020). This fruit can be consumed raw or processed into concentrated juices, fruit salads, candy, and jams. Canned pineapple accounts for a considerable portion of the harvested pineapple. The leaves, stem, crown, peel, core, and pomace of pineapple generate thousands of tonnes of waste as an agricultural by-product during various processes, and these wastes typically account for half of the entire fruit's weight. Therefore, growth in the production of pineapple will be commensurate with an increase in the production of waste (Hadidi et al., 2020).

Pineapple waste has high levels of bioactive chemicals such as dietary fiber (DF), polysaccharides, mineral compounds, vitamins, enzymes, proteins, and other volatile compounds which are spoilable, if not treated. Thus, causing not only a significant generation of waste but also contamination of the environment (Zhang et al., 2017; Luo et al., 2017). These results showed waste extraction and utilization are crucial in both practical and economic terms. Dietary fiber (DF) is produced mostly from cereals, legumes, fruits, and vegetables, and is characterized to be an edible component of entire plants or a carbohydrate polymer with favorable physiological qualities (Zhu et al., 2018).

Dietary fiber (DF) is much more resistant to digestion and absorption in the human small intestine, but it can be partly or fully fermented to produce short-chain fatty acids in the large intestine (Chen et al., 2018). Recently, DF has got a lot of attention because of its cost-effectiveness and good physiological benefits to human health, such as lowering the risk of cardiovascular disease, colon cancer, arteriosclerosis, diabetes, and obesity (Zheng et al., 2018) (Hu & Zhao, 2018). DF can be subdivided based on their solubility: insoluble and soluble dietary fibers (Luo et al., 2018).

Adsorption of heavy metals, bile acids, and cholesterol, reduction of postprandial blood glucose levels, improvement of intestinal microbiota, and prevention of diabetes and other cardiovascular illnesses are all benefits of soluble dietary fiber (SDF). On the other hand, insoluble dietary fiber (IDF) includes cellulose, lignin, and insoluble hemicelluloses, having a

great swelling capacity along with water-holding capacity which further increases fecal bulk and hence reduces the risk of colon cancer, constipation, and obesity (Wang et al., 2015). Not only does DF have physico-chemical and functional features, but it has also a higher viscosity and superior texture, making it easier to use in food preparation. As a result, finding DF from agricultural waste is a thrust area in the food industry's development (Hu & Zhao, 2018).

For the application of plant bioactive components, the extraction process is very critical. The composition and structure of DF are altered during processing, causing positive as well as detrimental effects on their physico-chemical and functional qualities (Al-Dhabi et al., 2020). Thermal, mechanical, chemical, enzymatic, and fermentation methods have been for the extraction of DF from pineapple waste in the past (Hu & Zhao, 2018) and these methods have several drawbacks, including long chemical reaction times, high solvent intake, excessive temperature requirements, and the insertion of a large number of ions throughout the reaction procedure.

Ultrasound-assisted extraction (UAE) has become a standard technique for extracting bioactive compounds such as phytochemicals, and other secondary metabolites from a variety of plants, owing to its significant reductions in reaction time, energy, and solvent usage. Furthermore, when compared with traditional extraction procedures, ultrasonication is more secure and affordable along with higher extraction yield (Fan et al., 2015). The breakdown of the cell matrix, improves mass transfer of liquid solvent used within the solid solutes, and accelerates the release of desired compounds from the matrix structure to the liquid solvent are all caused by sonication cavitation of bubbles in UAE (Gayas et al., 2020). The particle size of dietary fibre is connected to its functional characteristics. In general, smaller particle size equals more surface area and greater yields, which can be obtained using a UAE process that combines homogenization and hydrolysis to produce bioactive DF. Diabetes mellitus is a frequent and widespread disease that affects people all over the countries. Insulin insufficiency or insensitivity causes glucose to build up in the blood, resulting in a variety of symptoms (Gallagher et al., 2003). Plant extracts are a rich source of bioactive compounds such as DF for improving blood glucose disease and reducing chronic disease in people with diabetes (Chau et al., 2004). There is scant research on UAE of DF from pineapple waste especially from the most demanding variety Queen at the international level.

In the present study, UAE method was used to extract DF from Queen pineapple peel waste, and the conditions of extraction were adjusted using a Box-Behnken design using RSM. The

functional properties viz., water holding capacity (WHC), oil holding capacity (OHC), swelling capacity (SC), cation exchange capacity (CEC), emulsion activity (EA), and emulsion stability (ES) of DF were compared between UAE and alkali extraction methods. Furthermore, Fourier transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD), and scanning electron microscopy (SEM) were used to assess the potential structural features of DF. The thermal stability of DF was further examined using thermogravimetric analysis (TGA). The effect of extracted DF on hypoglycaemic activity through glucose adsorption capacity (GAC) and glucose dialysis retardation index (GDRI) was investigated. These findings could serve as a technical guide for making efficient use of highly demanding Queen pineapple in developing functional foods that can be further exploited in the food industries.

2.2 Materials and Methods

2.2.1 Raw materials and chemicals

Naturally ripened Queen variety of pineapples were collected from an agricultural farm located in Tripura (India) because the best Queen variety is grown in this region only (Das et al., 2021). All the chemicals were procured from Sigma-Aldrich Chemicals Pvt Ltd.

The pineapple peels were dried at 40°C till the equilibrium moisture content was obtained in an electric tray drier (Labotech, BDI-51, India) to avoid changes in functional attributes. Grinding of the pineapple waste sample was done using a lab-scale grinder. The ground powdered sample was then sieved through a 100-mesh size to get the dried powder and it was de-oiled using ethanol.

2.2.2 Extraction of dietary fiber

With a few minor modifications, the alkaline extraction technique as described by Ma and Mu (2016) and Zhang et al. (2017) was used to extract dietary fiber from Queen pineapple waste. The de-oiled pineapple waste dried powder was combined with 0.5M NaOH (pH 13.69) and agitated at 1000 rpm using a laboratory-based magnetic stirrer (RQT-12TD, Remi, Maharashtra, India) at 60°C for 15 minutes. The mixture was then neutralized with 0.5M HCl (pH 0.3). This is the sequence in which the Queen pineapple waste DF was extracted. The precipitate was collected by adding 85% ethanol (2:1 v/v) (Standing time was 1h). Finally, centrifugation was done at 7000 rpm for 30 min to collect the pellet and it was dried at 40°C

overnight. The extract was renamed alkaline extracted dietary fibre (AEDF) and stored in refrigeration conditions for further analysis.

Ultrasound-assisted extraction was used to extract the dietary fiber from queen pineapple waste. The pineapple waste dried powder was mixed with 0.5 M NaOH solution at different solid: liquid followed by the alkaline extraction method. Further, the sample was ultrasonicated with different time ranges from 10-30 min with a shutdown temperature of 60°C using a probe ultrasonicate (Q700-220 Digital Sonicator, Qsonica LLC, USA) with an ultrasonic amplitude of 20%-50%. Then, neutralization of the sample was done using 0.5 M HCl (pH 0.3) and followed all the remaining steps as AEDF to collect the pellet and dried overnight at 40°C to obtain UAE extracted DF (UAEDF) and stored for further analysis.

2.2.3 Experimental design

The process variables of UAE that affect the yield of DF viz., solid: liquid ratio, amplitude, and sonication time were optimized by response surface methodology (RSM) (Raj & Dash, 2020). The design for the experiments was chosen as Box-Behnken design (BBD) by implementing “Design Expert” software. The following equation was used to fit the experimental data:

$$Y = b_0 + \sum_i b_i x_i + \sum \sum b_{ij} x_i x_j + \sum b_{ii} x_i^2 \quad (1)$$

Where Y is the dependent variable, b_0 is the constant coefficient; b_i , b_{ii} , and b_{ij} are linear, quadratic, and interaction coefficients, respectively; x_i and x_j are coded independent variables. The values of R^2 and adjusted R^2 were checked for the model adequacies and the significance of the model was tested using analysis of variance (ANOVA).

The yield of the extracted DF was calculated using the following equation:

$$Y (\%) = \left(\frac{w_t}{w_i} \right) \times 100 \quad (2)$$

Where w_t is the weight of extracted DF; w_i is the initial weight of pineapple waste dried powder.

2.2.4 Chemical compositional analysis of extracted dietary fiber

The proximate analysis viz., total moisture content, crude protein, crude fat, crude fiber, and ash content of Queen pineapple peel waste dried powder was determined by A.O.A.C. (2016).

The total carbohydrate content was estimated by the weight difference from all the other compounds.

