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This study valorizes kinnow peel and pomace, agro-industrial citrus waste, through optimized conventional and microwave-assisted extraction of dietary fiber, contributing to waste reduction and circular bioeconomy. By converting underutilized by-products into high-value functional ingredients, the work promotes sustainable food processing and supports the UN Sustainable Development Goals, particularly SDG 12 (Responsible Consumption and Production) and SDG 3 (Good Health and Well-being). The use of microwave-assisted extraction also emphasizes energy efficiency, aligning with SDG 7 (Affordable and Clean Energy). This research fosters eco-innovation by integrating waste valorization, clean processing, and health-oriented food design.

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Microwave-Assisted Extraction of Dietary Fiber from Kinnow Mandarin By-products: Impact on Yield, Functional, Structural, and Thermal Properties

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Abstract

The extraction of dietary fiber (DF) from kinnow by-products offers a valuable opportunity for waste valorization and the development of functional food ingredients. This study compared conventional extraction methods (hot water, ethanol, and alkali extraction) with microwave-assisted extraction (MAE) to evaluate differences in fiber yield and functional, structural, and thermal properties. Among conventional methods, alkali extraction provided the highest fiber recovery, but MAE achieved comparable or higher yields with significantly reduced extraction time and lower chemical input. MAE-extracted dietary fiber exhibited superior functional properties, including water-holding capacity (7.78–9.12 g/g), oil-holding capacity (4.68–5.36 g/g), and glucose adsorption capacity (3.55–4.24 mmol/g), higher than fibers obtained via conventional methods. FTIR analysis confirmed that MAE-extracted fibers retained key functional groups, such as hydroxyl, carboxyl, and glycosidic linkages, indicating preservation of cellulose, hemicellulose, and pectin structures. XRD analysis showed that both peel and pomace fibers exhibited semi-crystalline structures, with pomace fibers showing slightly higher crystallinity, reflecting compositional differences between the two by-products. DSC

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analysis demonstrated good thermal stability in MAE-extracted fibers, with insoluble dietary fiber (ISDF) exhibiting higher thermal resistance than soluble dietary fiber (SDF). Overall, MAE proved to be a sustainable, efficient alternative for dietary fiber extraction, enhancing functionality while promoting kinnow by-product valorization.

Keywords: Dietary Fiber, Microwave-Assisted Extraction, Kinnow By-Products, Green Techniques, Functional Properties

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1. Introduction

The global citrus industry generates substantial quantities of by-products, such as peels and pomace, which are often discarded despite their potential value as sources of dietary fiber (DF). Dietary fiber refers to a diverse group of indigestible polysaccharides and lignin derived from plant cell walls, and it is now recognized as the "seventh essential nutrient" for human health [1]. Consuming adequate dietary fiber is associated with numerous health benefits, including a reduced risk of cardiovascular diseases, diabetes, obesity, and certain types of cancer, as stated in the literature by Alahmari [2]. Dietary fiber can be classified into soluble dietary fiber (SDF) and insoluble dietary fiber (ISDF) based on its solubility in water. Compared to ISDF, SDF offers superior functional and physiological properties, including a greater water and oil holding capacity, improved viscosity, and the ability to bind harmful compounds such as cholesterol and bile acids [2,3]. However, ISDF is the dominant form in most plant cell walls, while SDF typically exists in much lower proportions. Therefore, effective extraction techniques that maximize the recovery of both types, or even convert some ISDF into SDF, are highly desirable for enhancing the functionality and health benefits of dietary fiber [4,5].

Conventional methods such as hot water extraction (HWE), ethanol extraction (EtOH-E), and alkali extraction (NaOH-E) are widely used for extracting dietary fiber from plant materials. Hot water extraction is a simple and commonly applied method; however, it often requires prolonged high temperatures, which can degrade polysaccharides and lower fiber yields and functionality [6]. Ethanol extraction aids in separating fiber from soluble sugars but requires large solvent volumes and multiple rinsing steps, limiting its practicality for large-scale operations [7]. Alkali extraction, which employs sodium hydroxide to disrupt cell walls and enhance fiber release, often results in higher yields than water or ethanol methods. However, alkali treatment can modify fiber structure and may leave chemical residues, raising concerns for food applications [8]. To overcome these limitations, microwave-assisted extraction (MAE)

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has emerged as a green and sustainable alternative for extracting bioactives and dietary fiber from plant materials. Microwave technology works by inducing dipole rotation in the solvent, leading to rapid volumetric heating that breaks cell walls and enhances solubilization [9]. Unlike conventional heating, which relies on surface heat conduction, microwaves penetrate directly into the sample matrix, resulting in uniform and faster heating. This localized heating effect can also increase intracellular pressure, cause cell rupture, and release fiber and bioactive compounds more efficiently [1, 10]. The effects of microwave processing on various plant materials, particularly grains like barley, oat, millet, chickpea, and soybean, have been extensively studied. These studies have demonstrated that microwave treatment can alter starch crystalline structures, increase gelatinization temperature, and improve thermal stability and edible quality [11, 12]. Although these findings pertain to starch systems, they illustrate how microwave energy can modify plant carbohydrates in general, including the cellulose, hemicellulose, and pectin fractions that constitute dietary fiber. Such effects could similarly enhance fiber solubilization, conversion of ISDF to SDF, and preservation of functional properties during dietary fiber extraction.

Despite these promising advantages, comparative studies directly evaluating the efficiency, functional properties, and structural integrity of dietary fiber extracted using conventional versus microwave-assisted techniques remain limited, particularly for kinnow peels and pomace. Therefore, this study aims to compare conventional extraction methods (hot water, ethanol, and alkali) with microwave-assisted extraction (MAE) for dietary fiber recovery from kinnow by-products. The comparison will focus on dietary fiber yield, functional, structural, and thermal properties. It is hypothesized that microwave-assisted extraction will enhance dietary fiber recovery while preserving structural integrity and improving functional properties due to its efficient cell wall disruption, reduced thermal degradation, and shorter processing time. By addressing these research gaps, this study contributes to the broader goal of valorizing

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citrus processing waste into high-value functional ingredients, thereby promoting sustainability and reducing food processing waste.

2. Materials and Methods

2.1.Sample Preparation

The kinnow peels and pomace were procured from the local juice processing unit in Sangrur, Punjab, India. These by-products were dried in hot air at 45℃ overnight. The dried by-products were pulverized using a mini flour mill (Natraj, India). The powder was stored in air-tight containers for further experimentation.

2.2.Extraction of dietary fiber

To obtain dietary fiber from kinnow by-products, different extraction techniques were employed, including conventional extraction methods (hot water, ethanol, and alkali extraction) and MAE. The extracted dietary fiber was further characterized by functional properties, structural integrity, and composition.

2.2.1. Hot water extraction

Hot water extraction was carried out following the method described by Elleuch et al. [13] with minor modifications. The powdered kinnow peel/ pomace was mixed with distilled water (1:10 w/v) and heated at 100°C for 5 min. The suspension was subsequently centrifuged (5000 rpm, 10 min) to separate the fiber-rich precipitate from the solubilized sugars (glucose, fructose, and sucrose). To ensure complete removal of residual sugars, the precipitate was washed five times with water at 40°C and centrifuged under the same conditions. The fiber concentrate was oven-dried and stored under refrigeration for further analysis.

