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ORIGINAL ARTICLE

Extraction and evaluation of structural and physicochemical properties of dietary fiber concentrate from mango peels by using green approach

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Abstract
The non-conventional sources of dietary fiber such as fruit by-products have recently gained attention due to its well-known functional and physiological properties. Green extraction techniques are of major interest as it helps overcome the drawbacks associated with conventional techniques. Mango peels which are a major by-product of mango fruit are a promising raw material for the recovery of dietary fiber. The present work is focused on the comparison of enzymatic, ultrasound, and ultrasound-assisted enzymatic extraction techniques for dietary fiber concentrate from mango peels considering their structural and certain physicochemical parameters. The highest extraction yield (71%) of total dietary fiber was obtained using ultrasound-assisted enzyme extraction at 25 °C temperature, 40% amplitude, and 1:50 solid-to-liquid ratio after 9 min extraction time. The results specified that mango peels can be effectively utilized for dietary fiber recovery using ultrasound-assisted enzyme extraction.

Keywords Mango by-product · Dietary fiber · Ultrasonication · Valorization · Green techniques

1 Introduction

After the industrial processing of fruit, the discarded by-products if not disposed or treated properly cause serious environmental hazards due to emission of greenhouse gases during decomposition. Moreover, the biomaterials such as oil and pectin as well as sugar present in fruit wastes can stimulate aerobic bacterial action that speeds up the decomposition rate of biodegradable organic matter [1]. The unscrupulous disposal of these by-products in the environment leads to qualitative (nutrition, calorific value, etc.) and quantitative (volume or mass) losses, thus reducing the economic value as well as consumer acceptability of the product [2]. These problems can be circumvented by extraction of bioactive compounds from fruit by-products with functional benefits or its valorization into value-added products by using various green techniques [3].

The mango peels are one of the major by-products of mango fruit and represents about 15–20% of the fresh fruit [4]. Recently, mango peels have gained considerable interest among researchers as it is rich in numerous valuable compounds, such as dietary fiber, polyphenols, and carotenoids possessing functional and antioxidant properties [5, 6]. Dietary fiber is widely used as a functional food ingredient in processed foods as it imparts several health benefits [5]. It constitutes plant-derived or other carbohydrate oligomers and polymers which cannot undergo hydrolysis by endogenous enzymes in the small intestine of humans [7]. Dietary fiber intake causes a significant impact on human body as it may help improve blood cholesterol levels and lipid profiles as well as regulate blood glucose level. Some chronic diseases such as diabetes, obesity, and colon cancer can also be prevented by including dietary fiber in the diet [8–10].

Depending on its solubility in water, dietary fiber can be roughly classified into soluble (SDF) and insoluble (IDF) types. Among them, SDF constitutes soluble hemicelluloses, mucilage, pectic substances, and gums, while IDF contains cellulose, lignin, chitosan, and insoluble hemicelluloses [11]. Moreover, they also differ in their

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physiological response in the body. SDF performs a role in lowering blood glucose, increasing viscosity, and lowering plasma cholesterol, whereas IDF contributes in increasing fecal volume and facilitates defecation [12, 13]. Apart from its health-enhancing effects, the structure-forming properties of dietary fibers are of equal importance. Functional properties like solubility, water/oil holding capacity, gelling potential, and flowability are significantly influenced by soluble dietary fiber fraction [14]. This makes its utilization suitable in food systems as a thickening and gelling agent, stabilizing and encapsulating agent, etc. [8, 9].

The extraction processes of dietary fiber are very expensive and complex which compromises the extraction yield. Therefore, obtaining pure dietary fiber without the presence of impurities, like proteins and lipids, is still a challenging subject [14]. The high anti-radical efficiency and lower content of lipid (2.35%) and protein (4.28%) in mango peels dietary fiber are important parameters in a fiber concentrate [15] and make mango peels suitable for the extraction of dietary fiber and its application in food products. Chemical and enzymatic methods are extensively used for the recovery of dietary fiber from different food sources. The conventional techniques involve the use of high temperature, longer time, and toxic chemicals which can cause degradation of the extracted polysaccharide. Moreover, the processing condition leads to a significant impact on composition and microstructure of dietary fibers effecting the physiochemical and functional properties [16]. In the past years, the ultrasonication technique, due to its eco-friendly nature, has been successfully employed in research and development area in the food industries. It has been utilized in the extraction of polysaccharides, oils, and proteins [17]. Ultrasonic treatment has been reported not only to increase the extraction yield and reaction rate but also reduce the extraction time [18]. Ultrasound-assisted enzymatic extraction is a new and promising extraction method which holds twofold benefits. The involvement of enzymes in ultrasonication treatment may reduce solvent consumption and extraction time as well as improve the extraction yield of the target compound.

