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ORIGINAL PAPER

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EFFECTS OF ALKALINE EXTRACTION CONDITIONS ON THE YIELD AND PHYSICOCHEMICAL PROPERTIES OF DIETARY FIBER FROM CAT CHU MANGO (*MANGIFERA INDICA* L.) PEEL

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ABSTRACT

Background. Dietary fiber may undergo desirable and undesirable modifications in structure and composition under different extraction conditions, and demonstrate different yields and physicochemical properties. This study aimed to evaluate the effects of different alkaline extraction conditions on the recovery yield and various physicochemical properties of dietary fiber from Cat Chu mango peel.
Material and methods. Different sodium hydroxide concentrations (0.5–2%, w/v), extraction temperatures (30–50°C), extraction times (30–120 min), and solvent/material ratios (10–25 mL/g) were used to recover dietary fiber from Cat Chu mango peel. Its water solubility capacity, water holding capacity, oil holding capacity, emulsifying capacity, and emulsification stability were also assessed.
Results. Harsher exposure to alkaline solvents increased the recovery yield of dietary fiber to a certain extent; however, its physicochemical properties diminished significantly. The mild extraction condition of 1% alkaline and a solvent/material ratio of 20 mL/g at 30°C for 30 min recover dietary fiber with acceptable physicochemical properties, especially with respect to emulsification stability.
Conclusion. This study provides a fundamental understanding of changes in the yield and physicochemical properties of dietary fiber from mango peel under a wide range of alkaline extraction conditions. To gain more profound insights, it is essential to conduct component analysis and structural characterization of alkaline-extracted mango peel dietary fiber.

Keywords: alkaline extraction, dietary fiber, mango peel, physicochemical properties

INTRODUCTION

Mango (*Mangifera indica* L.) is the most popular tropical fruit, with global production of 44.36 million tons in 2022 (FAO, 2024). Vietnam is the 13th-biggest mango exporter globally, producing approximately 900,000 tons per year, mainly of Cat Chu and Cat Hoa Loc mango, located in the Mekong Delta (Nguyen et al., 2019; MARD, 2021). Mango fruit consists of pulp (35–70% w/w), peel, and kernel (30–65% w/w). The mango pulp is delivered primarily fresh/fresh-cut, pickled, or canned, or processed into juice, nectar, puree, leather, jam, or spices. On the other hand, a massive amount of mango peel and kernels (15–25 million

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tons per year) is usually eliminated from the manufacturing process of these products due to unsuitable technological properties such as bitterness and low solubility. Mango peel is vulnerable to deterioration and causes rotten smells during decomposition, leading to adverse environmental issues without proper treatment because of its high moisture content (66–75%) and rich biodegradable organic compounds (dietary fiber, protein, fat, vitamins, carotenoids and polyphenols) (Ajila et al., 2007; Brech and Sidhu, 2017; Marcal and Pintado, 2021). Researchers have made many efforts to utilize mango peel to develop value-added products, create new sources of income and alleviate biowaste in the environment (Cheok et al., 2018; Marcal and Pintado, 2021).