2.2.2. Ethanol extraction

Ethanol extraction was conducted according to Elleuch et al. [13] with modifications. The powdered kinnow peel/ pomace sample was mixed with 80% ethanol (1:10 w/v) and incubated to facilitate the solubilization of sugars. The suspension was centrifuged at 5000 rpm for 10

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min, and the fiber-rich residue was rinsed twice with ethanol. The final fiber precipitate was filtered, oven-dried at 40°C, and stored under refrigerated conditions.

2.2.3. Alkali extraction

The alkali extraction of dietary fiber was performed following the method of Lan et al. [14]. A 5 g powdered kinnow peel/ pomace sample was mixed with 75 mL of NaOH (50 mM) and stirred at 100 rpm, 45°C for optimal fiber release. After extraction, the pH of the slurry was adjusted to neutral (pH 7) using HCl (50 mM). The sample was centrifuged at 5000 rpm for 30 min, and the resulting fiber-rich residue was oven-dried for storage and further analysis.

2.2.4. Microwave-assisted extraction

MAE was conducted using a fabricated laboratory-scale microwave system following the method of Gan et al. [1]. A 5 g kinnow peel/ pomace sample was mixed with distilled water (1:10 w/v; 50 mL) in a round-bottom flask and subjected to microwave irradiation at 300 W for 30 min. After the microwave treatment, the suspension was centrifuged at 5000 rpm for 15 min, separating the insoluble dietary fiber (pellet) from the soluble fraction. For ISDF, the precipitate was washed, oven-dried, and stored for further analysis. Whereas, for SDF, the supernatant was mixed with four times its volume of 95% ethanol and left undisturbed for 12 h to precipitate soluble fiber. The precipitate was collected by centrifugation (5000 rpm, 10 min), dissolved in distilled water, and subjected to rotary evaporation for ethanol removal. The purified fiber was oven-dried and stored.

<Table 1 near here>

2.3.Characterization of dietary fiber extracted from kinnow by-products.

2.3.1. Functional Properties of Dietary Fiber

The functional properties of dietary fiber, including water-holding capacity (WHC), oil-holding capacity (OHC), and glucose adsorption capacity (GAC), were evaluated. WHC was determined by mixing 1 g of fiber with 25 mL of water, standing for 24 hours, followed by

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centrifugation and weighing the wet pellet to calculate WHC (g/g) [15]. OHC was assessed similarly, mixing 1 g of fiber with 10 mL of olive oil, followed by centrifugation and calculation based on the weight of the wet pellet. GAC was measured by incubating 1 g of fiber in 25 mL glucose solutions (50–200 mmol/L) at 37°C for 6 h, followed by centrifugation and measuring glucose content using the dinitrosalicylic acid (DNS) method. GAC (mmol/g) was calculated based on the difference between initial and final glucose concentrations [1].

$$\text{WHC}\left(\frac{\text{g}}{\text{g}}\right) = \frac{\text{Weight of wet pellet} - \text{Weight of dry sample}}{\text{Weight of dry sample}} \quad (1)$$

$$\text{OHC}\left(\frac{\text{g}}{\text{g}}\right) = \frac{\text{Weight of wet pellet} - \text{Weight of dry sample}}{\text{Weight of dry sample}} \quad (2)$$

$$\text{GAC}\left(\frac{\text{mmol}}{\text{g}}\right) = \frac{(c_i - c_f) \times V}{W} \quad (3)$$

Here, C_i = glucose level before adsorption (mmoL/L); C_f = glucose levels after adsorption (mmoL/L), W = sample weight (g), V = volume of glucose solution (L)

2.3.2. FTIR Analysis

The optimized dietary fiber concentrate of kinnow peels was analyzed to observe the functional groups present in the extracted sample, using FTIR (RX-I, FTIR, USA). The samples were oven-dried at 40 ºC and pulverized. KBr was added to the dried fiber, and FTIR spectra were recorded in the absorption range of 600 - 4000 cm^{-1}.

2.3.3. X-ray diffraction (XRD) Analysis

The XRD pattern of the dietary fiber was obtained using the XRD (D8 Advance, Bruker, Germany) instrument at the operating voltage of 30 kV and current of 20 mA. The diffraction angle (2θ) was scanned from 5° to 80° with a step-up of 0.2°/s.

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2.3.4. Thermal properties

The thermal properties of dietary fiber were analyzed using differential scanning colorimetry (DSC). Kinnow by-product sample (3-4 mg) was hermetically sealed in an aluminum pan and transferred into the instrument (PerkinElmer, DSC 4000, UK). The measurements were performed over a temperature range of 0-360°C at a constant rate of 10°C/min.

2.4. SEM analysis

The surface morphology of kinnow by-products and extracted dietary fiber was analyzed by Field Emission Scanning Electron Microscope (JSM 7610 F Plus, Jeol, Japan) operating at resolutions of 0.8 nm, 15 kV and 10 kV.

2.5.Statistical analysis

All experiments were conducted in triplicate, and data are presented as mean ± standard deviation (SD). One-way analysis of variance (ANOVA) was performed to assess the significant differences between treatments. Post-hoc comparisons were carried out using dunkan test at a significance level of $p < 0.05$. Statistical analysis was performed using SPSS software.

3. Results and Discussion

3.1.Screening of different conventional and green techniques for the extraction of dietary fiber from kinnow by-products.

3.1.1. Conventional Techniques

The efficiency of different conventional extraction methods, including hot water, ethanol, and alkali extraction, was evaluated for the recovery of dietary fiber from kinnow peels and pomace. Among these methods, alkali extraction yielded a higher dietary fiber content, with 34.34 ± 0.62% and 38.42 ± 0.77% in peels and pomace, respectively, compared to the other techniques. While hot water extraction is widely used for dietary fiber recovery from plant materials, it has inherent limitations. Prolonged exposure to high temperatures can lead to

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thermal degradation of polysaccharides, thereby reducing fiber yield and compromising its functional properties.

Chemical extraction methods, particularly alkali and ethanol extraction, demonstrated higher efficiency in dietary fiber recovery. According to Tejada-Ortigoza et al. [8], chemical treatments enhance water extractability and fiber release from plant cell walls. Alkaline extraction resulted in the highest fiber yield, followed by ethanol extraction and hot water extraction. The superior efficiency of ethanol extraction over hot water extraction may be attributed to its effectiveness in disrupting non-fibrous components, particularly in fruits and vegetables with lower starch, intracellular proteins, and polyphenol content [16, 17].

Alkaline extraction is particularly effective due to its ability to disrupt hydrogen and covalent bonds within the plant matrix, facilitating polysaccharide release. The presence of hydroxyl ions in the alkali solution enhances fiber solubilization by breaking hydrogen bonds, thereby increasing extraction efficiency [8]. However, despite its high yield, alkali extraction has significant drawbacks, including potential chemical modifications in fiber structure, the need for pH neutralization, and residual chemical contamination risks.

Although thermal and chemical extraction methods are widely used, they are associated with certain limitations, including high energy consumption, extended extraction time, and the use of toxic solvents. These challenges highlight the need for green extraction techniques, which offer higher efficiency, reduced environmental impact, and improved fiber functionality. Green extraction methods, such as microwave-assisted extraction (MAE), have emerged as promising alternatives that overcome these limitations by reducing processing time, minimizing chemical usage, and improving fiber recovery rates.