The extraction of dietary fiber from food industry by-products including mango peels using conventional methods have been abundantly studied [19–21]. However, limited studies have been performed using green techniques for the extraction of dietary fiber from food industry by-products [22]. In view of the above, the present investigation was carried out to evaluate the effect of different green extraction methods (ultrasound-assisted extraction, enzyme-assisted extraction, and ultrasound-assisted enzyme extraction) on the extraction of dietary fiber concentrate from mango peels as well as their structural and physicochemical properties.

2 Materials and methods

2.1 Materials

Mango (*Mangifera indica*) peels were procured from the local fruit processors, Longowal, Punjab, India. The sample was dried using sun drying method at ambient temperature for 3–4 days, grounded to fine powder, and stored in an air tight vessel at 4 ± 1 °C for further studies. All the chemicals used in the study were of analytical grade.

2.2 Proximate analysis of mango peel powder

Mango peel powder was analyzed for moisture, ash, protein, crude fiber, and fat, using the standard AOAC (1990) methods [23]. Nitrogen content was estimated by Kjeldahl method and was converted to protein by using factor 6.25. Available carbohydrate was estimated by subtracting percentage sum of other proximate constituents from 100, i.e., [100 − (%moisture + % ash + % crude protein + % crude fat + % crude fiber)].

2.3 Extraction of dietary fiber using conventional techniques

The conventional techniques including hot water extraction [24], ethanol extraction [25], and alkaline extraction [26] were utilized for the extraction of dietary fiber concentrate from mango peels.

2.4 Extraction of dietary fiber using green techniques

The green techniques including ultrasound-assisted extraction, enzyme-assisted extraction, and ultrasound-assisted enzyme extraction were utilized for the extraction of dietary fiber concentrate from the mango peels.

2.4.1 Ultrasound-assisted extraction

Extraction of dietary fiber from mango peels was carried out using the ultrasound method [14] with some modifications. The sample (1 g) was weighed in a beaker (400 mL), mixed with phosphate buffer (0.08 M, 50 mL), and pH adjusted to 6.0 ± 1. The mixture was ultrasonicated using ultrasonic probe (Snaptech NexTgen Lab 500) at 25 °C for 9 min with ultrasonic amplitude of 40%. After this, ethanol (twofold volume) was added to the solution, and the suspension was left (60 min) to precipitate. The residues obtained were filtered and washed with ethanol (80%) followed by oven drying (40 °C) to obtain dietary

fiber concentrate. The extraction yield was calculated using Eq. (1):

$$\textit{Dietary fiber concentrate}\ (\%w/w) = \frac{(\textit{Weight of residue})}{(\textit{Weight of sample})} \times 100 \quad (1)$$

2.4.2 Enzyme-assisted extraction

The extraction of dietary fiber from mango peels was carried out using enzyme-assisted extraction [27]. The sample (1 g) was added to phosphate buffer (50 mL, 0.08 M), and the pH (6.0) was adjusted. Then, α-amylase (50 µL) was added into the mixture, followed by placing it in boiling water bath (98–100 °C, 15 min). Later the mixture was enzymatically digested in sequence using protease (100 µL, pH 7.5) and amyloglucosidase (200 µL, pH 4.5) followed by incubation on water bath (60 °C, 30 min). After incubation, ethanol (twofold volume) was added to the solution, and the suspension was left for 60 min to precipitate. The residue obtained was filtered and washed with ethanol (80%) followed by oven drying (40 °C) to obtain dietary fiber concentrate. The extraction yield was calculated using the above Eq. (1).

2.4.3 Ultrasound-assisted enzyme extraction

Extraction of dietary fiber from mango peels was carried out using ultrasound-assisted enzyme extraction [14] with slight modifications wherein ultrasonication treatment was given prior to enzyme extraction. The sample (1 g) was weighed, mixed with phosphate buffer (0.08 M, 50 mL), and pH was adjusted to 6.0 ± 1. The suspension was sonicated using an ultrasonic probe (Snaptech NexTgen Lab 500) at 25 °C for 9 min with 40% ultrasonic amplitude. The subsequent steps were the same as in the enzymatic extraction procedure. The effect of time (3–12 min), extraction temperature (25–55 °C), amplitude (20–60%), and solid/liquid ratio (1:30–1:60) on yield of dietary fiber concentrate was investigated.