2.2.5 Mineral Content: The mineral contents of AEDF and UAEDF were quantified using an Atomic absorption spectrophotometer (Thermo Scientific, ICE 3000 Series, Newington, USA) (Begum and Deka; 2019)

2.2.6 FTIR analysis of extracted dietary fiber

FTIR spectroscopy (Nicolet Instruments 410 FTIR, Thermo Scientific, USA) was employed to identify the various functional groups in dried pineapple waste powder and extracted dietary fiber (both UAEDF and AEDF). The spectrum was analyzed within the wavelength range of 400-4000 cm^{-1} with a resolution of 4 cm^{-1} (Zhang et al., 2017).

2.2.7 SEM analysis of extracted dietary fiber

The dried powder of pineapple peel waste, ultrasound-assisted extracted dietary fiber, and alkaline extracted dietary fiber were examined using a SEM (JSM-6390LV, JEOL, Japan) at 20 kV with 500X and 1000X magnifications to study their surface morphology and microstructure.

2.2.8 XRD analysis of extracted dietary fibre

The X-ray diffraction (Bruker AXS, Bruker D8 FOCUS, Germany) of pineapple peel waste dried powder, UAEDF and AEDF was determined and XRD functioned at 60 kV. The study was executed at a 2θ range of 5-80° and the degree of crystallinity was calculated as follows:

$$C (\%) = \frac{X_c \times 100}{X_c + X_a} \quad (3)$$

Where, C is the degree of crystallinity; X_c is crystalline area; X_a is non-crystalline area on the graph (Ma & Mu, 2016).

2.2.9 Thermo-gravimetry analysis (TGA) of extracted dietary fiber

The thermal analysis of pineapple peel waste dried powder, ultrasound-assisted extracted dietary fiber, and alkaline extracted dietary fiber was investigated by TGA (NETZCH TG 209F1 Libra, Germany) (Zhang et al., 2017; Wongkom & Jimtaisong, 2017). Thermo

gravimetric analysis was carried out under controlled surroundings of nitrogen and a 30°-600°C temperature range at a rate of 10°C/min.

2.2.10 Functional properties of the extracted dietary fibre

2.2.10.1 Water holding capacity and oil holding capacity

Water holding capacity and oil holding capacity were determined using a slightly modified version of the Ma & Mu (2015) method. 2g of dietary fiber were mixed with 20 ml of either deionized water (for WHC) or vegetable oil (for OHC) and agitated for 30-35 minutes at 25°C. This was followed by centrifugation at 4000 rpm for 10 minutes. The water holding capacity and oil holding capacity of the extracted dietary fiber were expressed as the total weight gained by the dietary fiber (g/g dry weight) and were calculated using the following equation:

$$\text{WHC or OHC (g/g)} = (W_t - W_i) / W_i \quad (4)$$

Where W_t is the final weight of DF which contains water or oil and W_i is the initial weight of DF.

2.2.10.2 Swelling capacity (SC)

Swelling capacity (SC) was analyzed by the method of Daou & Zhang (2012) with a simple modification. The sample (1g) was placed and the initial volume was recorded by using a graduated glass cylinder. Subsequently, 30 ml of phosphate buffer was added (pH at 6.9) and the samples were allowed to swell for 60 min. The swelling capacity of the sample was determined by taking the final volume of the swollen sample and expressed as mL of final volume per g of the initial dry sample.

2.2.10.3 Cation exchange capacity (CEC)

CEC was analyzed by treating the sample with 2M HCl for 48 h and from this 0.5g of wet sample was taken and volume made to 100 ml with 5% NaCl in buffer (pH 6.9) and then filtered. The sample was then incubated and shaken at 37°C for 60 min and titrated and the final results were expressed as meq per g dry initial sample (Gorecka et al., 2000).

2.2.10.4 Emulsifying activity (EA) and emulsion stability (ES)

Sample (5g) was added to deionized water (1:2 ratio) and sonicated for 10 min with 5 mL refined oil. The solution was then centrifuged at 4000 rpm for 5min. Finally, the height of the emulsified layer and the total contents in the tube were evaluated (Abirami et al., 2014).

The EA was calculated as follows:

$$EA(\%) = \frac{\text{Height of the emulsified layer}}{\text{Height of total content}} \times 100 \quad (5)$$

Emulsion stability was evaluated by heating the emulsion at 85°C for 40 min and then followed by centrifugation at 4000 rpm for 5 min. The ES was calculated as follows:

$$ES(\%) = \frac{\text{Height of the emulsified layer after heating}}{\text{Height of emulsified layer before heating}} \times 100 \quad (6)$$

2.2.11 Hypoglycemic activity

2.2.11.1 Glucose adsorption capacity (GAC)

The GAC was analyzed following the method of Peerajit et al. (2012). Each sample of AEDF, UAEDF, and pineapple waste dried powder (1%) was added to 25ml of glucose solution of 1, 10, 50, 100, and 200 mM concentration. The mixture was agitated using a magnetic stirrer and incubated in a water bath at 37°C for 6 h at 300 rpm. Further, centrifugation at 4,000 rpm for 20 min was done and the glucose content in the supernatant was analyzed. Glucose adsorption capacity was estimated using the following formula:

$$GAC (mmol/g) = \frac{(G_1 - G_6)}{(W_s \times V_i)} \quad (7)$$

Where G_1 is the initial glucose concentration in mmol/L; G_6 is the final glucose concentration after 6 h in mmol/L; V_i is the volume of supernatant in ml; W_s is the weight of dietary fiber in g.

2.2.11.2 Glucose dialysis retardation index (GDRI)

GDRI analysis is one of the methods to determine the outcome of a sample for its glucose absorption in the interval of 30-180 min in the human gastric intestinal tract. The GDRI determination was executed by the method of Nsor-Atindana et al. (2012). The DF samples

(0.5g) were added to 25 mL of glucose solution (50 mmol/L) in a dialysis membrane (molecular weight 12,000 Da) bag. Individually every dialysis bag was dialyzed with 100 mL of distilled water (DW) at 37°C under a water bath with a slight rotation of 50 rpm. The glucose assay kit (Sigma GAGO-20) was used to check the glucose content in the dialysate to verify the glucose retardation as a function of time. The absorbance was determined using a spectrophotometer for each sample at 30, 60, 120, and 180 minutes after 1mL of glucose had diffused from the treated sample (pineapple DP, AEDF, UAE DF) and control (without addition of sample).

The GDRI was expressed using the following equation:

$$GDRI = 100 - [(G_{ts}/G_c) \times 100] \quad (8)$$

Where, G_{ts} is the total glucose diffused from the treated sample; G_c is the total glucose diffused from the control sample.

2.2.12 Statistical analysis

SPSS 16.0 was used for the statistical analysis of data. Evaluation of the means was done by investigation of variance (ANOVA) with Duncan's multiple range tests to identify the significant differences ($p < 0.05$).

2.3 Results and Discussion

2.3.1 Optimization of UAE method

Optimization of a single factor was conducted using the extraction yield (%) of DF as the dependent index to identify the ranges of the extraction of independent variables (Table 2.1). As shown in Fig 2.1(a,b,c) the extraction yield of DF initially raised to the highest level as the time was increased from 10 to 30 min, and then declined as the amplitude was increased from 20 to 50%. This implied that an increase in amplitude reduced the particle size and enhanced the specific area of the sample surface, thus speeding up the mass transfer of intracellular substances. Excessive amplitude, on the other hand, may cause the degeneration of DF. As a result, the optimal amplitude was determined to be 46.90%. The effect of sonication time on DF yield (Fig 2.1 [a,b,c]) revealed that as the sonication time increased from 10 to 30 min, the extraction yield of DF increased relatively quickly, indicating that a longer sonication time could increase the particle size of extracted DF and increased the mass transfer rate of DF. In this study, the optimum sonication time was set at 22.35 min and the extraction yield of DF

increased significantly as the solid: liquid ratio (dried powder (DP): 0.5 % NaOH solution) increased from 10% to 25%, after which it declined marginally (Fig 2.1).