3.2.Microwave-assisted extraction of dietary fiber from kinnow by-products

Microwave-assisted extraction (MAE) was employed as a sustainable alternative to conventional methods for the extraction of dietary fiber from kinnow by-products. To optimize

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the extraction conditions, a single-parametric analysis was conducted by varying one factor at a time while keeping the others constant. The process parameters evaluated included microwave power (100–800 W), extraction time (1–15 min), and liquid-to-solid ratio (LSR; 10:1–80:1). The effect of these parameters on the yield of insoluble dietary fiber (ISDF) and soluble dietary fiber (SDF) was assessed for both kinnow peels and pomace as presented in Fig. 1 and 2 respectively.

For kinnow peels, microwave power significantly influenced the extraction efficiency. The yield of both ISDF and SDF increased with power levels up to 400 W, beyond which a decline was observed. The reduction in dietary fiber content at higher power settings (600–800 W) can be attributed to the degradation of polysaccharides and simple sugars due to excessive heat exposure [18]. A similar trend was noted for extraction time, where a peak in dietary fiber yield was observed at 6–9 min, followed by a decline at longer durations. Prolonged microwave exposure likely led to thermal degradation of fiber components, reducing the overall recovery of both ISDF and SDF [19]. The liquid-to-solid ratio also played a crucial role in fiber extraction, with fiber yield increasing as LSR increased up to 50:1, likely due to enhanced mass transfer and improved solubilization of fiber components. However, further increases in LSR beyond this threshold had a negligible impact on extraction efficiency, indicating that an excess solvent volume does not significantly enhance fiber recovery.

<Figure 1 near here>

For kinnow pomace, a similar pattern was observed (Fig. 2), with dietary fiber yield being significantly influenced by microwave power, extraction time, and LSR. The highest fiber yield was obtained at 450 W, LSR of 50:1, and an extraction time of 9 min. Beyond these optimized conditions, fiber yield declined, reinforcing the hypothesis that excessive microwave power and prolonged extraction time lead to degradation of soluble fiber fractions [20]. The effect of LSR followed a similar trend to that of kinnow peels, where fiber yield increased with

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231 increasing solvent volume up to an optimal point but showed no further improvements at higher
232 LSR values.

233 **<Figure 2 near here>**

234 The results indicate that MAE is a highly efficient technique for dietary fiber extraction,
235 offering shorter extraction times, reduced solvent usage, and improved fiber recovery
236 compared to conventional techniques. Unlike hot water and ethanol extraction, which require
237 prolonged heating and multiple rinsing steps, MAE facilitates rapid cell wall disruption through
238 localized heating, enhancing fiber solubilization while minimizing structural degradation [20].
239 These advantages establish microwave-assisted extraction as a superior alternative to
240 conventional methods for dietary fiber extraction from kinnow by-products, supporting its
241 potential for large-scale food and nutraceutical applications.

242 **3.3.Comparison of conventional techniques with MAE**

243 The dietary fiber was extracted from kinnow by-products using various conventional extraction
244 techniques (hot water, alkali, and ethanol extraction) and microwave-assisted extraction
245 (MAE). Among these methods, MAE resulted in the highest dietary fiber yield, including
246 soluble dietary fiber (SDF), insoluble dietary fiber (ISDF), and total dietary fiber (TDF). The
247 detailed comparison of fiber yields obtained using each method is presented in Fig. 3.

248 <Figure 3 near here>

249 The superior performance of MAE can be attributed to its ability to efficiently disrupt plant
250 cell walls through rapid, uniform heating. Unlike conventional thermal treatments, which rely
251 on external heat transfer, microwave energy penetrates the plant matrix, generating localized
252 heating that facilitates better diffusion of fiber components into the solvent. This mechanism
253 enhances fiber solubilization while minimizing thermal degradation of polysaccharides.
254 Additionally, the shorter processing time in MAE reduces exposure to high temperatures,
255 preserving the structural and functional integrity of dietary fiber. The results indicate a

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significant improvement in fiber yield with MAE compared to conventional methods. As observed in Fig. 3, the TDF content in kinnow peels and pomace extracted using MAE was 43.58% and 40.52%, respectively, which is notably higher than those obtained using alkali (34.59% and 28.98%), ethanol (38.42% and 34.34%), and hot water extraction (28.9% and 27.52%). Similarly, MAE-extracted fiber exhibited higher SDF and ISDF contents, demonstrating its effectiveness in dietary fiber recovery. These findings highlight the advantages of microwave-assisted extraction over conventional methods, particularly in achieving higher fiber yields with reduced processing time and minimal chemical usage. The enhanced efficiency of MAE suggests its potential as a sustainable and scalable alternative for dietary fiber extraction from kinnow by-products in food and nutraceutical applications.

3.4.Characterization of dietary fiber

3.4.1. Functional Properties of Dietary Fibers

Functional properties such as water-holding capacity (WHC), oil-holding capacity (OHC), and glucose adsorption capacity (GAC) are critical parameters that determine the technological and nutritional applicability of dietary fibers in food formulations. Fibers with high WHC contribute to water retention, viscosity modification, and prevention of syneresis, thereby improving texture, mouthfeel, and shelf-life stability of food products. Similarly, fibers with high OHC can stabilize emulsified systems, reduce fat absorption during digestion, and improve the sensory quality of low-fat formulations. Meanwhile, GAC is closely related to the hypoglycemic potential of dietary fibers, as fibers with higher glucose-binding capacity can delay glucose diffusion and reduce postprandial glycemic response [21].

In the present study, the functional properties of dietary fibers obtained using different extraction techniques, i.e., hot water, ethanol, alkali treatments, and MAE, were compared (see Table 2). For MAE-extracted fibers, WHC ranged from 7.78 ± 0.10 g/g in pomace to 9.12 ± 0.11 g/g in peel**,** while OHC varied between 4.68 ± 0.06 g/g (pomace) and 5.36 ± 0.07 g/g

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(peel). These values are comparable to those reported for other citrus fibers, such as lemon peel (WHC = 8.24–25.28 g/g, OHC = 3.80–5.00 g/g) [15] and orange fibers (WHC = 8.64–12.28 g/g, OHC = 2.69–3.52 g/g) [22]. Likewise, the swelling capacities of grapefruit, lemon, and lemon albedo fibers (5.16–6.50 mL/g) [23] aligned well with the values observed for kinnow fibers. The relatively higher values obtained for peel compared to pomace across all properties may be attributed to the greater pectin and soluble fiber content in peel, along with a more intact porous microstructure, as observed in SEM analysis. In contrast, pomace fibers consist of a heterogeneous mixture of collapsed parenchymal tissues and vascular residues.

Notable differences emerged when compared with other extraction methods. Hot water extraction produced fibers with moderately higher hydration-related properties, with peel fibers exhibiting WHC of 12.00 ± 0.15 g/g and GAC of 2.60 ± 0.03 mmol/g (10 mmol/L), while pomace fibers showed WHC of 10.00 ± 0.13 g/g. This enhancement can be explained by the solubilization of pectic substances during hot water treatment, which increases fiber swelling and water retention [23]. Ethanol-treated fibers, on the other hand, showed reduced functional properties, with peel WHC dropping to 7.50 ± 0.09 g/g and pomace to 6.80 ± 0.08 g/g, likely due to removal of soluble polysaccharides and phenolics that normally contribute to swelling and glucose binding. Alkali treatment resulted in the most pronounced improvements, with peel fibers showing WHC of 21.50 ± 0.32 g/g, OHC of 6.50 ± 0.08 g/g, and GAC of 2.90 ± 0.04 mmol/g at 10 mmol/L. Pomace fibers also improved substantially (WHC 17.00 ± 0.23 g/g), reflecting the strong de-esterification of pectin and partial removal of lignin and hemicellulose during alkali treatment, which exposes hydrophilic carboxyl and hydroxyl groups and enhances swelling and glucose-binding sites [24].