Up to 45–78% of a mango's dietary fiber can be found in its peel, depending on mango variety and maturity (Larrauri et al., 1996; Ajila et al., 2007; Ajila and Rao, 2013). This offers a promising opportunity to exploit the health-promoting benefits of dietary fiber through extraction. Dietary fiber from mango peel comprises mainly pectin, β-glucan, cellulose and hemicellulose based on the determination of monosaccharide composition by high-performance liquid chromatography (Larrauri et al., 1996; Ajila and Rao, 2013). However, studies involving the extraction of bioactive compounds from mango peel have mainly focused on recovering pectin or polyphenols (Cheok et al., 2018; Marcal and Pintado, 2021), whereas other dietary fiber fractions have not received much attention. Pectin (a fraction of dietary fiber) from mango is usually extracted in acidic solutions (citric acid and hydrochloric acid) with(out) microwave or ultrasound assistance (Kermani et al., 2015; Wang et al., 2016; Oliveira et al., 2018; Sommano et al., 2018; Nguyen et al., 2019). However, alkaline extractions could be used to obtain dietary fiber more efficiently than acidic extractions (Niu et al., 2018; Wandee et al., 2019; Wang et al., 2021). This process involves treating raw materials with alkaline solutions, which solubilize pectin, β-glucan and hemicellulose, leaving behind a solid residue enriched in cellulose and lignin. Alkaline extraction has been successfully applied to obtain dietary fiber from many sources, such as coffee silverskin, tomato peel, papaya peel, flaxseed, pomelo peel, and kiwi fruit (Behrouzian et al., 2016; Zhang et al., 2017; Niu et al., 2018; Moczkowska et al., 2019; Wandee et al., 2019; Wang et al., 2021). The efficiency and selectivity of alkaline extraction are affected by multiple parameters, including alkaline solution concentration or pH, extraction time, temperature and solvent-to-material ratio (Zhang et al., 2017; Wandee et al., 2019). Along with recovery yield, extraction conditions also modify the composition and structure of the dietary fiber, causing both desirable and undesirable changes in its physicochemical and functional properties (Zhang et al., 2017; Moczkowska et al., 2019; Dong et al., 2020). Alkali treatment involves treating lignocellulosic fibers in an alkali solution to remove lignin, hemicellulose, waxy substances, and natural oils covering the external surface of the fiber cell wall. Treatment with an alkali solution is one of the most effective and cheapest methods of extraction (Tenazoa et al., 2021). However, this extraction method affects both the physicochemical and rheological parameters of obtained flaxseed fiber, which could be related to differences in structure (FT-IR spectra) and thermal properties (DSC thermogram) (Moczkowska et al., 2019). Moreover, the oil holding capacity of the fiber increases and its water holding capacity decreases when using alkali extraction (Moczkowska et al., 2019).

Studies of the recovery of dietary fiber mixture from mango peel using alkaline solutions and evaluation of its physicochemical properties are still limited. Therefore, this study was conducted to observe the changes in recovery yield and evaluate the physicochemical properties of dietary fiber from mango peel under different extraction conditions. The results provide fundamental information about how to obtain dietary fiber efficiently, contributing to the valorization of mango peel by-product.

MATERIALS AND METHODS

Chemicals

Ethanol (96%) was purchased from Vietnam. Sodium hydroxide (NaOH) was supplied by Xilong (China). Commercial soybean oil was purchased in a supermarket (Tuong An, Vietnam).

Preparation of alcohol-insoluble residues

Cat Chu mango peel (from commercial, ripened fruits) at Hung Hau Fruits and Vegetables Co., Ltd,

Vietnam, was obtained after each production shift (4–6 h), packed in polyamide (PA) packages (10 kg), and transported to the laboratory within 2 h at ambient temperature. The peel was washed with tap water and then immersed in 96% ethanol (the ratio of peel to ethanol was 1:2, w/v) at 4°C for 18 h to eliminate soluble substances (phytochemicals, sugars, and organic acids) and inactivate endogenous enzymes. The peel was finally washed with 70% ethanol (v/v), dried at 65°C to constant weight (moisture content of 8–10%, wet basis), ground and passed through an 80--mesh standard sieve. The fine powder from dried peel (called alcohol-insoluble residue, AIR) was stored in vacuumed PA at –18°C for subsequent extraction.

Alkaline extraction

The AIR (10 g) was used to extract dietary fiber under varying conditions, including alkaline concentration (0.5–2.0%, w/v), extraction temperature (30–50°C), extraction time (30–120 min), and solvent/material ratio (10–25 mL/g). The investigation was conducted as series of single-factor experiments. For the experiment on the effect of alkaline concentration, a solvent/material ratio of 15 mL/g, an extraction temperature of 50°C and an extraction time of 30 min were used as constant factors. The mass of the sample was 10 g, and the volume of alkali was varied to determine the effect of solvent/material ratio (10–25 mL/g) on extraction yield and the physicochemical properties of the fiber. The results of former experiments were used to determine fixed parameter values for the later experiments. The extract was used for precipitation with 96% ethanol in which the extract:ethanol ratio was 1:4 (v/v). The slurry was left to stand overnight at 4°C. The precipitant was collected using a vacuum filter system and dried at 65°C to constant weight. The dietary fiber powder was collected by grinding and passing through an 80-mesh standard sieve. The dietary fiber powder was stored in vacuumed PA in the freezer (about from –18 to –20°C) for further analysis.

Analysis methods

Recovery yield. The yield was calculated according to the equation:

$$Y = \frac{M_1}{M_0} \times 100 \quad (1)$$

where Y is the recovery yield (%), and M_0 and M_1 are the weights of AIR and dietary fiber (g).