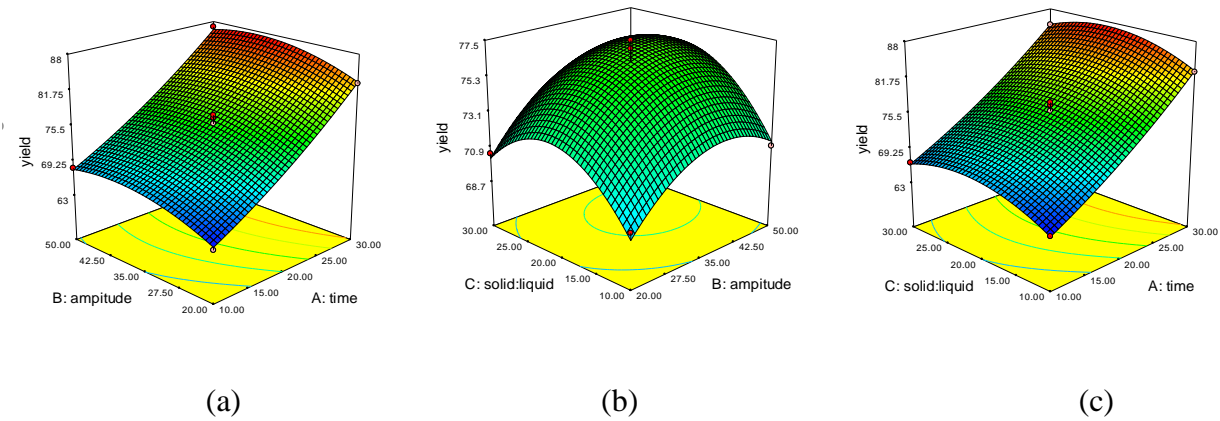


Fig 2.1: The effects of (a) Sonication time (min), (b) Amplitude (%), and (c) Solid: liquid ratio (g/mL) on the extraction yield of dietary fiber (DF)

The influence of a constant hydrolysis temperature has the potential to affect the rate of mass transfer and the density of the liquid solvent simultaneously (Mao et al., 2021). The activity of the alkali solution and molecular movement might be increased at a high enough temperature, speeding up the dissolving of DF. Higher hydrolysis temperatures, on the other hand, may limit yield activity and promote DF degradation. As a result, a constant hydrolysis temperature of 60° C was adopted (Hu & Zhao, 2018).

Table 2.1: Ranges of independent variables with their corresponding levels

Coded variable levels	Independent variables	Levels		
		-1	0	+1
A	Sonication time (min)	10	20	30
B	Amplitude (%)	20	35	50
C	Solid:liquid ratio (g/mL)	10	20	30

2.3.2 Determination of significance factors

The DF extraction utilizing ultrasonication was done with a BBD design and created three independent variables, with the total yield of DF as the dependent variables (Table 2.2). The model's fit was expressed by the R^2 value (0.93), and the statistical significance level was verified by the F-test. The analysis of variance regression model was determined to be significant ($p > 0.05$), indicating that the model is legitimate (Table 2.3). The DF extraction yield is explained by the quadratic regression equation:

$$Yield = +76.49 + 9.42 * A + 1.69 * B + 1.15 * C - 0.05 * A * B - 0.12 * A * C + 0.42 * B * C + 1.20 * A^2 - 2.25 * B^2 - 3.08 * C^2 \quad (9)$$

Where, A = sonication time (min); B = amplitude (%); C = solid:liquid (g/mL)

Table 2.2: Box-Behnkan design for extraction of dietary fiber (DF) using ultrasound-assisted extraction (UAE)

Run	Time (min)	Amplitude (%)	Solid: Liquid (g/mL)	Yield of DF
	A	B	C	%
1	20	50	10	71
2	10	20	20	63.8
3	20	35	20	76
4	20	35	20	76
5	30	35	30	85
6	30	35	10	82.8
7	20	20	10	69.17
8	20	35	20	77.5
9	30	20	20	83
10	30	50	20	87
11	20	35	20	75.95
12	10	35	30	66.67
13	10	50	20	68

14	20	35	20	77
15	10	35	10	64
16	20	50	30	74
17	20	20	30	70.5

Table 2.3: ANOVA for response surface quadratic model for the yield of dietary fiber (DF)

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	Significant level
Model	811.39	9	90.15	193.51	< 0.0001	***
A	709.33	1	709.33	1522.54	< 0.0001	***
B	22.88	1	22.88	49.12	0.0002	**
C	10.58	1	10.58	22.71	0.002	**
AB	1.00E-02	1	1.00E-02	0.021	0.8877	*
AC	0.055	1	0.055	0.12	0.7407	*
BC	0.7	1	0.7	1.5	0.2608	*
A ²	6.11	1	6.11	13.12	0.0085	**
B ²	21.22	1	21.22	45.55	0.0003	**
C ²	39.88	1	39.88	85.6	< 0.0001	***
Residual	3.26	7	0.47	-	-	-
Lack of Fit	1.21	3	0.4	0.79	0.5611	not significant
Pure Error	2.05	4	0.51			
Cor Total	814.65	16				
R ² = 0.93						

A = Sonication time (min), B = Solvent: solute (mL/g), C = Ultrasound amplitude (%)

* Not significant (p > 0.05), ** Significant different (p < 0.05), *** Extremely significant different (p < 0.01)

2.3.3 Optimum conditions for extraction

The optimum conditions for UAEDF has been shown in Table 2.4. The optimum yield of DF was obtained at 22.35 min of sonication time with solid: liquid ratio of 27.5 g/mL, and

ultrasonic amplitude of 46.90%. The optimum conditions for extraction finally provided the value for the predicted extraction yield of DF which was 86.67%. This predicted value matched well with the experimental value with a desirability of 0.93.

Table 2.4: Validation of optimum extraction conditions

Factors (independent variables)	Optimum conditions	Predicted value, DF yield (%)	Experimental value, DF yield (%)	Relative deviation (%)
Sonication time (min)	22.35	86.23	86.67	0.51
Amplitude (%)	46.9			
Solid:liquid (g/mL)	27.5			
Desirability = 0.93				

2.3.4 Chemical composition of pineapple waste and extracted dietary fiber

The chemical composition of Queen pineapple waste DP and extracted AEDF, UAE DF are presented in Table 2.5 on the dry basis (DB). Total dietary fiber (TDF) content of UAE DF (86.67g/100g) was significantly higher as compared to Queen pineapple waste DP and AEDF.

Table 2.5: Chemical composition of alkaline extracted dietary fiber (AEDF) and ultrasound-assisted extracted dietary fiber (UAEDF)

Chemical composition (g/100g)	Pineapple waste DP	AEDF	UAEDF
Moisture content	15.43 ± 0.12 ^b	10.83 ± 0.10 ^b	10.04 ± 0.07 ^b
Crude protein	3.3 ± 0.13 ^b	1.6 ± 0.12 ^b	1.21 ± 0.04 ^a
Crude fat	1.84 ± 0.03 ^a	0.55 ± 0.03 ^a	0.52 ± 0.59 ^d
Ash	4.4 ± 0.05 ^a	11.23 ± 0.11 ^b	10.16 ± 0.18 ^c
Crude fiber	58.32 ± 0.41 ^d	-	-
Carbohydrate	16.71 ± 0.23 ^c	-	-
TDF	-	64.43 ± 0.41 ^b	80.11 ± 0.70 ^c
IDF	-	62.89 ± 0.21 ^a	75.43 ± 0.46 ^b
SDF	-	1.54 ± 0.38 ^b	4.68 ± 0.18 ^a

(Values represent mean± SD;)

*AEDF- alkali extracted dietary fiber UAEDF- ultrasound-assisted extracted dietary fiber; TDF-total DF, IDF- Insoluble dietary fiber, SDF- Soluble dietary fiber

Moreover, it was found to be higher than that of yellow passion fruit waste (71.79g/100g), lemon peel (70.76g/100g) and mango processing waste (74g/100g) (Begum & Deka, 2020). Less amount of fat was found to be present in UAE DF (0.23g/100g). Protein content of UAE DF was found to be less than that of AEDF and DP and can be attributed to the protein denaturation by the high intensity ultrasound (Begum & Deka, 2020).

The ash content of AEDF and UEDF decreased from the pineapple waste dried powder (DP) which could be evident from the decrease in mineral contents shown in Table 2.6 (Begum and Deka 2019).

Table 2.6: Mineral content of alkaline extracted dietary fiber (AEDF) and ultrasound-assisted extracted dietary fiber (UAEDF)

Mineral (mg/100g)	UAEDF	AEDF	Pineapple waste DP
Cu	46.60±0.06 ^c	60.11±0.02 ^a	95.62±0.24 ^b
K	671.96±0.31 ^b	476.89±0.03 ^a	466.72±0.03 ^a
Mg	29.92±0.06 ^c	34.26±0.04 ^a	41.21±0.11 ^b
Fe	16.00±0.02 ^a	19.10±0.10 ^b	25.11±0.11 ^b

(Values represent mean± SD; n=3. Mean with different superscript letters in the same row represent a significant difference at p<0.05)

*AEDF- alkali extracted dietary fiber UAEDF- ultrasound-assisted extracted dietary fiber; TDF-total DF, IDF- Insoluble dietary fiber, SDF- Soluble dietary fiber

2.3.5 FTIR analysis of extracted dietary fibre

FTIR spectroscopy is an analytical method for characteristic organic functional groups in polysaccharides which detected within the wavelength of 400-4000 cm⁻¹. The functional groups and bonding configurations of the FT-IR spectra of UAEDF, and AEDF are shown in Fig. 2.2. While some characteristic bands differed between the overall spectral profiles of AEDF and UAEDF. The strong and broad absorption at 3408 cm⁻¹ corresponds to OH group

stretching due to inter- or intra-molecular hydrogen bonds. This OH vibration mainly arises from hemicellulose (xylose, mannose, arabinose, galactose) and pectin (galacturonic acid) (Zhang et al., 2017). Weak absorption bands at 2810 and 1628 cm^{-1} , originating from CH stretching, indicate the presence of methylene polysaccharide structures (Zhang et al., 2017). The carbonyl group from GalA units (CH, CH₂, CH₃) pectin, and methoxy group of glucose uronic acid hemicellulose can be studied from the band stretching of 1255-1837 cm^{-1} (Zhang et al., 2017).