GAC, another important functional property, reflects the fiber's ability to bind glucose in the intestinal tract, contributing to the potential reduction of postprandial glucose levels. In this study, the GAC of MAE-extracted fibers ranged from 1.34 ± 0.02 to 2.24 ± 0.03 mmol/g at a

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10 mmol/L glucose concentration, increasing to 15.75 ± 0.20 to 19.11 ± 0.25 mmol/g at a 200 mmol/L glucose concentration**,** demonstrating that GAC is directly proportional to glucose concentration. These values are consistent with previous findings on citrus peel fibers, where GAC ranged from 3.68 to 5.14 mmol/g at lower glucose concentrations, rising to 18.5 to 23.3 mmol/g at higher glucose concentrations [25, 26]. This suggests that MAE-extracted kinnow dietary fibers exhibit a strong glucose-binding capacity, further supporting their in vitro hypoglycemic potential.

Overall, the comparative analysis suggests that MAE yields functional properties that are intermediate between those of hot water and alkali extraction. While alkali treatment dramatically improves WHC and GAC by chemically deconstructing cell wall polymers, MAE achieves competitive functionality without the use of harsh reagents, aligning better with sustainable and clean-label processing approaches. Ethanol treatment, although useful for purification, diminishes functional values by reducing soluble fractions. Thus, MAE-extracted kinnow peel and pomace fibers represent a balanced option, offering good WHC, OHC, and GAC while maintaining eco-friendly processing advantages.

<Table 2 near here>

3.4.2. FTIR

The structural characteristics and functional groups of kinnow peels and pomace were analyzed using Fourier Transform Infrared (FTIR) spectroscopy, with the spectra presented in Fig. 4. To assess the impact of microwave-assisted extraction (MAE) on the fiber structure, spectra of the raw materials (peels and pomace before extraction) were compared with those of the corresponding extracted fibers.

In the spectra of raw peels and pomace, a broad absorption band between 3200 and 3400 cm^{-1} was evident, corresponding to O–H stretching vibrations of hydroxyl groups from cellulose, hemicellulose, and pectin, as well as bound water associated with the fiber matrix [27]. This

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band is a hallmark of plant-based cell wall materials and was still visible in the MAE-extracted fibers, although the peak shape became sharper and slightly more defined, indicating disruption of the cell wall and exposure of additional hydroxyl groups. A prominent peak near 2925 cm^{-1}, attributed to C–H stretching of methyl and methylene groups in the polysaccharide backbone [28], was observed in both raw and extracted samples, though with somewhat reduced intensity in pomace fibers compared to peel fibers, consistent with lower lipid and wax content in pomace.

In the carbonyl region, the raw peel showed a clear absorption around 1730–1750 cm^{-1}, assigned to C=O stretching of esterified carboxyl groups in pectin and hemicellulose [15]. This band was weaker in raw pomace and further decreased in intensity after MAE, particularly in pomace fibers, reflecting partial de-esterification and solubilization of pectin during extraction. Meanwhile, the band around 1600–1630 cm^{-1}, which corresponds to C=C stretching vibrations of lignin as well as absorbed water molecules [29], was present in all samples but appeared relatively more pronounced in pomace, consistent with its higher lignin fraction.

The fingerprint region (1200–1000 cm^{-1}) was dominated by strong absorptions attributed to C–O stretching vibrations of glycosidic bonds and pyranose rings of cellulose and hemicellulose. In raw samples, these peaks were broad, while in extracted fibers they appeared sharper and more intense, particularly around 1245 cm^{-1} and 1020–1050 cm^{-1}, confirming the exposure of structural polysaccharides after MAE. A small but characteristic peak near 890 cm^{-1}, associated with β-glycosidic linkages of polysaccharides, was also observed, reaffirming the carbohydrate nature of the matrix [11].

Overall, comparison of raw and MAE-treated samples confirms that the main functional groups characteristic of dietary fibers, i.e., O–H, C–H, C=O, and C–O stretching bands, were preserved after extraction, while certain changes in peak intensity and sharpness reflect the structural modifications induced by MAE. Specifically, reduced ester C=O peaks and enhanced

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carbohydrate fingerprint bands demonstrate the partial removal of soluble pectin fractions and exposure of cellulose/hemicellulose domains. These findings indicate that MAE effectively disrupts the native matrix while maintaining the integrity of key polysaccharide structures necessary for desirable functional properties such as water-holding, oil-holding, and glucose adsorption capacity (Section 3.4.1). The observed profiles are consistent with earlier studies on citrus-derived dietary fibers, where similar spectral characteristics have been reported [15, 22].

<Figure 4 near here>

3.4.3. XRD analysis

The crystalline characteristics of dietary fibers extracted from kinnow peels and pomace using microwave-assisted extraction were evaluated using X-ray diffraction (XRD). Both samples exhibited typical semi-crystalline profiles, with distinct diffraction peaks appearing at approximately $2\theta = 17°$ and 22°, corresponding to the characteristic diffraction peaks of cellulose I, the predominant crystalline form found in plant cell walls. These peaks reflect the ordered arrangement of cellulose chains within the fiber matrix, which is a key contributor to the structural integrity and mechanical properties of dietary fiber [30].

The intensity of these peaks was higher in pomace-derived fibers compared to peel-derived fibers, indicating that pomace fibers exhibit a slightly higher degree of crystallinity. This may be attributed to the higher proportion of structural cellulose in pomace fibers, while peel fibers likely contain more amorphous pectin, hemicellulose, and phenolic compounds, which contribute to the broader background signal and lower overall crystallinity. The presence of broad peaks, particularly in the 10° to 30° range, suggests that both fiber samples contain a mixture of crystalline cellulose and amorphous polysaccharides, which is consistent with the heterogeneous composition of plant-derived dietary fiber [15, 22].

The semi-crystalline nature observed for both samples is a desirable property for functional dietary fiber applications, as the amorphous regions contribute to higher water and oil holding

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capacities by providing more accessible binding sites, while the crystalline regions enhance structural stability, allowing the fiber to retain its functionality during processing [31]. The results indicate that microwave-assisted extraction did not cause extensive disruption of the natural cellulose crystalline regions, preserving the structural characteristics of the extracted fiber, which is advantageous for maintaining the desired functional and textural properties in food applications.

3.4.4. Thermal properties

The thermal properties of dietary fiber concentrates extracted from kinnow peels and pomace using microwave-assisted extraction were evaluated using Differential Scanning Calorimetry (DSC), with separate analyses performed for soluble dietary fiber (SDF) and insoluble dietary fiber (ISDF). The resulting thermograms are presented in Fig. 5. Two distinct thermal events were observed, corresponding to the typical thermal behavior of plant-based dietary fibers.