Water solubility capacity (WSC, %). The water solubility of dietary fiber samples was determined according to the method described by Dong et al. (2020) with minor modifications. A 0.1 g sample of dietary fiber was gently mixed with 5 mL of distilled water in a centrifuge tube. Subsequently, the mixture was heated at 90°C for 30 min with shaking after each interval of 5 min in a thermostat-controlled water bath, followed by centrifugation at 1,000 g for 20 min. The supernatant was collected, dried at 105°C to constant weight, cooled in a desiccator for 15 min and weighed. The WSC was calculated as follows:

$$WSC\ (\%) = \frac{W_1}{W} \times 100 \quad (2)$$

where W_1 and W are the weights of the dried supernatant and the sample, respectively.

Water holding capacity (WHC, g/g) and oil holding capacity (OHC, g/g). WHC and OHC were determined according to the method of Dong et al. (2020) with some modifications. Briefly, 20 mL of distilled water or 5 mL of commercial soya bean oil were mixed with 0.5 g of dried dietary fiber sample, and left at room temperature for 24 h (WHC) or 4°C for 1 h (OHC), respectively. After centrifugation (1,000 g, 30 min), the excess supernatant was decanted and the residue was weighed. WHC or OHC was caculated as below:

$$\text{WHC or OHC}\left(\frac{g}{g}\right) = \frac{W_1}{W} \quad (3)$$

where W_1 and W are the weights of the water/oil adsorbed and the sample, respectively.

Emulsifying capacity (EC, %) and emulsification stability (ES, %). The EC of dietary fiber was estimated according to the method described by Dong et al. (2020) with slight modifications. A 0.5 g sample of dietary fiber was mixed with 50 mL water and then homogenized using an ultra-turrax at 10,000 rpm for 1 min. The obtained fibrous suspension was homogenized with 50 mL soya bean oil at the same condition,

then centrifuged at 1,200 g for 5 min, and the volume of the resultant emulsion was determined. The percentage of total emulsion mixture that remained emulsified after centrifugation was denoted as the EC index, and the whole volume (W_v) of the system and emulsified layer volume (ELv_i) were determined. EC was calculated as follows:

$$EC\ (\%) = \frac{EL_{vi}}{W_v} \times 100 \quad (4)$$

ES was measured by heating the prepared emulsions at 80°C for 30 min, cooling to room temperature and centrifuging at 1,200 g for 5 min. The ES obtained was expressed in terms of the ratio between the remaining emulsified layer volume (ELv_f) and the original emulsion volume (ELv_i). ES was calculated as follows:

$$EC\ (\%) = \frac{EL_{vf}}{EL_{vi}} \times 100 \quad (5)$$

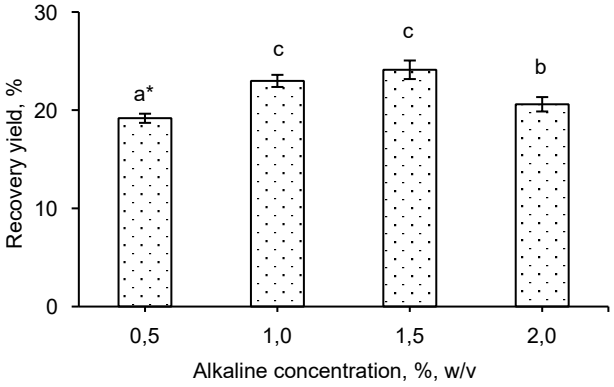
Statistical analysis

The data were statistically analyzed using Statgraphics Centurion software (version 19.1, Statgraphics Technologies, Inc., The Plains, Virginia). The differences between the means of three replications were assessed by analysis of variance (ANOVA) with a Least Significant Difference (LSD) test using a significance level of 95%.

RESULTS AND DISCUSSION

Effects of sodium hydroxide concentration

Figure 1 shows that the alkali (NaOH) concentration significantly affected the recovery yield of dietary fiber ($p < 0.05$). The recovery yield increased from 19.18 ±0.47% to 24.12 ±0.95% when NaOH concentration increased from 0.5% to 1.5% and tended to decrease at a higher NaOH concentration. Dietary fiber from mango peel mainly consists of pectin, β-glucan, cellulose, and hemicellulose (Larrauri et al., 1996; Ajila and Rao, 2013). Hydroxyl ions from NaOH solutions cause plant cell walls to swell, break down intermolecular hydrogen links between cellulose and other polysaccharides, hydrolyze the ester links of dietary



*Data are expressed as mean ±standard deviation (n = 3) and different letters above each bar indicate statistically significant differences at $p < 0.05$.