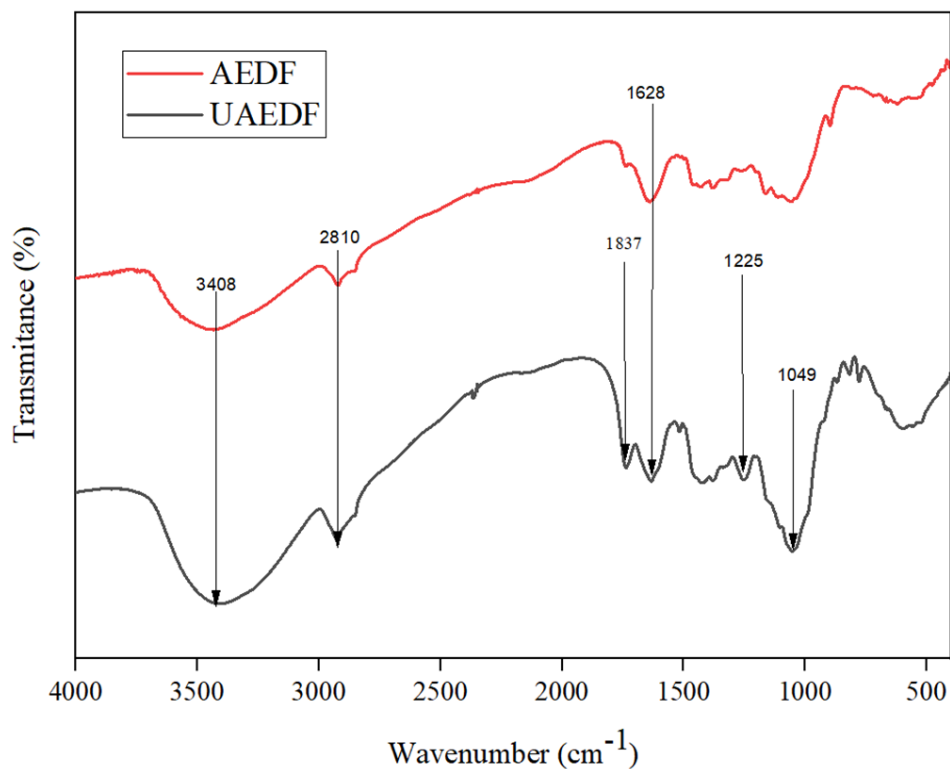
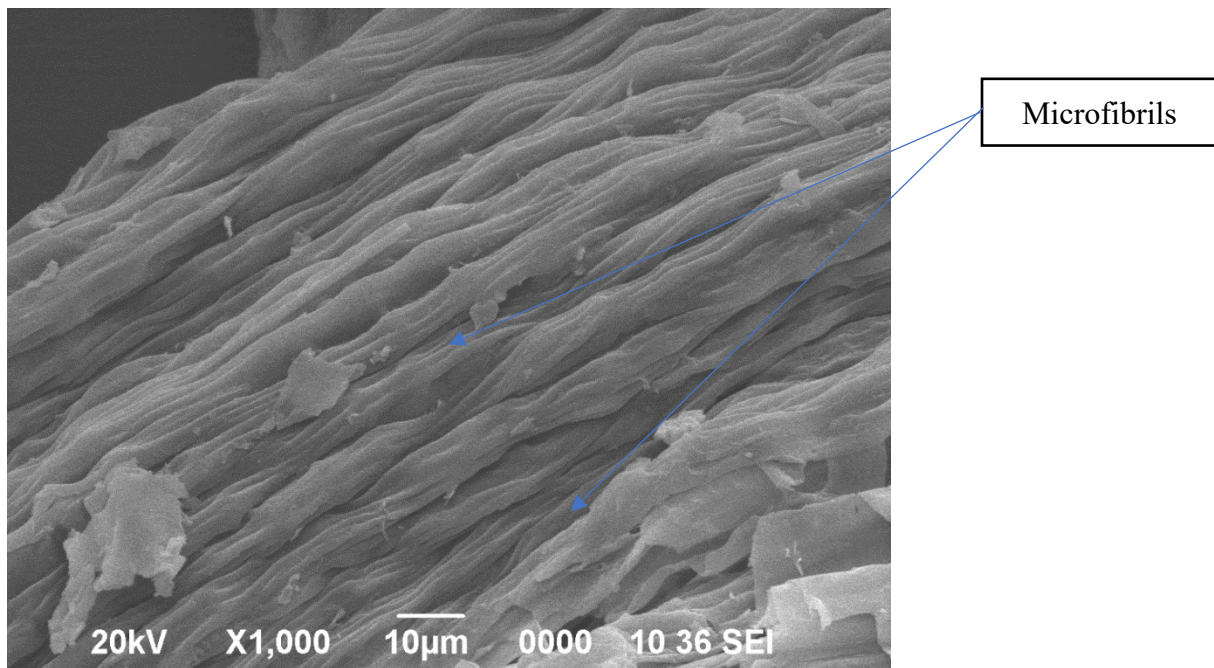


Fig 2.2: FT-IR spectrum of ultrasound-assisted extracted dietary fiber (UAEDF), and alkaline extracted dietary fiber (AEDF)

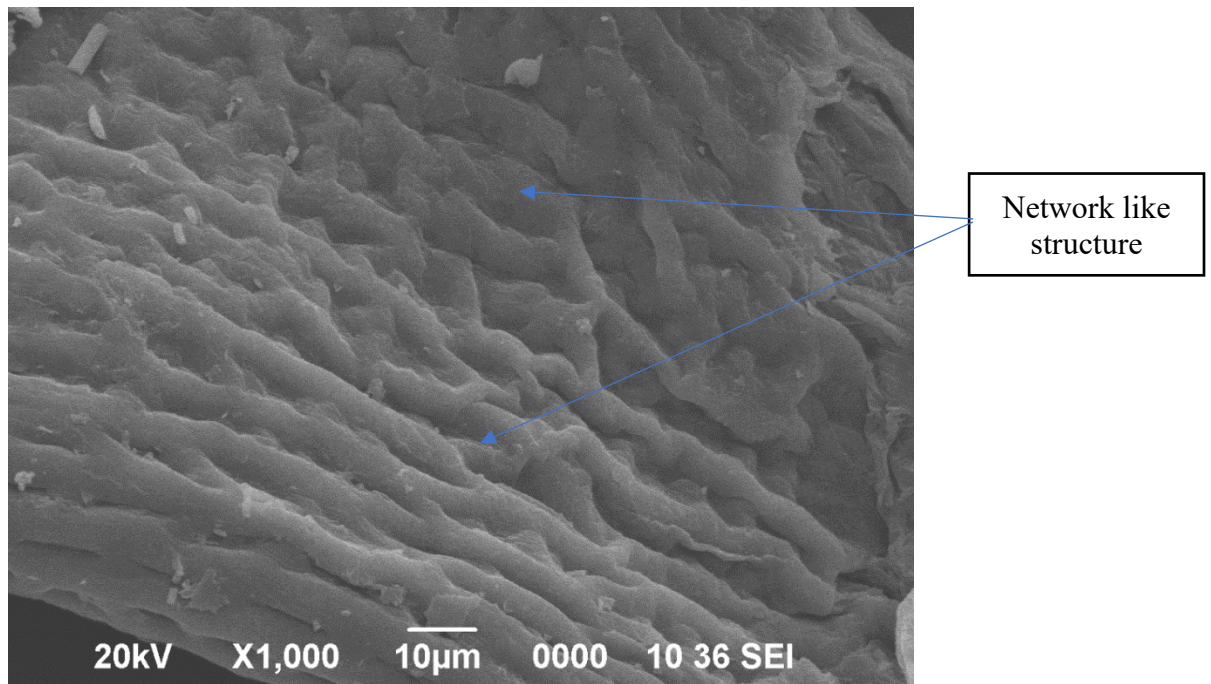
The band peaks at 1650 cm^{-1} correspond to aromatic hydrocarbons of lignin and CH₂ stretching of cellulose, respectively. Additionally, the peak at 1049 cm^{-1} represents the CO stretch bond, indicating the breakdown of oligosaccharides from dietary fiber (Bockuviene & Sereikaite, 2019). The presence of these stretching bands and peaks confirms the typical functional groups of polysaccharides in the extracted dietary fiber from pineapple waste in AEDF and UAEDF.