The first thermal event, an endothermic peak occurring between approximately 60°C and 120°C, corresponds to the release of bound water and initial structural relaxation within the fiber matrix. This transition reflects the disruption of hydrogen bonds between bound water molecules and fiber components, particularly in the more hydrophilic SDF fraction, which exhibited a more pronounced response in this temperature range compared to ISDF. This observation is consistent with the higher water-holding capacity (WHC) measured for SDF (Section 3.4.1) and aligns with previous studies indicating that plant-derived fibers often contain hygroscopic components such as pectins, gums, and mucilages, which contribute to their water-binding properties [1]. This endothermic peak, representing moisture loss and structural relaxation, is a critical indicator of fiber hydration behavior, which influences functional properties such as viscosity, gel formation, and syneresis control in food applications.

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405 The second thermal event, occurring between 300°C and 350°C, corresponds to the exothermic
406 decomposition of polysaccharide components, including cellulose, hemicellulose, and residual
407 pectin or lignin fractions. The ISDF fractions from both peels and pomace exhibited higher
408 thermal stability (delayed decomposition) compared to SDF, a difference attributed to the more
409 ordered, crystalline structure of insoluble fibers relative to the amorphous polysaccharide
410 fractions characteristic of soluble fibers. The slightly higher decomposition temperatures
411 observed for peel-derived fibers (both SDF and ISDF) compared to pomace-derived fibers
412 likely reflects compositional differences, with peel fibers containing a greater proportion of
413 structural polysaccharides and lignin, while pomace fibers may have higher pectin content,
414 which decomposes at lower temperatures.

415 These observations are consistent with reported DSC findings for other plant-derived fibers.
416 For example, jackfruit peel cellulose exhibited an endothermic peak at 323.5°C with an
417 enthalpy change (ΔH) of 0.3152 mW/mg, highlighting the thermal degradation of cellulose at
418 elevated temperatures [32]. Similarly, cellulose powder showed a lower-temperature
419 endothermic peak around 74°C with a ΔH of 72 J/g, attributed to the release of loosely bound
420 water [33]. In contrast, ethyl cellulose displayed an exothermic decomposition peak at
421 334.46°C with an enthalpy of 1.10 kJ/g, reflecting the influence of chemical modification on
422 its thermal behavior [34]. Comparable exothermic peaks were reported for cotton cellulose
423 (315°C, ΔH = 7.65 J/g) and raw banana fibers (350°C, ΔH = 35.7 J/g), with acid-treated banana
424 fibers displaying a much lower endothermic peak at 113.7°C, with a ΔH of 1279 J/g, indicating
425 extensive structural modification caused by acid treatment [35, 36]. These comparisons
426 illustrate that the thermal properties of dietary fibers are highly dependent on source material,
427 structural composition, and pre-treatment methods.

428 The thermal behavior observed for kinnow-derived dietary fibers in this study falls within the
429 general range reported for other plant-derived fibers, reinforcing that microwave-assisted

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extraction preserves the thermal stability of both soluble and insoluble fractions. This thermal resilience, especially in the ISDF fraction, enhances the potential applicability of kinnow dietary fiber in thermally processed food products, such as baked goods, extruded snacks, and meat analogues, where maintaining fiber functionality under heat is essential.

<Figure 5 near here>

3.4.5. SEM analysis

SEM analysis (Figure 6) revealed distinct morphological differences in kinnow peel and pomace before and after microwave-assisted extraction (MAE). Before extraction (Figure 6a, 6c), the peel exhibited a dense, compact, and relatively intact surface matrix, with minimal fibrillation and a continuous epidermal layer, indicative of native cell wall integrity. In contrast, the pomace displayed a more irregular and porous structure, reflecting the partial disruption from prior juice processing. Post-MAE images (Figure 6b, 6d) showed pronounced structural modifications in both substrates, with smoother, flattened lamellar layers in the peel and a more fragmented, open network in the pomace. The removal of soluble constituents and partial breakdown of the cell wall matrix exposed underlying cellulose microfibrils and increased surface roughness, which are characteristic of thermal–mechanical disruption during MAE. These changes confirm the efficiency of MAE in altering plant cell wall architecture, thereby enhancing accessibility to bound bioactive compounds.

<Figure 6 near here>

4. Conclusion

This study compared conventional extraction methods (hot water, ethanol, and alkali extraction) with microwave-assisted extraction for recovering dietary fiber from kinnow peels and pomace, and evaluated their functional, structural, and thermal properties. Among the techniques, MAE demonstrated superior performance, yielding higher dietary fiber content with enhanced soluble dietary fiber recovery compared to conventional methods. Dietary fiber

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extracted using MAE exhibited higher water-holding capacity, oil-holding capacity, and glucose adsorption capacity, indicating its potential application as a functional ingredient for improving moisture retention, texture, and glycemic control in food products. FTIR and XRD analyses confirmed that MAE preserved key functional groups and maintained the semi-crystalline structure of the fibers. DSC analysis showed that the extracted fibers possessed good thermal stability, with ISDF exhibiting greater thermal resistance than SDF. Overall, microwave-assisted extraction proved to be an efficient, sustainable, and scalable technique for extracting high-quality dietary fiber from kinnow by-products, making it suitable for incorporation into functional foods and nutraceuticals, while supporting the valorization of citrus processing waste. Future research should focus on optimizing process parameters for industrial-scale applications, evaluating bioavailability and sensory characteristics, and exploring the incorporation of these fibers into a wider range of functional food products.

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5. Declarations

1. **Author Contributions:** Investigation, Methodology, and Formal Analysis (S.K.), Writing- original draft (S.K.), Writing- reviewing and editing (S.K., V.S., P.S.P., H.K.C.), Resources and Supervision (P.S.P., H.K.C.)
2. **Data Availability Statement:** All data generated or analysed during this study are included in the manuscript. No new crystallographic data was generated. This study did not involve any human participants.
3. **Conflict of Interest:** Authors declare no known competing interests
4. **Funding:** No funding was received for this research
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617 **Table 1: Comparison of Extraction Methods**

Extraction Method	Solvent Used	Temperature (°C)	Time (min)	Centrifugation (rpm/min)	Advantages	Limitations
Hot Water Extraction (HWE)	Water	100	5	5000 / 10	No chemical solvent, simple	High temperature may degrade polysaccharides
Ethanol Extraction (EtOH-E)	80% Ethanol	Room Temp	~10	5000 / 10	Effective for sugar removal	Requires solvent recovery
Alkali Extraction (NaOH-E)	NaOH (50 mM)	45	30	5000 / 30	High fiber yield, breaks cell walls	May alter fiber composition
Microwave-Assisted Extraction (MAE)	Water	50	9	5000 / 15	Shorter extraction time, better yield	Equipment required

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621 **Table 2: Functional properties of dietary fiber concentrates from kinnow peel and**

622 **pomace obtained using different extraction methods**

Extraction Method	WHC (g/g)	OHC (g/g)	GAC (mmol/g) at 10 mmol/L	GAC (mmol/g) at 200 mmol/L
Kinnow Peels				
Hot water	12.00 ± 0.15	5.50 ± 0.07	2.60 ± 0.03	20.00 ± 0.28
Ethanol	7.50 ± 0.09	4.50 ± 0.06	2.00 ± 0.03	17.50 ± 0.22
Alkali	21.50 ± 0.32	6.50 ± 0.08	2.90 ± 0.04	23.00 ± 0.30
MAE	9.12 ± 0.11	5.36 ± 0.07	2.24 ± 0.03	19.11 ± 0.25
Kinnow Pomace				
Hot water	10.00 ± 0.13	5.00 ± 0.06	1.70 ± 0.02	18.00 ± 0.23
Ethanol	6.80 ± 0.08	4.00 ± 0.05	1.20 ± 0.02	14.50 ± 0.19
Alkali	17.00 ± 0.23	6.00 ± 0.08	1.90 ± 0.03	19.50 ± 0.25
MAE	7.78 ± 0.10	4.68 ± 0.06	1.34 ± 0.02	15.75 ± 0.20