2.4.4 Estimation of soluble, insoluble, and total dietary fiber content

The estimation of soluble and insoluble dietary fiber content was carried out using the procedure of Wang et al. [26]. The mixture obtained after each extraction treatment was subjected to centrifugation (5000 g, 15 min) to separate soluble and insoluble dietary fiber content. The residue obtained was separated, rinsed by distilled water, and dried to obtain insoluble dietary fiber. The supernatant was collected, added with fourfold volume of ethanol (95%), and kept for 2 h to precipitate. The collected residue was washed with ethanol (100%) followed by drying to obtain soluble dietary fiber. The total dietary fiber content was determined as the sum of soluble and insoluble dietary fiber content [14].

2.5 Structural properties

2.5.1 Scanning electron microscopy (SEM)

The surface morphology and the microstructure of dietary fiber concentrate obtained using green techniques were studied using scanning electron microscope (JSM, 7610 F plus, JEOL, Japan). The samples were uniformly dispersed on a conductive surface, and coating was done using a thin layer of gold in an argon atmosphere using an iron sputter coater. The images of the samples were taken at different magnifications [28].

2.5.2 X-ray diffraction (XRD)

The XRD patterns of dietary fiber concentrate were obtained using a PAN-analytic-Xpert PRO MRD (Almelo, Netherlands). Diffractograms were taken between 5 and 50° at a rate of 0.21°/s and with a step length of 0.05° [29].

2.6 Physicochemical properties

2.6.1 Water holding capacity

To find the water holding capacity (WHC) of dietary fiber concentrate from mango peels, the sample (0.5 g) was added to distilled water (20 mL) and mixed well. After soaking the sample for 24 h, the slurry was centrifuged (4000 g, 10 min). The supernatant was discarded, and the weight of residue was noted. WHC was calculated as the amount of water retained by the sample using Eq. (2) [30].

$$\textit{WHC}\ (g/g) = \frac{W_2 - W_1}{W_1} \quad (2)$$

where W_2 is the final weight of residue and W_1 is the initial weight of the sample.

2.6.2 Oil holding capacity

The oil holding capacity (OHC) of dietary fiber concentrate from mango peels was determined by a modified method as given [31]. The sample (0.5 g) and vegetable oil (10 mL) were mixed and centrifuged (4000 g, 10 min) after resting at ambient temperature for 1 h. The supernatant was discarded, and the weight of the residue was noted. The OHC was calculated using Eq. (3):

$$\textit{OHC}\ (g/g) = \frac{O_2 - O_1}{O_1} \quad (3)$$

where O_2 is the final weight of residue and O_1 is the initial weight of the sample.

3 Statistical analysis

The data obtained for the total, soluble, and insoluble dietary fiber content from mango peels using different green techniques were analyzed by using GraphPad Prism (La Jolla, CA, USA) (version 5.01) software. The results have been expressed as means ± SD. Differences between the means were tested for statistical significance using a two-way ANOVA and followed by Bonferroni post hoc test. The significance level was set at 5% ($P < 0.05$) for all calculations.

4 Results and discussion

The proximate analysis of the mango peels was carried out, and the yield of dietary fiber concentrate obtained from mango peels along with their structural and physicochemical parameters was evaluated using different extraction techniques which have been discussed below.

4.1 Proximate analysis of mango peel powder

The proximate composition of mango peel powder is shown in Table 1. The results showed that the moisture content was 11.4%, the fat content was 1.55%, the ash content was 3.41%, and the total protein content was 6.4%. The carbohydrate content in mango peel powder was found to be 66.4%. The proximate composition reported was comparable to the earlier reported values of mango peels [32–34].

Table 1 Proximate composition of mango peel powder

Component	Content (g/100 g dry matter)
Protein	6.4 ± 0.12*
Carbohydrate	66.4 ± 0.05
Fat	1.55 ± 0.25
Ash	3.41 ± 0.34
Crude fiber	10.74 ± 0.19
Moisture	11.4 ± 0.45

*Mean ± SD ($n = 3$)

4.2 Effect of conventional and green techniques on extraction yield of dietary fiber concentrate

The comparative evaluation of the extraction yield of dietary fiber concentrate obtained using conventional (hot water extraction, ethanol extraction, alkaline extraction) and green techniques (ultrasound-assisted extraction, enzyme-assisted extraction, and ultrasound-assisted enzyme extraction) revealed that the extraction of total dietary fiber using hot water gave the lowest yield (26%) followed by ethanol extraction (44%) and alkaline extraction (59%). Even though extraction of dietary fiber from plant sources using hot water is one of the most commonly utilized methods, long exposure to heating at high temperature may lead to the degradation of the extracted polysaccharide, thereby giving lower yield. Also, the water extractability is lower than that achieved by chemical methods [22]. The extraction yield was found to be more in ethanol extraction but even greater in the case of the alkaline method. This might be because alcohol extraction is particularly suitable for fruits and vegetable matter containing a small amount of starch, intracellular proteins, and polyphenols [35]. Alkaline extraction releases various polysaccharides from the cell wall due to the disruption of hydrogen and covalent bonds. Also, the hydroxyl ions disrupt the hydrogen bonds [22], thus leading to better extraction yield (Fig. 1).