Fig. 1. Effect of alkaline concentration on the recovery yield of dietary fiber

fiber and mobilize them into the solution (Bergmans et al., 1996). Therefore, increasing NaOH concentration facilitates the effects of OH-on components made of plant cell walls. However, high NaOH concentrations might cause excessive hydrolysis, resulting in the loss of dietary fiber. The yield decreased by roughly 14.5% (from 24.12 ±0.95% to 20.60 ±0.73%) when the NaOH concentration was increased from 1.5% to 2% (Fig. 1). Zhang et al. (2017) and Wandee et al. (2019) found a similar tendency when extracting dietary fiber from papaya peel and pomelo peel, with the highest extraction yields recorded at NaOH concentrations of 1% and 2%, respectively.

OHC varied with alkaline concentration in a similar way to the recovery yield. OHC reached its highest levels of 1.13 ±0.31 g/g and 1.35 ±0.20 g/g at NaOH concentrations of 1.0 and 1.5% ($p > 0.05$), respectively. Other physicochemical properties, such as WHC, WSC, EC and ES, witnessed a considerable decline at NaOH concentration greater than 1% due to excessive hydrolysis of dietary fiber (Fig. 2). Moreover, saponification reactions between the hydroxyl and ester groups of dietary fiber could weaken its gel and emulsifying formation properties (Wandee et al., 2019). The highest values of WSC, EC, and ES were observed at NaOH concentrations of 0.5% and 1.0%, and the lowest could be seen at a NaOH concentration of 2%, while WHC only showed the highest level of

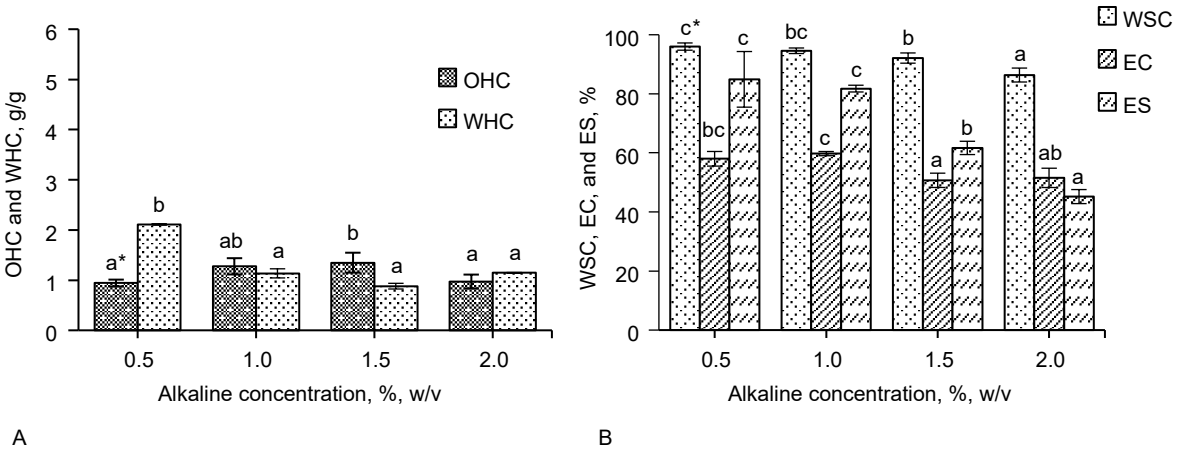


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Fig. 2. Effects of alkaline concentration on (A) oil holding capacity (OHC) and water holding capacity (WHC), and (B) water solubility capacity (WSC), emulsifying capacity (EC), and emulsification stability (ES)

2.11 ±0.14 g/g at a NaOH concentration of 0.5%. The dietary fiber obtained at a NaOH concentration of 1% clearly gave a higher yield of 22.98 ±0.62% and better physicochemical properties than the others. Therefore, a NaOH concentration of 1% was chosen to obtain high-quality dietary fiber in the follow experiments.