623 The values are presented as mean ± SD

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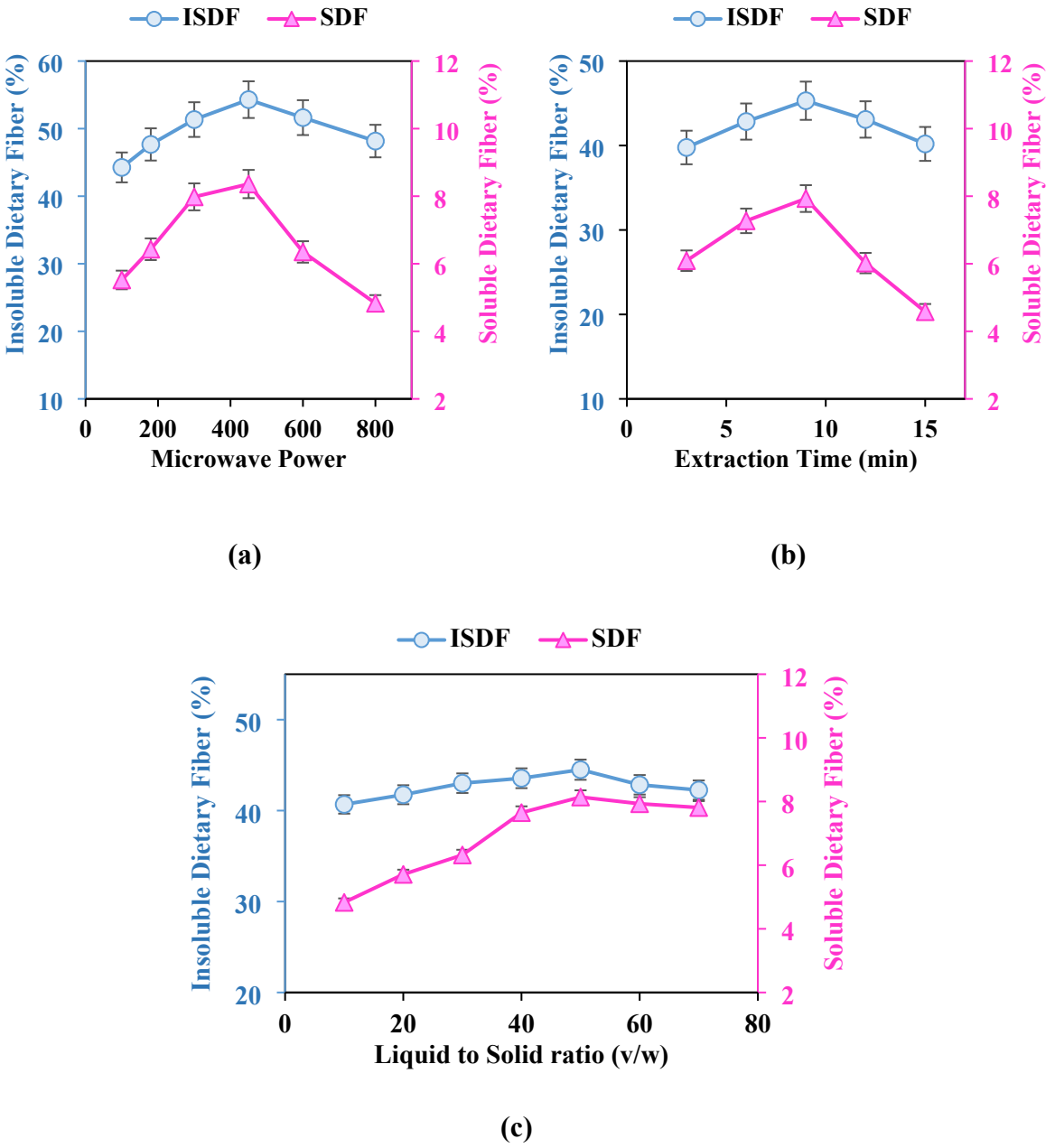

627 **Figure 1: Effect of a) microwave power, (b) extraction time, and (c) liquid to solid ratio**

628 **on yield of dietary fiber from kinnow peels during microwave-assisted**

629 **extraction**

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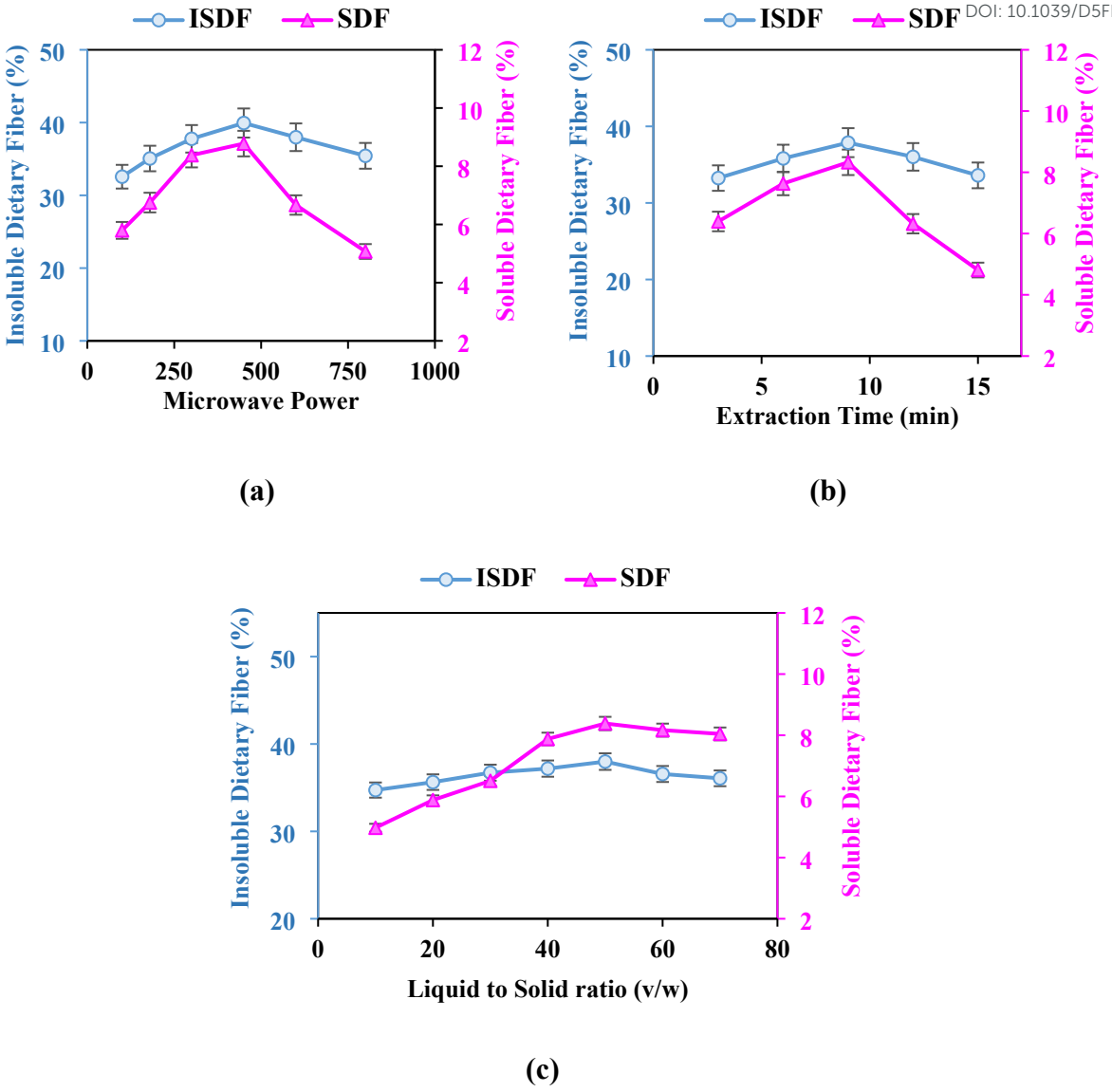