Chemical or thermal extraction methods are highly efficient, but only a portion of the extracted polysaccharides obtained through these processes can be absorbed during digestion in the upper gut. On the other hand, the use of enzymes resulting in extraction conditions similar to those present in the digestive system increases the viscosity and facilitates the greater release of polysaccharides from the mango peel matrix. The yield of dietary fiber concentrate in case of green techniques was found to increase in the following order: enzymatic extraction (55%), ultrasound extraction (60%), and ultrasound-assisted enzymatic extraction (79%). Here, the yield of dietary fiber concentrate in the case of enzymatic extraction was found lesser than obtained through the alkaline method. This may be due to the destruction of the cell wall to a greater extent in case of alkaline solution enabling better penetration and release of fiber contents [36]. Even though thermal and chemical treatments for dietary fiber are efficient to a large extent, they require high temperature, longer extraction time, and toxic solvents. These shortcomings can be overcome using green techniques which involve shorter duration, lower temperature, and non-toxic solvents with better extraction yield. Ultrasonication extraction gave even more yield than enzymatic treatment, and this yield was further enhanced when enzymatic treatment was assisted by ultrasonication. It disrupts the cell structure of fruit wall tissue increasing the accessibility of

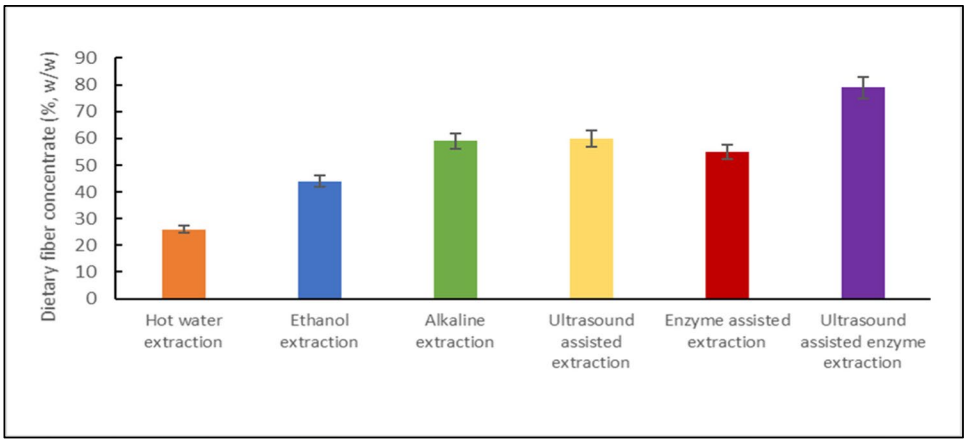

Fig. 1 Extraction of dietary fiber concentrate from mango peel using different extraction techniques

the solvent to the internal structure, thus releasing the cell components more effectively [22].

4.3 Effect of extraction parameters on extraction yield of dietary fiber concentrate obtained using ultrasound-assisted enzymatic method

The effect of extraction parameters including time, temperature, amplitude, and liquid/solid ratio on extraction yield of dietary fiber concentrate obtained using ultrasound-assisted enzymatic method was investigated. The variation in extraction yield with change in extraction parameters is shown in Fig. 2. The extraction yield was found to decrease with increase in temperature. The yield was found to gradually decrease in initial stage and a significant drop was observed in the later stage which indicates a noteworthy impact of temperature on dietary fiber concentrate yield. This might be due to the sensitivity of the material to temperature. Moreover, it also depends on the polysaccharide to be recovered and matrix characteristics [22]. The findings were similar to that found in case of soybean residue where the highest yield of dietary fiber concentrate was observed at 30 °C [37].