Effects of extraction temperature

In this experiment, we examined the effects of varying the temperature from 30°C to 50°C on the extraction of dietary fiber from mango peel. The results show that the recovery yield decreased when the extraction temperature was above 40°C (as shown in Fig. 3). A loss of dietary fiber at higher temperatures has also been observed in some previous research (Zhang et al., 2017; Moczkowska et al., 2019). The cleaving reaction of hydroxyl groups (OH-) on hydrogen and ester bonds between and within plant cell wall polysaccharides (such as cellulose, lignin, hemicellulose, and pectin) is promoted as a consequence of the increased transfer rate under a higher extraction temperature (Bergmans et al., 1996; Zhang et al., 2017). This reaction turns larger molecular-weight polysaccharides into smaller ones, and very low molecular-weight polysaccharides result in a loss of dietary fiber during precipitation and vacuum filtration (Wandee et al., 2019). We found that the highest levels of dietary fiber from mango peel were recovered at extraction temperatures of 30 and 40°C, with recovery rates of 25.45 ±1.01% and 25.18 ±1.07%, respectively ($p > 0.05$). However, at 50°C, only 22.98 ±0.62% of dietary fiber concentrate was obtained. Proteins, lipids, and soluble substances were also co-extracted in aqueous solutions and considered impurities during dietary fiber extraction (Oliveira et al., 2016; Moczkowska et al., 2019). Moczkowska et al. (2019) showed that the level of protein in fiber extracted by alkali was 22.08%. The study observed

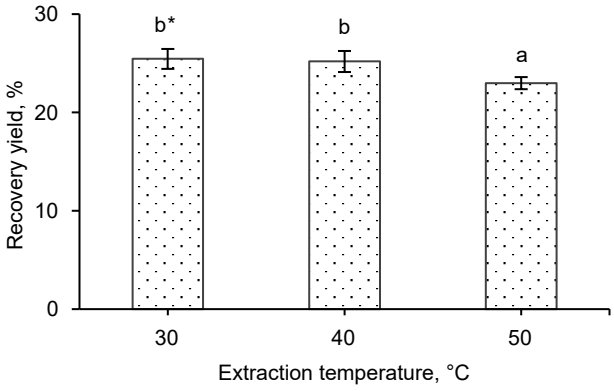

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Fig. 3. Effect of extraction temperature on the recovery yield of dietary fiber

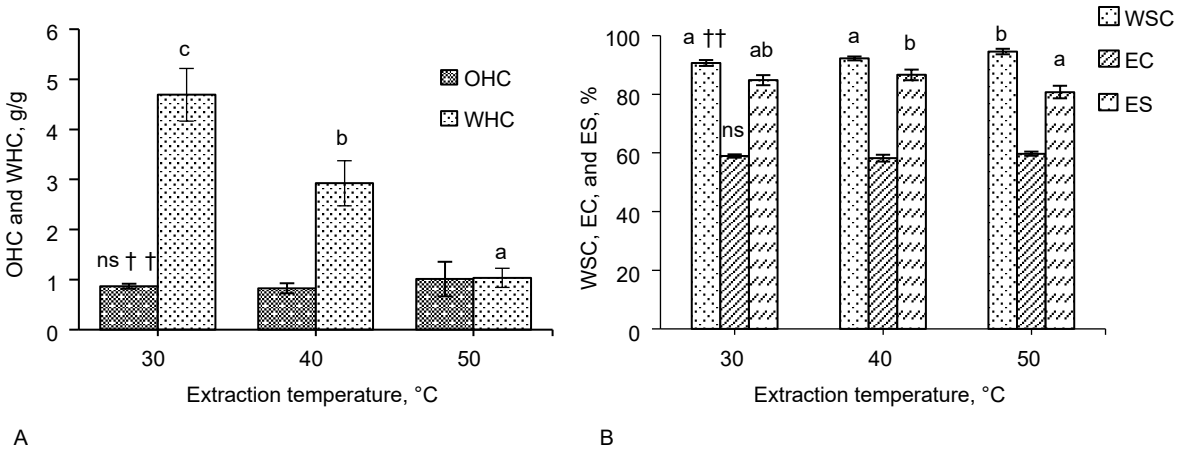

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Fig. 4. Effects of extraction temperature on (A) oil holding capacity (OHC) and water holding capacity (WHC), and (B) water solubility capacity (WSC), emulsifying capacity (EC), and emulsification stability (ES)

