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Fruit peels valorization into cutin-rich extracts

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ABSTRACT Fruit peels can be valorized as a source of cutin-rich materials with useful functional properties. This study compared cutin-rich extracts (CREs) from tomato, watermelon, honeydew, and papaya peels obtained through alkaline hydrolysis. The extracts were characterized for extraction yield, structure, thermal stability, water sorption, hydrophobicity, total phenolic content, and antioxidant activity. CRE properties varied among the fruit sources. FTIR spectra showed functional groups associated with cutin in all samples. Tomato CRE had the lowest water sorption and better thermal stability, whereas watermelon CRE showed similar hydrophobicity and higher antioxidant activity. Watermelon CRE recorded the highest total phenolic content (71.23 ± 0.91 mg GAE/g), DPPH radical scavenging activity (19.41 ± 0.80%), and ABTS radical scavenging activity (88.64 ± 0.17%). The results show that fruit peel source affects CRE properties and supports their potential as sustainable bio-based materials for applications in packaging, coatings, and other material systems.

KEYWORDS: Cutin; Fruit peels; Waste valorisation; Hydrophobicity; Antioxidant activity
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INTRODUCTION

Increasing fruit consumption generates large amounts of fruit waste, particularly peels and seeds, which can contribute to environmental problems (Guo *et al*., 2021; Rahim *et al*., 2022). Valorising these wastes into functional materials can help reduce their environmental impact. Fruit peels are particularly valuable as they contain a protective cuticle rich in cutin, a natural biopolymer with potential functional applications. The plant cuticle is a hydrophobic layer with an amorphous polyester structure that protects against water loss, microbial invasion, and environmental stress (Mroczkowska *et al*., 2024; Singha *et al*., 2021). Cutin, which accounts for 40–85% of the cuticle, contributes to its protective function through its hydrophobicity, thermal stability, and mechanical strength (Eslami *et al*., 2024; Li *et al*., 2024). These characteristics make cutin a promising bio-based material for various applications because of its biodegradability and antioxidant properties (Mroczkowska *et al*., 2024; Simões *et al*., 2023).

Most previous work has focused on tomato-derived cutin (Arrieta-Baez *et al*., 2024; Eslami *et al*., 2024; Simões *et al*., 2023), while other fruit peel sources and their properties remain less studied. In this study, fruit peel wastes from tomato, papaya, honeydew, and watermelon were valorised into CREs. These fruits were selected because their cuticle composition differs, which may influence the properties of the extracted material (Mellinas *et al*., 2022). Tomato peel was included as a benchmark due to its extensive use in previous cutin related studies (Mellinas *et al*., 2022; Singha *et al*., 2021). The CREs were analysed for structural, thermal, moisture-related, and antioxidant properties to evaluate their functional characteristics and potential as sustainable bio-based materials.

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MATERIALS AND METHODS

Materials

Tomato, watermelon, papaya, and honeydew were obtained from a market in Senawang, Negeri Sembilan. Fruits with similar maturity and no visible defects were selected. Monosodium phosphate, hydrochloric acid and sodium hydroxide were obtained commercially from Thermo Fisher Scientific (Leicestershire, UK).

Extraction and Yield

Cutin-rich extracts (CREs) were obtained from fruit peels using alkaline hydrolysis (Tedeschi *et al*., 2018) with modifications. The peels were washed to remove remaining pulp, cut into pieces, and dried at 40 °C for 24 h. After drying, the peels were ground and passed through a 500 µm sieve. Alkaline hydrolysis was carried out using 0.5 M NaOH at a solid-to-liquid ratio of 1:10 at 100 °C for 6 h. After cooling and filtration, the solution was acidified to pH 3 using 3 M HCl. The precipitate was centrifuged four times at 10,000 rpm for 20 min at 25 ± 2 °C and washed with distilled water after each centrifugation. Finally, the CREs were freeze-dried at −50 °C (≤10 Pa) for 48 h and stored at 4 °C until further use. Extraction yield was calculated gravimetrically using Equation (1).

$$\text{Yield} = \frac{m_1}{m_2} \text{x} 100 \quad (1)$$

where m_1 is the extracted CRE weight and m_2 is the initial dry peel weight.

Fourier Transform Infrared Spectroscopy

The FTIR spectra of the CREs were analysed using an FTIR–ATR spectrometer (Nicolet iS10, Thermo Scientific, Madison, USA) from 4000 to 500 cm^{-1}. Each spectrum was recorded using 64 scans at a resolution of 4 cm^{-1} (Mellinas *et al*., 2022). The spectra were processed using Omnic 8 software, and the bands were identified based on previous studies.

Thermogravimetric Analysis

TGA was performed using a TGA Q500 thermal analyser (TA Instruments Inc., New Castle, USA) (Manrich *et al*., 2017). The samples were subjected to heating from ambient temperature to 800 °C at a rate of 10 °C/min in synthetic air (80% N_2 and 20% O_2), with a flow rate of 60 mL/min.