When the extraction time was increased, the yield was also found to increase until 9 min after which a gradual drop in the yield was observed [38]. Ultrasonic amplitude significantly influenced the extraction yield of dietary fiber concentrate. The extraction yield was found to increase with the increase in amplitude up to 50% after which no significant impact was observed. As the ultrasonic waves of larger amplitude progress through the medium, more bubbles are created which are very unstable and collapses over a short time. It may have resulted in increased penetration

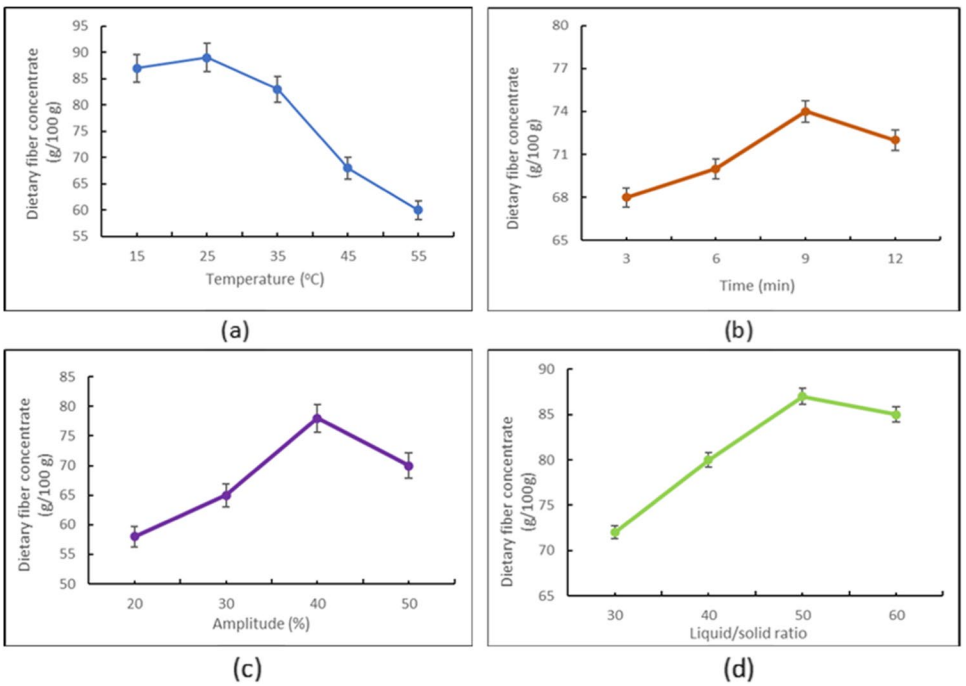

Fig. 2 Effect of extraction parameters on the yield of dietary fiber concentrate obtained from mango peels using ultrasound-assisted enzyme extraction. **a** Temperature. **b** Time. **c** Amplitude. **d** Liquid/solid ratio

of solvent into the tissues leading to the disruption of cell wall and release of intracellular product [39]. Similar trend was observed in case of increase in solid/liquid ratio, where the yield of dietary fiber concentrate obtained from mango peels gradually increased from 1:30 to 1:50 after which the yield declined slightly [37].

4.4 SEM analysis

Structural characterization of the fiber samples obtained using ultrasound-assisted extraction, enzyme-assisted extraction, and ultrasound-assisted enzyme extraction techniques was done by means of scanning electron microscopy (SEM) which revealed irregular shaped granules of uneven size and dense surfaces. The compactness of the particles was found more in case of enzyme-assisted extraction method as compared to those in ultrasonicated samples. The ultrasonic treatment disrupted the crosslinks between polysaccharide molecules more effectively and resulted in numerous significantly smaller sized particles [14]. The samples which had undergone ultrasonic treatment showed more disrupted and looser structure which might be due to pore expansion during ultrasonication [40]. The distortedness and number of cracks and holes were even more in samples treated using ultrasound-assisted enzyme technique which indicated the greater