a yield decrease in enzymatic-ultrasonic extraction of flaxseed dietary fiber at 55°C compared to 20°C. It was concluded that protein denaturation at a higher temperature was the main factor causing the loss in extraction yield. In the study of Kaushik et al. (2017), the amount of protein was 15.1% in flaxseed gum extracted at a high temperature (90°C). The protein content of mango peel (1.5–6.6%, dry basis) was not as high as in flaxseed (20.32%, dry basis) (Moczkowska et al*.,* 2019; Marcal and Pintado, 2021), but this may contribute to the loss of extraction yield in our study.

The effects of extraction temperature on the physicochemical properties of dietary fiber are presented in Figure 4. WHC and ES decreased markedly as the temperature increased (from 4.69 ±0.53 to 1.04 ±0.19 g/g and 84.82 ±1.72 to 80.77 ±2.14%, respectively). Alkaline extraction significantly reduced the molecular weight and porosity of the obtained dietary fiber (Moczkowska et al., 2019; Wandee et al., 2019; Wang et al., 2021). This might correlate with a reduction in these physicochemical properties, especially WHC (Elleuch et al., 2010). The findings also showed a slight increase in WSC (from 90.68±1.00% at 30°C to 94.58 ±0.94% at 50°C), which could be associated with the decline in WHC. This is due to the fact that dietary fiber has a low capacity to retain water within its structure if it is easily soluble (Elleuch et al., 2010). The other properties were unchanged ($p > 0.05$) with increasing temperature. Indeed, the values of OHC and EC were around 0.83 ±0.10 to 1.01 ±0.35 g/g and 58.22 ±1.17 to 59.75 ±0.71%. Therefore, the temperature of 30°C was suitable for subsequent experiments.

Effects of extraction time

During the alkaline extraction of dietary fiber from various sources, an extraction period of 10–120 min was generally applied (Zhang et al., 2017; Niu et al., 2018; Wandee et al., 2019; Wang et al., 2021). The extraction time is an important factor that affects the extraction efficiency, as dietary fiber requires a specific time to mobilize to the extraction solvents. However, there is a risk of thermal degradation if the extraction time is too long (Zhang et al., 2017). In the present study, increasing the extraction time of dietary fiber from mango peel produced similar results (refer to Fig. 5). The recovery yield of dietary fiber decreased from 25.20 ±0.85% to 22.93 ±1.81% when the extraction time increased from 30 min to 120 min. This may have been due to thermal degradation and excessive contact in the alkali solution. These results are similar to those of Yılmaz (2013), who found that increasing the extraction time resulted in a decreased residual fraction (corn husk fibers) using alkali extraction. However, there was no significant difference ($p > 0.05$)

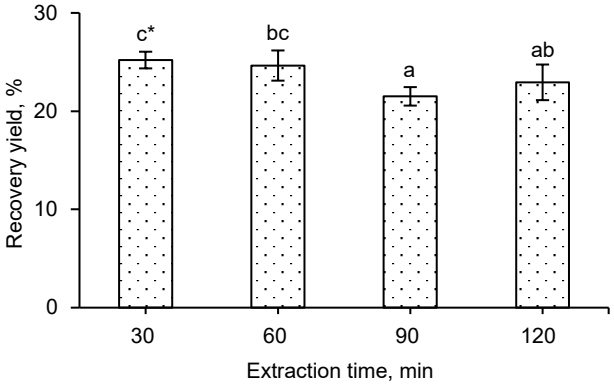

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Fig. 5. Effect of extraction time on the recovery yield of dietary fiber

in the recovery yield between extraction times of 30 and 60 min or between those of 90 and 120 min. This indicates that extraction time has less of an impact on the recovery yield than the concentration of the alkaline solution or the extraction temperature.

The physicochemical properties of dietary fiber underwent various changes with increasing extraction time, as seen in Figure 6. WHC significantly decreased from 4.69 ±0.53 to 3.48 ±0.19 g/g when extraction time was extended to 120 min. This is probably due to the differences in dietary fiber content, especially soluble fractions, as a lower WHC has previously been observed for a lower dietary fiber content (Elleuch et al., 2010). The values of OHC and WSC increased to 1.00 ±0.04 g/g and 93.47 ±0.45% at 120 min, respectively. However, extraction time had less of an influence on EC and ES ($p > 0.05$); their values remained at high levels of 56.13 ±0.26–58.95 ±0.57 and 84.82 ±1.72–87.16 ±1.95%, respectively. In conclusion, an extraction time of 30 min was found to be most suitable for obtaining dietary fiber with the highest yield and most desirable properties.