631 **Figure 2: Effect of a) microwave power, (b) extraction time, and (c) liquid to solid ratio**
632 **on yield of dietary fiber from kinnow pomace during microwave-assisted**
633 **extraction**

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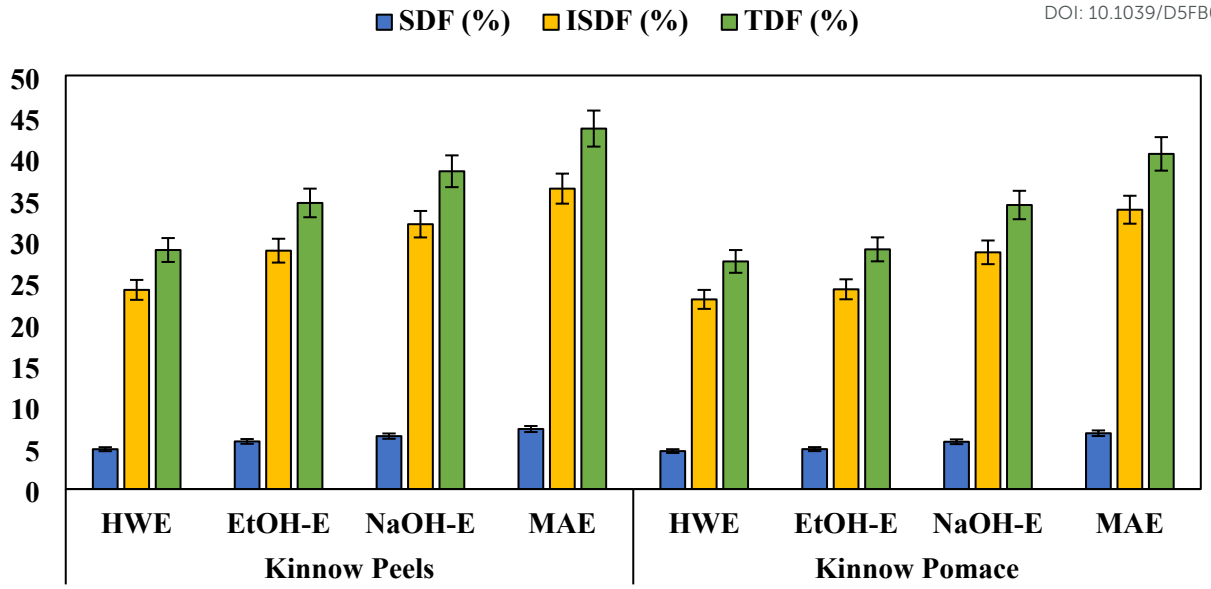

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636 (Values are presented as mean ± percentage error of triplicate analyses)

637 **Figure 3: Comparison of different techniques for the extraction of dietary fiber from**

638 **kinnow by-products**

639 Here, HWE =Hot water extraction; NaOH-E =Alkali extraction; EtOH- E = Ethanol extraction;

640 MAE = Microwave-assisted Extraction; SDF= Soluble Dietary Fiber; ISDF= Insoluble Dietary

641 Fiber; TDF = Total Dietary Fiber

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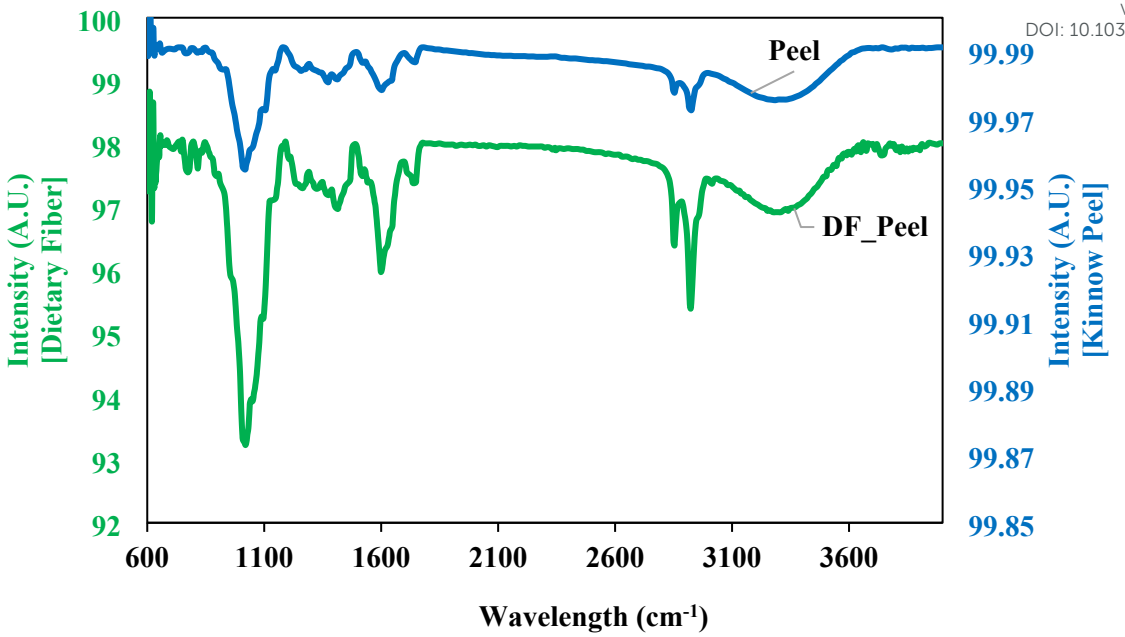
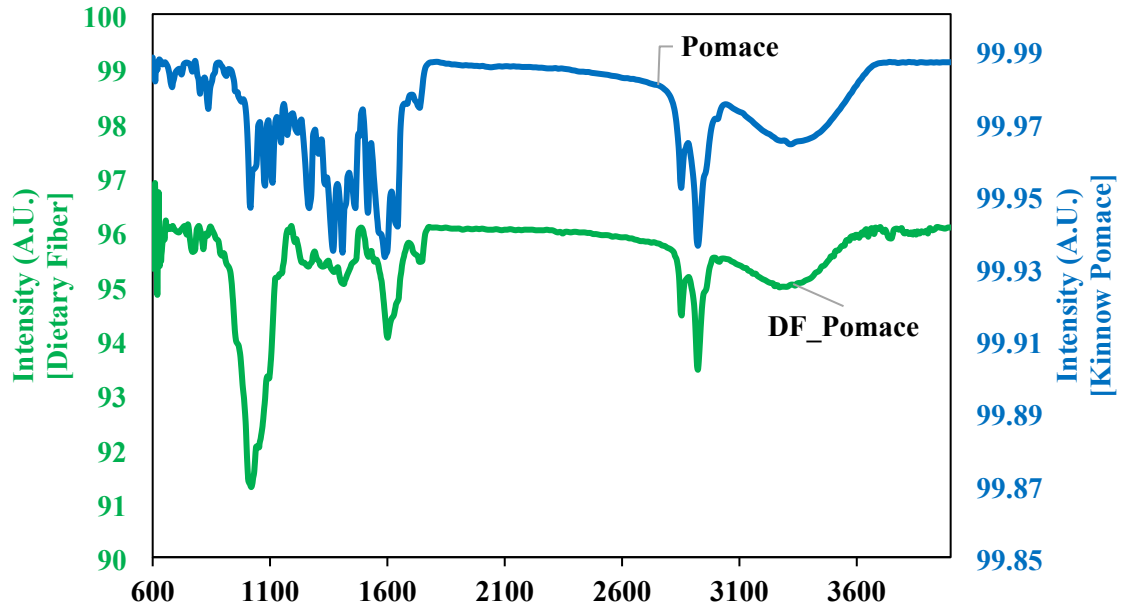