2.3.6 Scanning electron microscope analysis of the extracted dietary fiber

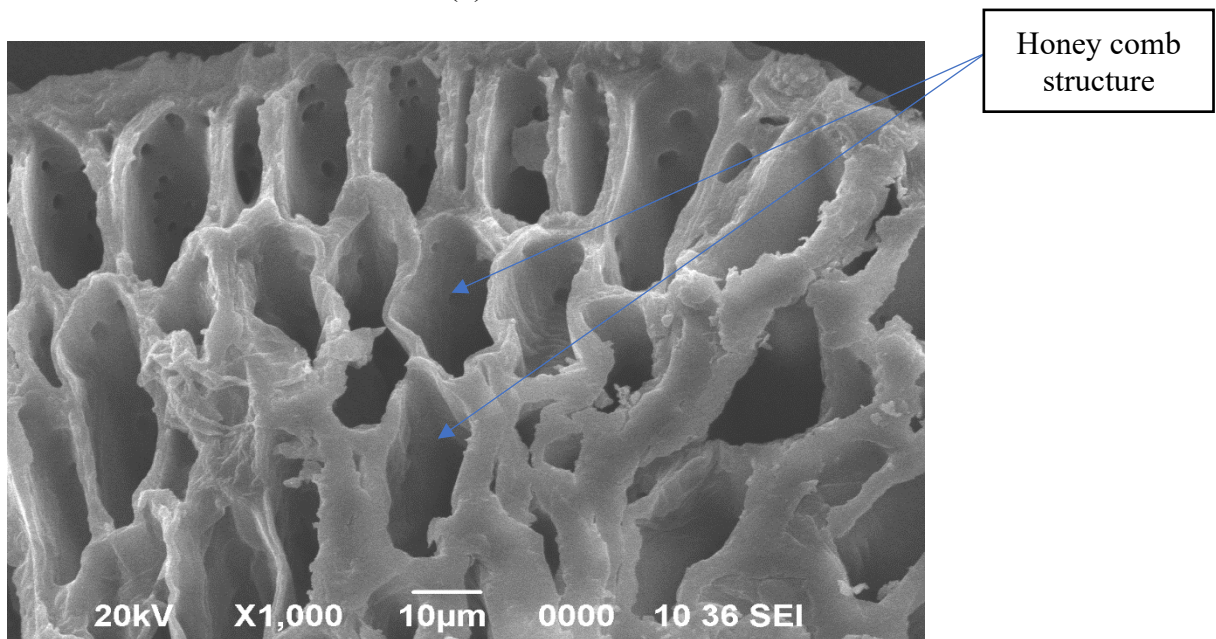
The structural analysis of pineapple waste DP, UAE DF, and AEDF are represented in Fig 2.3 (a,b,c). Pineapple waste DP exhibited a compact texture, while the surface of AEDF displayed a fibrous network-like structure. In contrast, the surface of UAEDF was shown much looser and more porous structures. These differences in structural analysis by SEM explained that ultrasonication treatment disrupts the interaction between the solute and solvent, resulting in the honeycomb structure of UAEDF. Begum & Deka (2019) reported that a well-alienated fibrous network structure was perceived in the case of AEDF and a distinctive honeycomb arrangement was found in the case of UAEDF. The obtained results could be accompanied by the ultrasonication treatment which indicates the formation of large pores in the outer cell wall and hence untying the complex bond between the microfibrils of DF.



(a)



(b)



(c)

Fig 2.3: SEM micrograph of (a) Pineapple waste dried powder (DP), (b) alkaline extracted dietary fiber (AEDF), and (c) ultrasound-assisted extracted dietary fiber (UAEDF)

2.3.7 XRD analysis of the extracted dietary fiber

XRD analysis was conducted to evaluate the changes in crystallinity and amorphous region of DF and thus distinguish the cluster region of the AEDF and UAE DF. XRD derived the patterns of both the AEDF and UAE DF (Fig 2.4). The changes in crystallinity of UAE DF, and AEDF were evaluated from an X-ray diffractogram. The analysis of both patterns of AEDF and UAE DF revealed characteristics major peaks at 22.17° and 37.89° whereas the non-crystalline minor peaks at 18.19° . These results of AEDF and UAE DF evinced the co-existence of crystalline and amorphous region, which led to a typical cellulose crystallinity structure (Zhang et al., 2017). Similarity in the outline of UAE DF and AEDF specifies that ultrasonication changed the type of crystallinity. The degree of crystallinity of UAE DF (30.21%) was significantly ($p < 0.05$) higher than that of AEDF (27.54%) because of the ultrasonic treatment of the DF which made more loosen tissue.

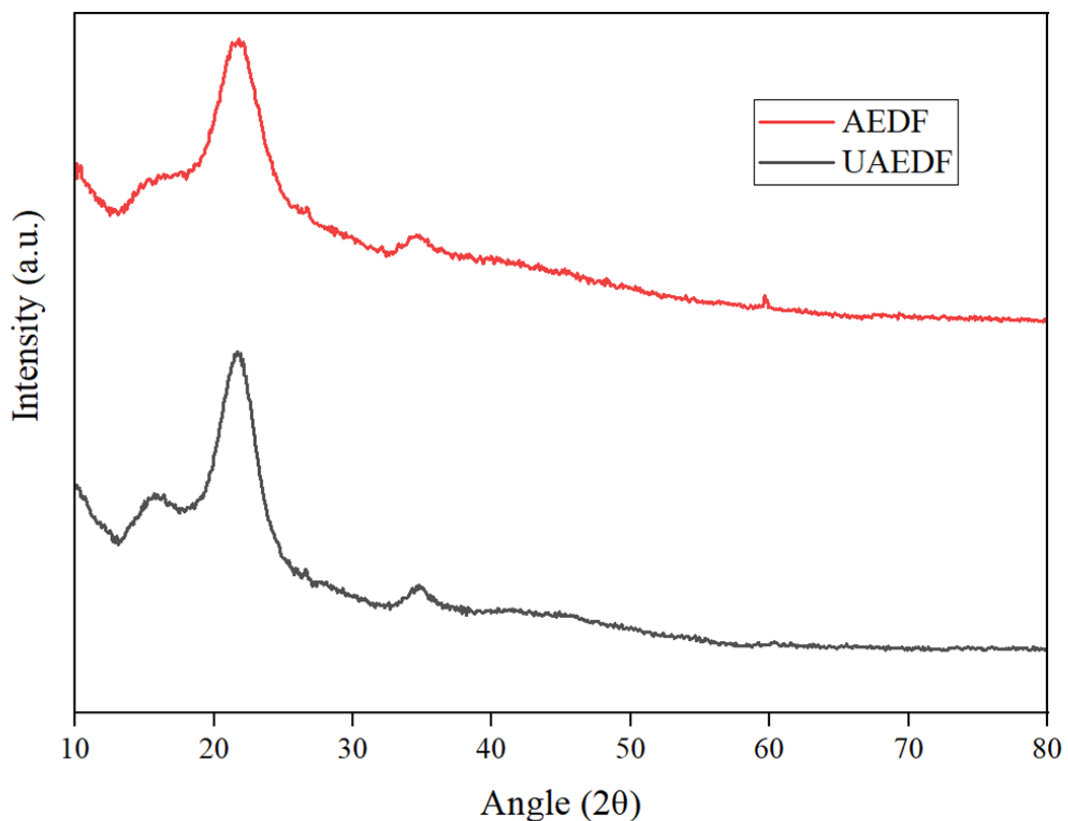


Fig 2.4: XRD graph of alkaline extracted dietary fiber (AEDF), and ultrasound-assisted extracted dietary fiber (UAEDF)

According to the findings, ultrasonication treatment substantially disrupted the cell structure of UAE DF, which makes hydrolysis of cellulose and hemicellulose part of DF into strong ordered crystalline structure from less ordered crystalline or even disordered amorphous structure. Thus, characteristics crystalline structure was found in UAE DF and the interaction between the DF molecules weakens due to the removal of amorphous structure.

2.3.8 Thermo-gravimetric analysis (TGA) of extracted dietary fibre

The thermal stability of a polysaccharide is one of the important parameters during the thermal processing of food unit operations such as baking. TGA was performed to differentiate the changes in the thermal behaviors and thermal stability of UAEDF, and AEDF. TGA was done at a temperature of 0-700°C range (Fig 2.5).