Effects of solvent/material ratio

The solvent-to-material ratio had the opposite effect on recovery yield. As shown in Figure 7, the recovery yield started at 19.43 ±0.98% and increased significantly to 25.21 ±0.85% when a ratio of 15 mL/g was employed. This increase was due to the greater contact surface between alkaline solutions and plant materials. However, increasing the solvent-to-material ratio further did not result in any significant increase in recovery yield ($p > 0.05$). This finding differs from the results of Zhang et al. (2017), who found that the recovery yield significantly decreased at a liquid-to-solid ratio above

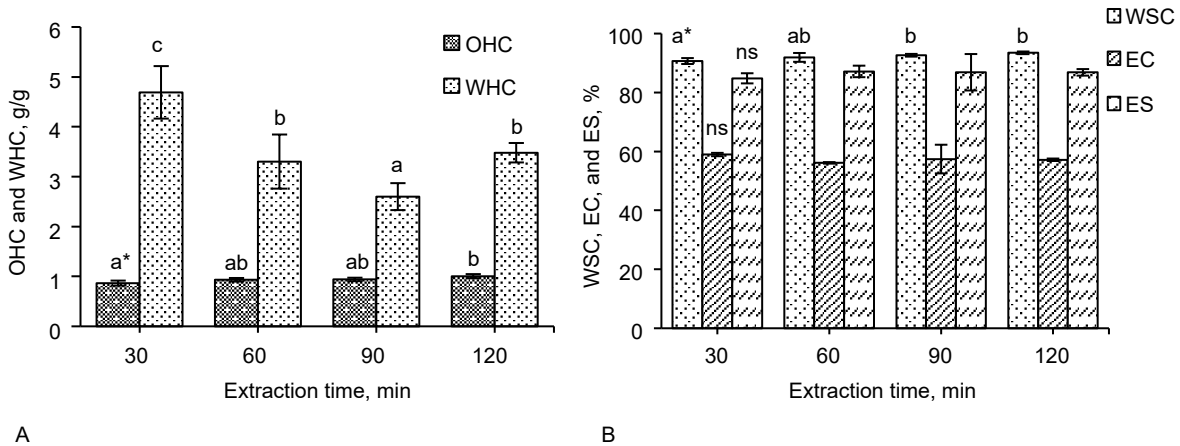

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Fig. 6. Effects of extraction time on (A) oil holding capacity (OHC) and water holding capacity (WHC), and (B) water solubility capacity (WSC), emulsifying capacity (EC), and emulsification stability (ES)

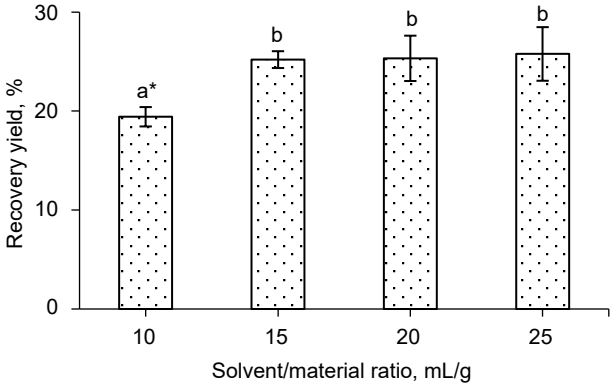

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Fig. 7. Effect of solvent/material ratio on the recovery yield of dietary fiber

15 mL/g. The differences in the two studies might be attributed to variations in the extraction conditions and food matrices. Zhang et al. (2017) extracted dietary fiber from papaya peel using 1% NaOH at 50°C for 30 min when evaluating the effect of liquid-to-solid ratio on yield, whereas we used a temperature of 30°C to recover dietary fiber in this experiment.