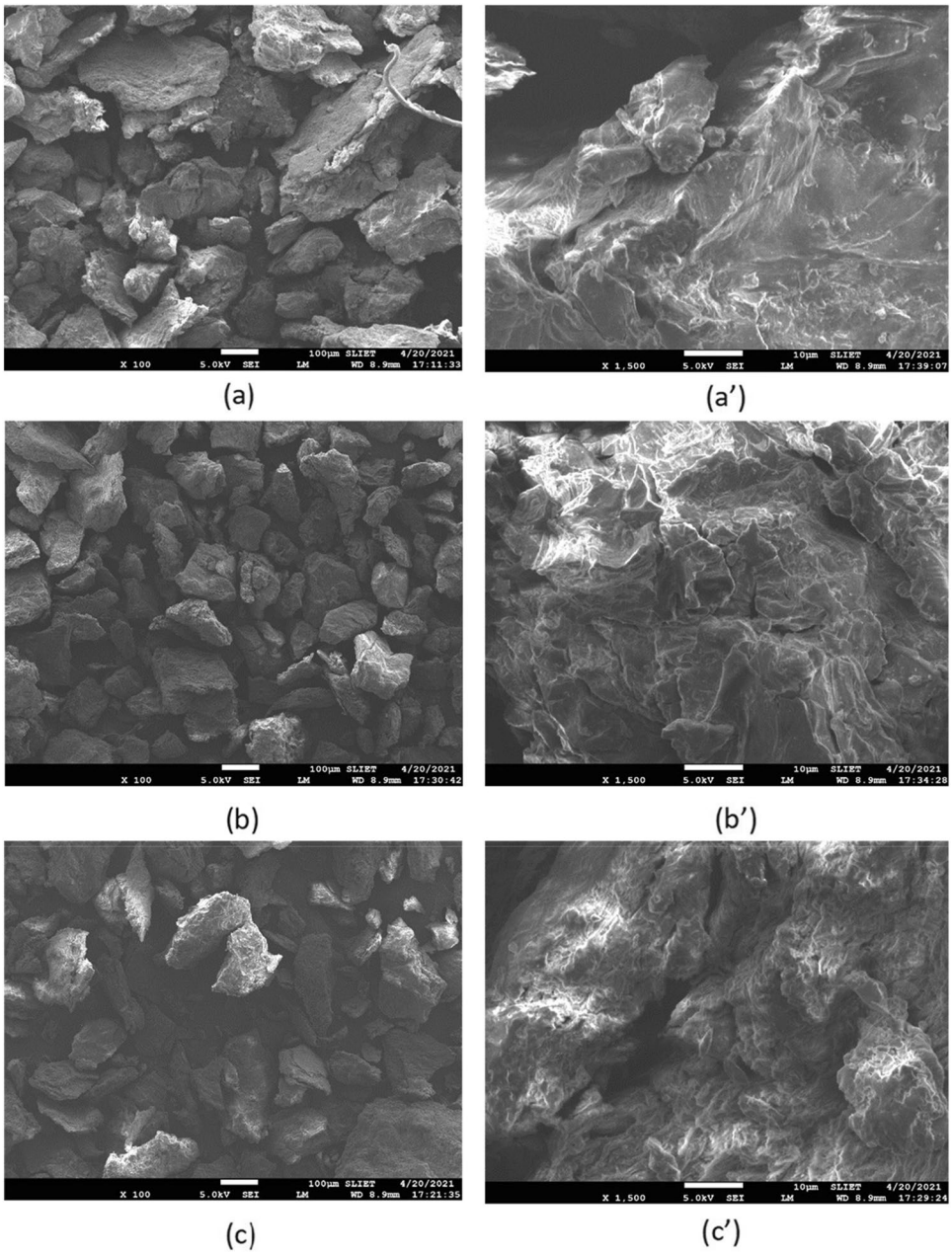

Fig. 3 Scanning electron microscopy of dietary fiber extracted from mango peels using green techniques. (**a** EAE × 100; **a'** EAE × 1500; **b** UAE × 100; **b'** UAE × 1500; **c** UEE × 100; **c'** UEE × 1500)

efficiency of this method in dietary fiber extraction among all the green techniques (Fig. 3).

4.5 XRD analysis

The influence of different extraction treatments on the crystal structure of the polymers in the fiber concentrate of mango peels were analyzed by X-ray diffraction. Different XRD spectra corresponding to the extraction techniques (ultrasound-assisted extraction, enzyme-assisted extraction, and ultrasound-assisted enzyme extraction) have been given in Fig. 4. The results showed several low- or high-intensity peaks at $2\Theta^\circ = 21.9$; 22.1, 25.9, 31.6, and 45.4°. The high-intensity peaks indicated the presence of cellulose molecules, whereas the low-intensity peaks were linked to the presence of hemicellulose and lignin. It has been reported that dietary fiber comprises 70% orderly crystalline and 30% amorphous regions. The crystalline cellulose is characterized by sharp XRD peaks with an extended crystalline, whereas amorphous region is composed of non-crystalline cellulose, hemicellulose, and lignin [29]. It should be noted that the degree of crystallinity could be affected by the treatment method used for extraction of dietary fiber concentrate from mango peel [25]. In this case, ultrasonicated samples were found to possess more crystallinity as compared to individual enzyme-treated sample. This might be due to the ultrasonication treatment causing the hydrolysis of hemicellulose and removal of amorphous portion of cellulose resulting in increased crystallinity [41].