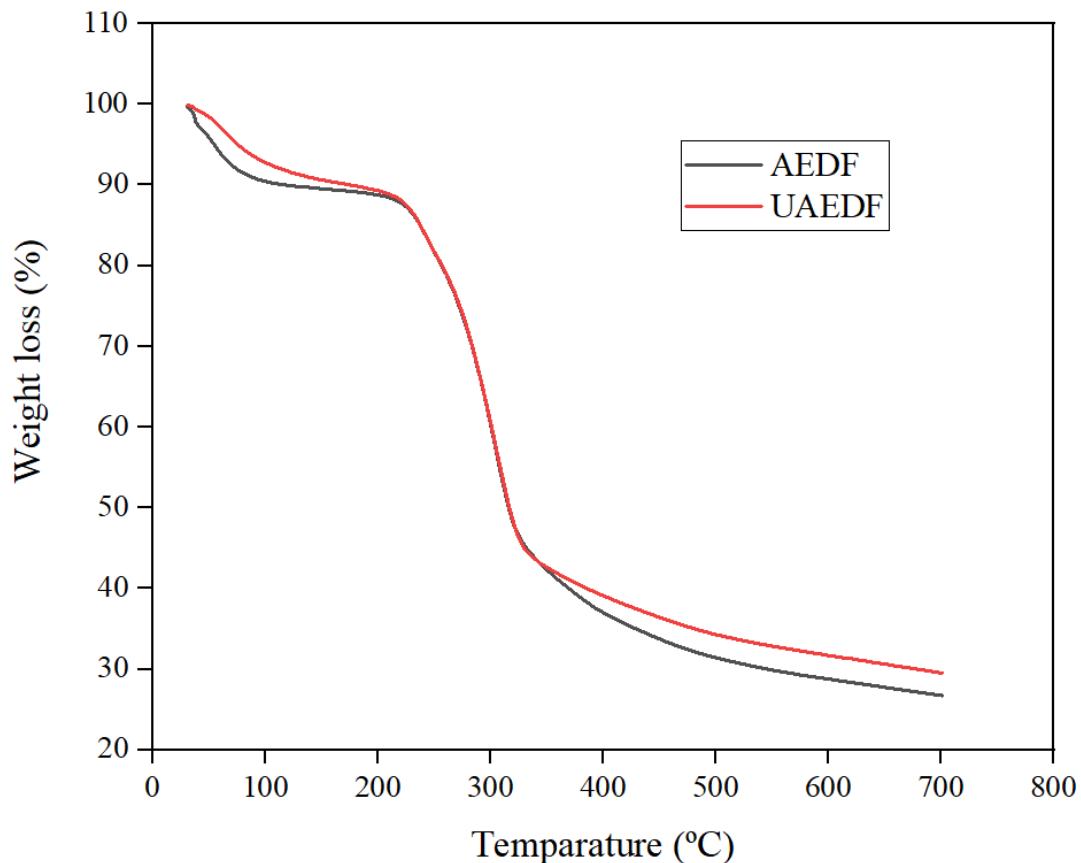


Fig 2.5: TGA analysis of alkaline extracted dietary fiber (AEDF), and ultrasound-assisted extracted dietary fiber (UAEDF)

Results evinced changes in both DF at the different temperature ranges (40-200°C, 200-350°C and 350 up to 700°C) and DF was observed to have different degradation kinetics at these temperature ranges and was impactful. The results revealed that the evaporation of absorbed water took place during the first temperature range of 40-200°C (Ma & Mu, 2016). Studies have shown that hemicellulose is degraded at 210 to 350°C very rapidly (Moriana et al., 2011; Yang et al., 2007), while the degradation of cellulose started at 315°C and continued up to 400°C (Liu et. al., 2021). Due to the presence of different functional groups in DF viz., lignin, cellulose, and hemicellulose variable thermal breakdown temperatures have been observed. According to the results, UAEDF absorbed more water than AEDF. The temperature ranges show that hemicellulose depolymerization and cellulose breakdown occurred (Begum & Deka, 2019). The polysaccharide pyrolytic degradation of hemicelluloses and pectic polysaccharides contained in the DF can be related to the relative strength intensity of the peaks (Carrier et al., 2011). The thermal degradation of char especially when the pyrolytic temperature ascended to the 2nd temperature range (200-350°C), the changes in mass were very slow. As per the temperature range investigation (40-200°C, 200-350°C and 325 up to 700°C), the weight loss of ultrasound-assisted extracted dietary fiber was less than alkaline extracted dietary fiber, signifying that ultrasound-assisted extracted dietary fiber had greater thermal stability than alkaline extracted dietary fiber and pineapple waste dried powder.

2.3.9 Functional properties of extracted dietary fibre

The differences in the physicochemical and functional properties of extracted DF were observed (Table 2.7) in which the WHC, OHC, and SC of UAEDF were 12.72 g/g, 5.22 g/g, and 7.38 mL/g samples, respectively. The capacity of any compound to hold moisture content includes hydrodynamic, physical entrapment, and moisture linkage under some controlled conditions representing the water holding capacity (Alfredo et al., 2009). It was found to have a significantly higher value as compared to pineapple waste dried powder and AEDF with UAEDF. The initial WHC of AEDF and pineapple waste dried powder was 8.64±0.06 and 6.38±0.07 g/g, respectively which is more than the water holding capacity of DF from the banana pseudo stem (4.71±0.31 g/g), malt bagasse (3.68± 0.08 g/g), oat hull (2.13±0.11 g/g), passion fiber (7.2 g/g) and rice hull (2.58 ± 0.28 g/g) (Wang et al., 2015) but lower than that of chia fiber (15.41 g/g) (Alfredo et al., 2009). Moreover, the WHC of ultrasound-assisted extracted dietary fiber (12.72±0.167 g/g) was significantly greater ($p < 0.05$) than that of AEDF (8.64 ± 0.09g/g). Reduced particle size, porosity, surface, and microstructure of DF also led to

an increase in WHC. Water interacts with DF in such a way that either water is held in capillary structure because of the strength of surface tension or water is embraced in hydrogen bond to create the dipole moment (Begum & Deka, 2019).

The OHC of AEDF and UAEDF were 3.53 ± 0.12 g/g and 5.22 ± 0.10 g/g, respectively. The increase in OHC of UAEDF can be attributed to the moderately loose texture due to the ultrasonication treatment as shown by the SEM analysis. A comparative study of the SC of UAEDF from pineapple waste dried powder with the SC of AEDF revealed that the SC of UAEDF (7.38 ± 0.28 mL/g) was higher than that of AEDF (4.23 ± 0.10 mL/g).

Table 2.7: Functional properties of Pineapple waste dried powder (DP), alkaline extracted dietary fiber (AEDF), and ultrasound-assisted extracted dietary fiber (UAEDF)

Functional properties	Pineapple waste DP	AEDF	UAEDF
WHC(g/g)	6.38 ± 0.07^a	8.64 ± 0.09^a	12.72 ± 0.17^{ab}
OHC (g/g)	1.18 ± 0.17^{bc}	3.53 ± 0.012^{ab}	5.22 ± 0.10^a
SC (mL/g)	1.68 ± 0.28^d	4.23 ± 0.10^{ab}	7.38 ± 0.28^{ab}
EA (%)	18.06 ± 1.78^e	31.89 ± 1.88^d	41.89 ± 1.42^{cd}
ES (%)	8.5 ± 0.07^a	17.8 ± 1.3^c	29.5 ± 2.1^d
CEC (mM/g)	1.27 ± 0.15^b	3.98 ± 0.14^{ab}	6.34 ± 0.62^c

(Values represent mean \pm SD; n=3. Mean with different superscript letters in the same row represent a significant difference at $p < 0.05$.)

*WHC-water holding capacity; OHC-oil holding capacity; SC- Swelling Capacity; EA- Emulsion activity; ES- Emulsion Stability, CEC- Cation exchange Capacity

The emulsion activity (EA) value of extracted DF was significantly reduced ($p < 0.05$) using the UAE method. The stability index of EA values was less than 50% (Table 2.6), which indicated that UAEDF is not a good emulsifier and our results are in line with several authors who have reported low EA values of DF (Abdul-Hamid & Luan, 2000; Daou & Zhang, 2011). A bad emulsion's stability index is just 50%, while a good emulsion is larger than 94% (Wang & Kinsella, 1976). Lower protein content in both AEDF and UAEDF samples may have contributed to the lower EA values in this study (Table 2.6). The cation exchange capacity (CEC) of the Queen pineapple waste extracted DF was significantly ($p < 0.05$) enhanced by

the UAE method. The existence of phenol groups in insoluble DF (lignin) determines the CEC of fibre (Liu et al., 2021). The UAEDF has a greater CEC value which could be due to its higher IDF content (Table 2.6). The CEC acts to be connected to the fibre fluid permeability, which may be enhanced due to a more porous surface and less ordered fibre structure which raised the CEC of the DF (Fig 2.3 a,b,c). The increased fluid permeability into DF was also demonstrated by the higher WHC and SC values of UAEDF (Table 2.6). Ultrasonic treatment also assisted to improve the water holding capacity, oil holding capacity, and swelling capacity of DF in addition to the degree of polymerization. Wang et al. (2016) showed a similar effect in the case of ultrasound-treated DF. The variation in findings of functional properties of extracted DF results can be attributed to the different extraction methods (UAE and AE) from the Queen pineapple waste sample.