According to the results shown in Figure 8, the solvent-to-material ratio did not have a significant effect on the WHC, WSC, and ES of dietary fiber ($p > 0.05$). However, increasing the ratio slightly could improve the OHC and EC of dietary fiber. The OHC and EC gradually increased and reached their highest point at a solvent-to-material ratio of 20 mL/g. Therefore, this ratio was found to be optimal for obtaining dietary fiber with the highest yield and properties. Under this condition, the dietary fiber had excellent WSC (95.48 ±2.57%) but lower WHC (4.77 ±0.57 g/g) and OHC (1.05 ±0.03 g/g) compared to papaya peel (4.93 ±0.10 g/g WHC, 1.15 ±0.09 g/g OHC), flaxseed (4.85 ±0.03 g/g WHC, 1.67 ±0.06 g/g OHC) (Zhang et al., 2017; Moczkowska et al., 2019) or *Nannochloropsis oceanica* dietary fiber obtained by alkaline extraction (90.85 % for WSC, 1.17 g/g WHC, 1.04 g/g OHC) (Ding et al., 2020). Although the WHC was also considerably lower than that of pectin extracted from Cat Chu mango peel using citric acid (9.5 ±0.0 to 14.9 ±1.1 g/g) (Nguyen et al., 2019), the EC and ES (57.56 ±2.66 and 88.27 ±2.20%) were superior (11.8 ±0.9 to 24.2 ±2.0% and 35.5 ±6.0 to 60.6 ±2.7%, respectively). The two methods have different effects on dietary fiber composition, structural properties, and other factors such as porosity, particle size, ion form, and pH. These changes are linked to various physicochemical properties (Elleuch et al., 2010; Niu et al., 2018; Wandee et al., 2019; Wang et al., 2021). Alkaline extractions

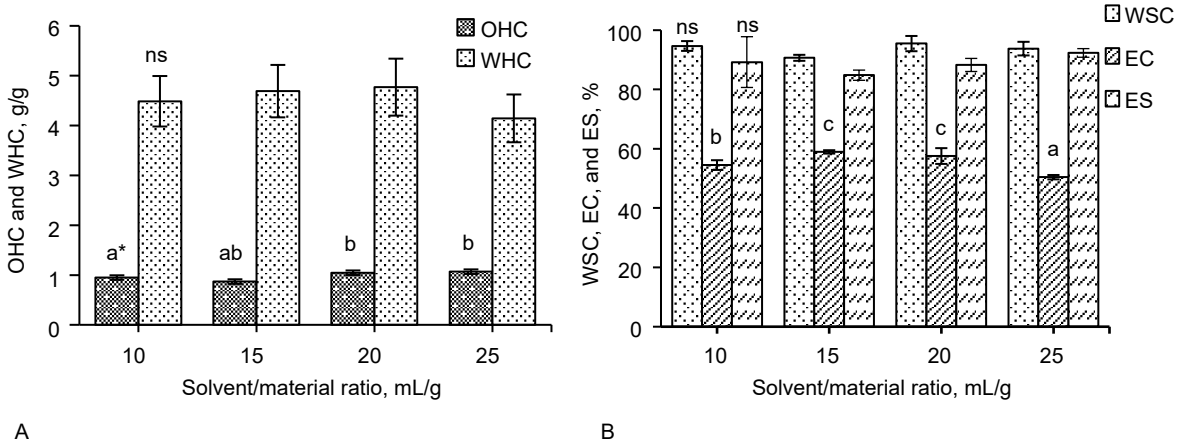

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Fig. 8. Effects of solvent/material ratio on (A) oil holding capacity (OHC) and water holding capacity (WHC), and (B) water solubility capacity (WSC), emulsifying capacity (EC), and emulsification stability (ES)

could solubilize some components of plant cell walls (cellulose and hemicellulose), while acidic extractions mainly affect soluble dietary fiber (pectin and β-glucan) (Wang et al., 2021).

CONCLUSION

It is possible to modify the process to optimize the yield and the quality of the extracted dietary fiber by controlling the alkaline extraction conditions. Dietary fiber from Cat Chu mango peel was recovered efficiently using mild alkaline extraction, showing good physicochemical properties, particularly emulsification properties. However, the component analysis and structural characterization of alkaline-extracted mango peel dietary fiber need to be carried out in further studies.

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DECLARATIONS

Data statement
All data supporting this study has been included in this manuscript.

Ethical Approval
Not applicable.

Competing Interests
The authors declare that they have no conflicts of interest.

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