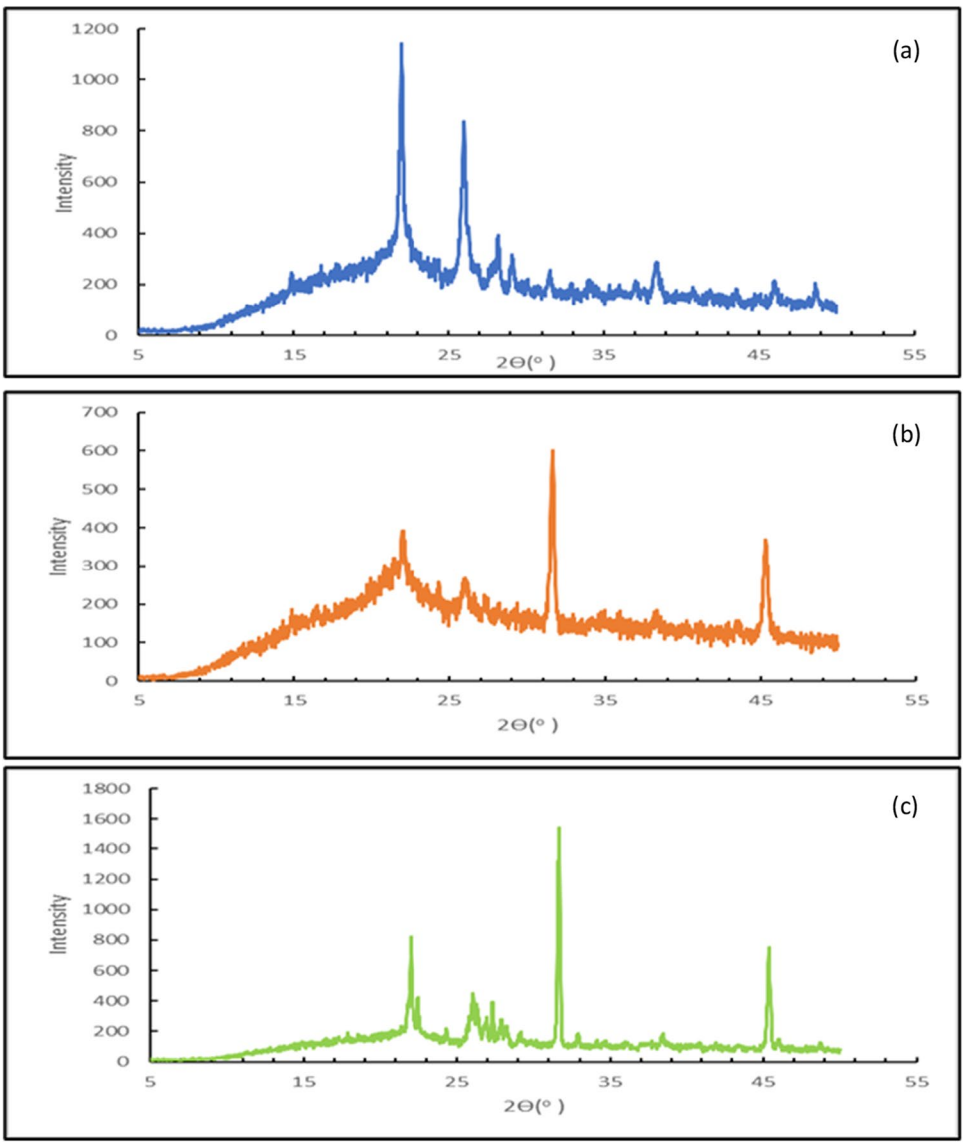

Fig. 4 XRD analysis of dietary fiber concentrate from mango peel obtained using green techniques: **a** ultrasound-assisted extraction; **b** enzyme-assisted extraction; **c** ultrasound-assisted enzyme extraction

Table 2 Total dietary fiber, soluble dietary fiber, insoluble dietary fiber, water holding capacity (WHC), and oil holding capacity (OHC) of fiber concentrate obtained from mango peels using different extraction techniques

S. No.	Extraction method	Total dietary fiber (g/100 g dry matter)	Soluble dietary fiber (g/100 g dry matter)	Insoluble dietary fiber (g/100 g dry matter)	Water holding capacity (g/g)	Oil holding capacity (g/g)
1.	Ultrasound-assisted extraction	68 ± 0.03 [b]	26.4 ± 0.03 [a]	41.1 ± 0.03 [b]	4.3 ± 0.05 [b]	1.56 ± 0.12 [a]
2.	Enzyme-assisted extraction	66 ± 0.02 [a]	27.2 ± 0.04 [c]	38.8 ± 0.05 [a]	4.5 ± 0.17 [c]	1.55 ± 0.23 [a]
3.	Ultrasound-assisted enzyme extraction	71 ± 0.04 [c]	26.9 ± 0.02 [b]	44.6 ± 0.01 [c]	4.0 ± 0.09 [a]	1.58 ± 0.34 [a]