2.3.10 Glucose adsorption capacity

The GAC of pineapple waste dried powder, AEDF, and UAEDF at different glucose concentrations (1mM, 10mM, 50mM, 100mM, and 200mM) have been shown in Table 2.8. All the extracted DF could adsorb glucose efficiently and the GAC was directly related to the glucose concentration in a direct proportional way.

Table 2.8: Glucose adsorption capacity (GAC mmol/g) of Pineapple waste dried powder (DP), alkaline extracted dietary fiber (AEDF), and ultrasound-assisted extracted dietary fiber (UAEDF)

Glucose Concentration (mM)	Pineapple waste DP	AEDF	UAEDF
1	3.58	5.35	11.12
10	6.83	8.84	20.94
50	13.69	16.48	40.38
100	20.84	25.84	59.87
200	27.47	33.09	80.52

The GAC of Queen pineapple peel waste extracted DF increased significantly as a result of the increasing number of cavities or voids on the surface of the DF due to the treatment. One of the most important variables in determining DF's ability to adsorb lipophilic components is its ability to bind to glucose. The amount of glucose molecules entrapped inside the fibre network increases as the porosity and specific surface area of DF increases, resulting in a greater GAC. (Chau et al., 2007; Zheng et al., 2021). There are several similarities in the effects of extracted DF on glucose. The increased GAC of UAEDF could be related to more fibre binding sites being exposed.

2.3.11 Determination of glucose dialysis retardation index

GDRI is an *in vitro* method for analyzing the effect of samples on the delay in glucose absorption in the gastric intestinal tract. Analysis of variations in glucose diffusion in dialysate with the addition of pineapple waste dried powder, AEDF, UAEDF along with the control has been presented in Table 2.9. With the increase of time from 30 to 180 min, the concentration of glucose in the dialysis membrane with the samples viz., pineapple dried powder, AEDF and UAEDF elevated from 15.16-168.53 $\mu\text{g/mL}$, 13.38-142.38 $\mu\text{g/mL}$, 12.86-135.93 $\mu\text{g/mL}$, respectively. In comparison with the control, the pineapple waste dried powder, AEDF, and UAEDF significantly decreased the quantity of glucose diffused in dialysate during 30 to 180 min time period.

Table 2.9: Glucose dialysis retardation index (GDRI) Pineapple waste dried powder (DP), alkaline extracted dietary fiber (AEDF), and ultrasound-assisted extracted dietary fiber (UAEDF)

Glucose in dialysate (μmol)				
Sample	30 min	60min	120 min	180min
Control	15.72	51.28	148.76	190.6
DP	15.16	40.87	139.37	168.53
AEDF	13.38	26.28	105.29	142.38
UAE DF	12.86	30.32	110.28	135.93

DP- Dried powder, AEDF- alkaline extracted dietary fiber, UAE DF- ultrasound-assisted extracted dietary fiber;

Control means dialysis bag without sample containing glucose solution with distilled water only

The retardation index in diffusion of glucose among the different samples viz., pineapple dried powder, AEDF and UAEDF is expressed by the GDRI value at the time period of 30, 60, 120, 180 min (Table 2.9). At 30 min GDRI of the pineapple peel waste dried powder, AEDF, and UAEDF were found to be 3.53%, 14.87%, and 18.13%, respectively. With the increase in time from 30 to 180min the GDRI values of pineapple peel waste dried powder, AEDF, and UAEDF were 3.53-11.58%, 14.87-25.3%, 18.13-28.68%, respectively. The results evinced that the UAEDF delayed the diffusion of glucose out of the dialysis membrane effectively than AEDF and pineapple waste dried powder. Similar GDRI values were also observed in citrus peel extracted DF (Abirami et al., 2014) and millet bran DF (Zheng et al., 2021).

2.4 Conclusion

Results of the present investigation have evidenced that the Queen pineapple waste is remarkably high in DF and can be utilized as a dietary supplement. The optimized parameters of DF extraction by ultrasonic method revealed a solid:liquid ratio of 27.5 g/mL, an amplitude of 46.90%, extraction period of 22.35 min at constant hydrolysis temperature (60°C). Ultrasonic extraction evinced a better yield (86.67%) of DF compared to alkaline extraction (64.43%) and the former also revealed better functional properties than the latter. FT-IR, XRD, SEM, and TGA of the extracted UAEDF results evinced a typical polysaccharide structure with all the functional groups, semi-crystalline pattern, honeycomb structure with a rough surface, and good thermal stability as compared to the AEDF and dried powder, respectively. UAE increased the surface porosity and semi-crystallinity of the DF which resulted improved GAC and GDRI. The lower emulsifying capacity and emulsion stability was observed due to the decrease in protein content. Changes in UAEDF physicochemical and functional properties were attributed to a more porous structure in the surface and exposure of DF edging sites due to semi-crystallinity, and chemical composition alterations. Thus, UAEDF can be used as a useful source of natural dietary fiber and as a supplement to improve the functional properties of foods. Therefore, it may be a noble DF for developing different food products e.g., bakery products (viz., whole grain bread, noodles, biscuits, and steamed bread.), meat products (viz., sausages, meatballs, surimi, meat emulsion), dairy-based products (viz., yogurt, milk-based beverages) and also as an additive for product development.

Bibliography:

1. Abdul-Hamid, A., and Luan, Y. S. (2000). Functional properties of dietary fibre prepared from defatted rice bran. *Food Chemistry*, 68(1), 15-19.
2. Abirami, A., Nagarani, G., and Siddhuraju, P. (2014). Measurement of functional properties and health-promoting aspects-glucose retardation index of peel, pulp, and peel fiber from *Citrus hystrix* and *Citrus maxima*. *Bioactive Carbohydrates and Dietary Fibre*, 4(1), 16-26.
3. Al-Dhabi, N.A., and Ponmurugan, K. (2020). Microwave-assisted extraction and characterization of polysaccharide from waste Jamun fruit seeds, *International Journal of Biological Macromolecules*, 152, 1157-1163
4. Alfredo, V. O., Gabriel, R. R., Luis, C. G., and David, B. A. (2009). Physicochemical properties of a fibrous fraction from chia (*Salvia hispanica* L.). *LWT-Food Science and Technology*, 42(1), 168-173.
5. Begum, Y. A., and Deka, S. C. (2019). Effect of processing on structural, thermal, and physicochemical properties of dietary fiber of culinary banana bracts. *Journal of Food Processing and Preservation*, 43(12), e14256.
6. Begum, Y. A., Chakraborty, S., and Deka, S. C. (2020). Bread fortified with dietary fibre extracted from culinary banana bract: its quality attributes and in vitro starch digestibility. *International Journal of Food Science & Technology*, 55(6), 2359-2369.
7. Bockuviene, A., and Sereikaite, J. (2019). Preparation and characterisation of novel water-soluble β -carotene-chitooligosaccharides complexes. *Carbohydrate Polymers*, 225, 115226.
8. Carrier, M., Loppinet-Serani, A., Denux, D., Lasnier, J. M., Ham-Pichavant, F., Cansell, F., and Aymonier, C. (2011). Thermogravimetric analysis as a new method to determine the lignocellulosic composition of biomass. *Biomass and Bioenergy*, 35(1), 298-307.
9. Chau, C. F., Chen, C. H., and Lin, C. Y. (2004). Insoluble fiber-rich fractions derived from *Averrhoa carambola*: hypoglycemic effects determined by in vitro methods. *LWT-Food Science and Technology*, 37(3), 331-335.
10. Chau, C. F., Wang, Y. T., and Wen, Y. L. (2007). Different micronization methods significantly improve the functionality of carrot insoluble fibre. *Food Chemistry*, 100(4), 1402-1408.
11. Chen, H., Zhao, C., Hussain, J. Li, Yan, S., and Wang, Q. (2018). Effects of extrusion on structural and physicochemical properties of soluble dietary fiber from nodes of lotus root, *LWT- Food Science Technology*, 93, 204-211.