(a)



(b)

643 **Figure 4: FTIR plot of (a) Kinnow peel and its Dietary fiber, and (b) Kinnow pomace and**

644 **its dietary fiber, extracted using microwave-assisted extraction.**

645

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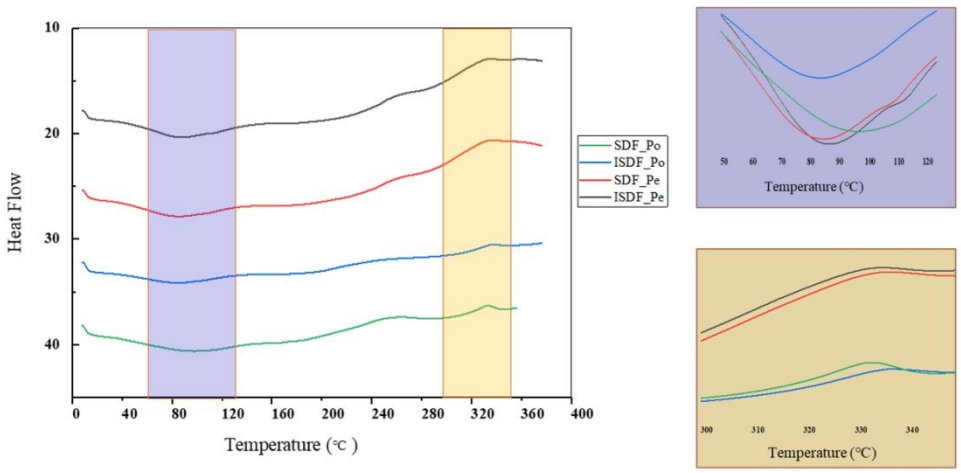

646 **Figure 5: DSC plot of dietary fiber obtained using microwave-assisted extraction of**
647 **kinnow byproducts.**

648 Here Po= pomace, Pe= Peels, SDF= Soluble dietary fiber, ISDF= Insoluble dietary fiber

649

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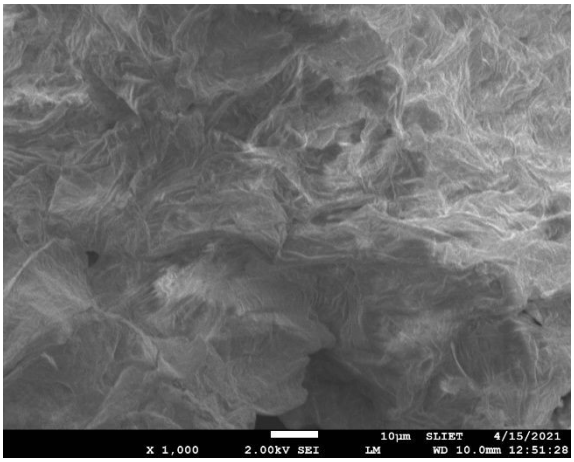
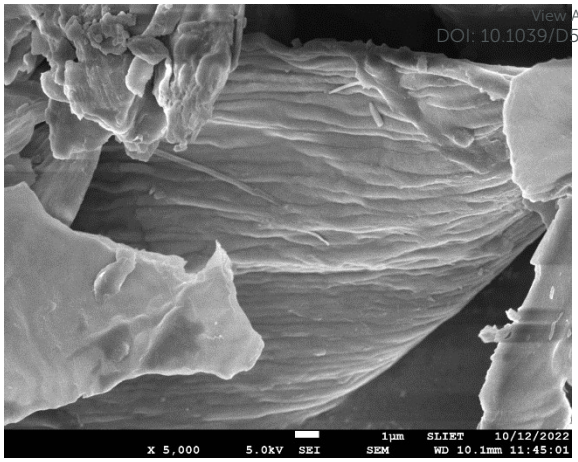
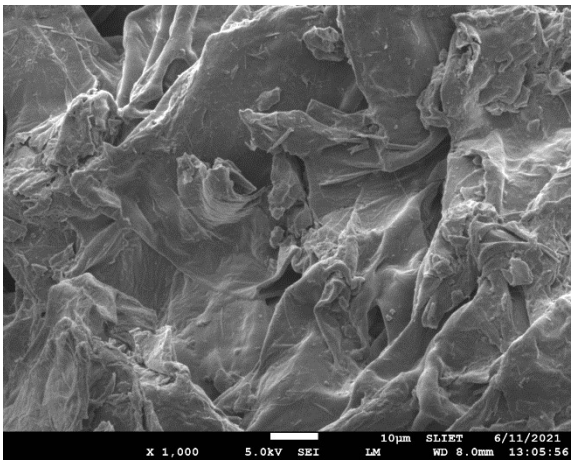


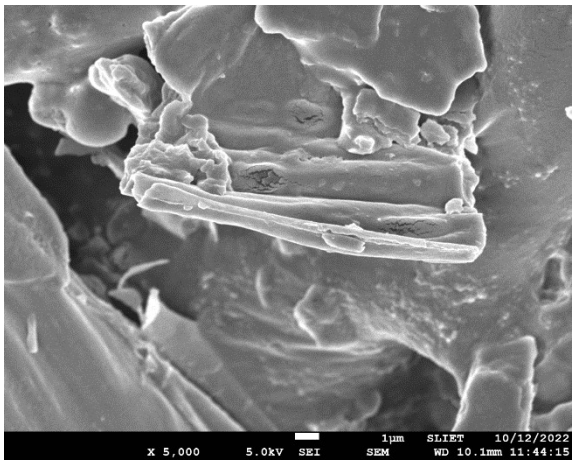
Kinnow Peel (Before)



Kinnow Peel (After)



Kinnow Pomace (Before)



Kinnow Pomace (After)

650 **Figure 6: SEM images of Kinnow Peels and Kinnow pomace before and after MAE**

651 **extraction**

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Data availability statement

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All data generated or analysed during this study are included in the manuscript. No new crystallographic data was generated. This study did not involve any human participants.

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