*Mean ± SD ($n = 3$)

[a*]Different upper case superscripts in the same columns indicate the significant difference ($p < 0.05$)

4.6 Total, soluble, and insoluble dietary fiber content, water holding capacity, and oil holding capacity of dietary fiber concentrate obtained using different green techniques

Total dietary fiber content of the fiber concentrate obtained from mango peels using ultrasound-assisted extraction, enzyme-assisted extraction, and ultrasound-assisted enzyme extraction is presented in Table 2. The results indicated that the highest total dietary fiber content was obtained using ultrasound-assisted enzyme extraction (71 g/100 g) followed by ultrasound-assisted extraction (68 g/100 g) and enzyme-assisted extraction (66 g/100 g). There was not much difference observed between the total dietary fiber content obtained using ultrasound-assisted and enzymatic method. However, their combined effect led to an enhancement in the total dietary fiber content. The result was in coherence with previous studies where the maximum content of the total dietary fiber obtained from mango peels was found to be 70–72.5% [5, 19].

The soluble dietary fiber content was found maximum using enzymatic extraction (27.2 g/100 g) followed by ultrasound-assisted enzyme method (26.9 g/100 g) and ultrasound method (26.4 g/100 g). This indicated that enzymatic hydrolysis had a significant impact on the content and composition of fiber samples [28]. On the other hand, ultrasound treatment caused cell wall damage and negatively influenced soluble dietary fiber content [30]. The results were in accordance with a previous study on quinoa, amaranth, and millet, where enzymatic treatment gave higher content of soluble dietary fiber as compared to ultrasound- and ultrasound-assisted enzymatic extraction [42]. In the case of insoluble dietary fiber content, least value was obtained with enzymatic extraction (38.8 g/100 g). This might be due to the amount of protein remaining in the sample due to the formation of protein–polysaccharide complex. A comparatively higher content of insoluble dietary fiber content was obtained using ultrasound method (41.1 g/100 g) and ultrasound-assisted enzymatic extraction method (44.6 g/100 g). The results correlated with the previous findings on flaxseed gum where least content of insoluble dietary fiber was obtained using enzymatic method in comparison to ultrasound-assisted enzymatic extraction [14].

The water holding capacity is an important functional property of food hydrocolloids. The WHC of a food material expresses its ability to retain water after processes like compression and centrifugation. The WHC of dietary fiber concentrate obtained using different extraction methods revealed the highest WHC in case of enzymatic method (4.5 g/g) followed by ultrasound-assisted enzyme extraction (4.3 g/g) and ultrasound-assisted extraction (4.0 g/g). The greater value of WHC in case of enzymatic method might be due to the breakage of intermolecular hydrogen bonds of cellulose and hemicelluloses present in fiber concentrates. This increased the surface exposure of hydrated hydroxyl and carboxyl groups, thus increasing the capillary action of the fibers that, in turn, led to an increase in WHC [43].

Oil holding capacity is another important technological property which depends on the chemical and physical structure of plant polysaccharides. The OHC of fiber concentrate plays a significant role in the prevention of fat loss during food processing and in the reduction of serum cholesterol levels [44]. The values obtained were similar to that reported in earlier studies [19]. The lower content of OHC observed in the fiber concentrate of mango peels indicates that the food supplemented with this fruit by-product will not have high oil retention capacity.

5 Conclusions

Green techniques (ultrasound-assisted extraction, enzyme-assisted extraction, and ultrasound-assisted enzyme extraction) had been successfully utilized in the present study for the extraction of total dietary fiber from mango peels. Among these techniques, ultrasound-assisted enzyme extraction gave the highest yield of total dietary fiber (71%) at 25 °C temperature, 40% ultrasonic amplitude, and 1:50 solid-to-liquid ratio after 9 min of extraction time. The study indicated that mango peels possess considerably high

content of dietary fiber, which can serve as a potential functional food ingredient in applications of food processing, and the ultrasound-assisted enzyme extraction method is an effective green technique for the extraction of dietary fiber. However, further studies can be performed to scale up the process for increasing the extraction efficiency of dietary fiber from mango peels, thus expanding areas of their application.

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