12. Daou, C., and Zhang, H. (2011). Physico-chemical properties and antioxidant activities of dietary fiber derived from defatted rice bran. *Advance Journal of Food Science and Technology*, 3(5), 339-347.
13. Daou, C., and Zhang, H. (2012). Study on functional properties of physically modified dietary fibres derived from defatted rice bran. *Journal of Agricultural Science*, 4(9), 85.
14. Das, U., Bhattacharyya, R. K., Sen, D., Bhattacharjee, P., and Choudhury, P. (2021). Organic Pineapple Production Technology in Tripura-The Lone AEZ for Fruits in North East India. *International Journal of Agriculture, Environment and Biotechnology*, 14(2), 149-158.
15. Fan, T., Hu, J., Fu, L., and Zhang, L. (2015). Optimization of enzymolysis-ultrasonic assisted extraction of polysaccharides from *Momordica charabtia* L. by response surface methodology, *Carbohydrate Polymers*, 115, 701-706.
16. FAO (2020). Medium-Term Outlook: Prospects for Global Production and Trade in Bananas and Tropical Fruits 2019 to 2028, Rome.
17. Gallagher, A. M., Flatt, P. R., Duffy, G. A. W. Y., and Abdel-Wahab, Y. H. A. (2003). The effects of traditional antidiabetic plants on in vitro glucose diffusion. *Nutrition Research*, 23(3), 413-424.
18. Gayas, B., Kayas, G., and Singh, A., (2020). Ultrasound assisted extraction of apricot kernel oil: effect on physicochemical, morphological characteristics, and fatty acid composition, *Acta Alimentaria*, 49 (1) 23-31.
19. Gorecka, D., Lampart-Szczapa, E., Janitz, W., and Sokolowska, B. (2000). Composition of fractional and functional properties of dietary fiber of lupines (*L. luteus* and *L. albus*). *Food/Nahrung*, 44(4), 229-232.
20. Hadidi, M., Amoli, P. I., Jelyani, A. Z., Hasiri, Z., Rouhafza, A., Ibarz, A., ... and Tabrizi, S. T. (2020). Polysaccharides from pineapple core as a canning by-product: Extraction optimization, chemical structure, antioxidant and functional properties. *International Journal of Biological Macromolecules*, 163, 2357-2364.
21. Hadidi, M., Khaksar, F.B., Pagan, J., and Ibarz, A. (2020). Application of Ultrasound-Ultrafiltration Assisted Alkaline Isoelectric Precipitation (UUAaip) technique for producing alfalfa protein isolate for human consumption: optimization, comparison, physicochemical, and functional properties, *Food Research International*, 130, 108907.
22. Hu, H., and Zhao, Q. (2018). Optimization extraction and functional properties of soluble dietary fiber from pineapple pomace obtained by shear homogenization-assisted extraction. *RSC Advances*, 8(72), 41117-41130.

23. Liu, Y., Zhang, H., Yi, C., Quan, K., and Lin, B. (2021). Chemical composition, structure, physicochemical and functional properties of rice bran dietary fiber modified by cellulase treatment. *Food Chemistry*, 342, 128352.
24. Luo, X., Wang, Q., Fang, D., Zhuang, W., Chen, C., and Zheng, Y. (2018). Modification of insoluble dietary fibers from bamboo shoot shell: structural characterization and functional properties, *International Journal of Biological Macromolecules*, 120, 1461-1467.
25. Luo, X., Wang, Q., Zheng, B., Lin, L., and Chen, B. (2017). Hydration properties and binding capacities of dietary fibers from bamboo shoot shell and its hypolipidemic effects in mice, *Food and Chemical Toxicology*, 109, 1003-1009.
26. Ma, M., and Mu, T. (2016). Modification of deoiled cumin dietary fiber with laccase and cellulase under high hydrostatic pressure. *Carbohydrate Polymers*, 136, 87-94.
27. Ma, M., Mu, T., Sun, H., Zhang, M., Chen, J., and Yan, Z. (2015). Optimization of extraction efficiency by shear emulsifying assisted enzymatic hydrolysis and functional properties of dietary fiber from deoiled cumin (*Cuminum cyminum* L.). *Food Chemistry*, 179, 270-277.
28. Mao, Y., Robinson, J., and Binner, E. (2021). Understanding heat and mass transfer processes during microwave-assisted and conventional solvent extraction. *Chemical Engineering Science*, 233, 116418.
29. Moriana, R., Vilaplana, F., Karlsson, S., and Ribes-Greus, A. (2011). Improved thermomechanical properties by the addition of natural fibres in starch-based sustainable biocomposites. *Composites Part A: Applied Science and Manufacturing*, 42, 30–40.
30. Nsor-Atindana, J., Zhong, F., and Mothibe, K. J. (2012). In vitro hypoglycemic and cholesterol lowering effects of dietary fiber prepared from cocoa (*Theobroma cacao* L.) shells. *Food & Function*, 3(10), 1044-1050.
31. Nuerxiati, R., Abuduwaili, A., Mutailifu, P., Wubulikasimu, A., Rustamova, N., Jingxue, C., Aisa, H.A., and Yili, A. (2019). Optimization of ultrasonic-assisted extraction, characterization and biological activities of polysaccharides from *Orchis chusua* D. Don (Salep), *International Journal of Biological Macromolecules*, 141, 431-443.
32. Peerajit, P., Chiewchan, N., and Devahastin, S. (2012). Effects of pretreatment methods on health-related functional properties of high dietary fibre powder from lime residues. *Food Chemistry*, 132(4), 1891-1898.

33. Raj, G. B., and Dash, K. K. (2020). Ultrasound-assisted extraction of phytochemicals from dragon fruit peel: Optimization, kinetics and thermodynamic studies. *Ultrasonics Sonochemistry*, 68, 105180.
34. Sepulveda, L., Roman ' 'i, A., Aguilar, C. N., and Teixeira, J. (2018). Valorization of pineapple waste for the extraction of bioactive compounds and glycosides using autohydrolysis, *Innovative Food Science and Emerging Technologies*. 47, 38-45.
35. Wang, J. C., and Kinsella, J. E. (1976). Functional properties of alfalfa leaf protein: Foaming. *Journal of Food Science*, 41(3), 498-501.
36. Wang, L., Xu, H., Yuan, F., Pan, Q., Fan R., and Gao, Y. (2015). Physicochemical characterization of five types of citrus dietary fibers, *Biocatalysis and Agricultural Biotechnology*., 4, 250-258.
37. Wang, W., Ma, X., Jiang, P., Hu, L., Zhi, Z., Chen, J., ... and Liu, D. (2016). Characterization of pectin from grapefruit peel: A comparison of ultrasound-assisted and conventional heating extractions. *Food Hydrocolloids*, 61, 730-739.
38. Wang, W., Ma, X., Xu, Y., Cao, Y., Jiang, Z., Ding, T., ... and Liu, D. (2015). Ultrasound-assisted heating extraction of pectin from grapefruit peel: Optimization and comparison with the conventional method. *Food Chemistry*, 178, 106-114.
39. Wongkom, L., and Jimtaisong, A. (2017). Novel biocomposite of carboxymethyl chitosan and pineapple peel carboxymethylcellulose as sunscreen carrier. *International Journal of Biological Macromolecules*, 95, 873-880.
40. Yang, H., Yan, R., Chen, H., Lee, D. H., and Zheng, C. (2007). Characteristics of hemicellulose, cellulose and lignin pyrolysis. *Fuel*, 86, 1781-1788.
41. Yang, Y. Y., Ma, S., Wang, X. X., and Zheng, X. L. (2017). Modification and application of dietary fiber in foods. *Journal of Chemistry*, (1), 9340427.
42. Zhang, W., Zeng, G., Pan, Y., Chen, W., Huang W., and Li, Y., (2017). Properties of soluble dietary fiber-polysaccharide from papaya peel obtained through alkaline or ultrasound-assisted alkaline extraction, *Carbohydrate Polymers*, 172, 102-112
43. Zheng, Y., Li, Y., Xu, J., Gao, G. and Niu, F. (2018). Adsorption activity of coconut (*Cocos nucifera* L.) cake dietary fibers: effect of acidic treatment, cellulase hydrolysis, particle size and pH, *RSC Advances*, 8, 2844-2850
44. Zheng, Y., Wang, X., Tian, H., Li, Y., Shi, P., Guo, W., and Zhu, Q. (2021). Effect of four modification methods on adsorption capacities and in vitro hypoglycemic properties of millet bran dietary fibre. *Food Research International*, 147, 110565.

45. Zhu, Y., Chu, J., Lu, Z., Lv, F., Bie, X., and Zhao, H. (2018). Physicochemical and functional properties of dietary fiber from foxtail millet (*Setaria italica*) bran, *Journal of Cereal Science*, 79, 456-461.