Water Sorption

Water sorption of the CREs was determined using a sealed chamber maintained at 98% relative humidity with an oversaturated monosodium phosphate solution. Samples were exposed at room temperature for approximately 10 h until equilibrium. Water sorption was calculated from the change in sample weight following Matas *et al*. (2004) with modifications using the following Equation (2).

$$\text{Water sorption (\%)} = \frac{(w1-w0)}{w0} \text{ x } 100 \quad (2)$$

where w_0 and w_1 are the weight of extract before and after absorbing moisture.

Hydrophobicity

The hydrophobicity of the CRE samples was evaluated following Yadav *et al*. (2014) with modification. Dried CRE was dissolved in ethanol (8.0 µg/µL), and 250 µL was used to coat Whatman No. 1 filter paper discs (78.5 mm^2). The coated discs were weighed before and after coating to determine CRE loading. Five replicates were used. The discs were then immersed in distilled water for 5 min and weighed and hydrophobicity was determined by using Equation (3).

$$\text{\% hydrophobicity} = \frac{W1-W2}{W1} \text{ X100} \quad (3)$$

where w_1 and w_2 correspond to the water absorbed by the untreated and CRE-loaded paper discs (g).

Total Phenolic Content

Total phenolic content (TPC) was determined using the Folin–Ciocalteu method originally described by Singleton and Rossi. (1965) with slight modifications adopted from Perozzi *et al*. (2021). CRE (25 mg) was extracted in 3 mL of ethanol, and 0.5 mL of the resulting extract was combined with 2.5 mL of Folin–Ciocalteu reagent (1:10, v/v). After 5 min, 2 mL of 7.5% sodium carbonate was added. The reaction mixture was incubated in the dark for 2 h before measuring the absorbance at 740 nm. A gallic acid standard curve (0–1000 ppm) was prepared for quantification, and TPC was reported as mg GAE/g sample.

DPPH Radical Scavenging Activity

DPPH radical scavenging activity was determined following Nor Adilah *et al*. (2018) with modification. A 0.05 mL extract was combined with 1.95 mL of 0.1 mM DPPH prepared in ethanol. After vortexing, the reaction mixture was kept in the dark for 30 min. Absorbance was then recorded at 517 nm using a UV–Vis spectrophotometer (Shimadzu UV-1601, Japan). DPPH radical scavenging activity (%) was calculated using Equation (4).

$$\text{DPPH} = \frac{(\text{Abs DPPH} - \text{Abs extract})\text{x } 100}{\text{Abs DPPH}} \quad (4)$$

where Abs DPPH is the absorbance of the DPPH solution and Abs extract is the absorbance of the sample extract.

ABTS Radical Scavenging Activity

The ABTS radical scavenging assay was based on the method of Re *et al*. (1999), with modifications adopted from Sui Chin *et al*. (2017). A 7 mmol/L ABTS solution containing 140 mmol/L potassium persulfate was allowed to react in the dark at 25 °C for 12–16 h. The solution was then adjusted with 50% ethanol until an absorbance of 0.70 ± 0.02 at 734 nm was reached. The extract and ABTS solution were combined at a ratio of 0.1:1.9 (v/v) and kept in the dark for 30 min. Absorbance was measured at 734 nm, and ABTS radical scavenging activity was calculated using Equation (5).

$$\text{ABTS} = \frac{(\text{Abs control} - \text{Abs sample extract})\text{ x } 100}{\text{Abs control}} \quad (5)$$

where Abs control is the control absorbance and Abs sample is the sample absorbance.

Statistical Analysis

All analyses were performed in triplicate unless otherwise stated. Data were analysed using one-way analysis of variance (ANOVA) with Minitab statistical software (Version 18, State College, PA, USA). Significant differences were determined using Tukey's test at $p < 0.05$.

RESULTS AND DISCUSSION

Yield and Moisture-Related Properties

Alkaline hydrolysis was selected as an established approach for cutin depolymerization and recovery, as previously demonstrated for cutin extraction from tomato peel (Simões *et al*., 2023). Although ultrasound-assisted extraction offers shorter extraction times (Mellinas *et al*., 2022), it requires specialized ultrasonic equipment and controlled operating conditions, while enzymatic cutin isolation (Gómez-Patiño *et al*., 2020) may involve multiple treatment and solvent-based dewaxing steps, making the process more complex and time-consuming. Therefore, alkaline hydrolysis provides a relatively straightforward approach for the comparative recovery of CREs from different fruit peel sources. The extraction yield of CREs ranged from 15.45% to 22.33% (Table 1). Tomato had a yield of 22.33 ± 0.44%, which was within the range reported in previous studies (Cifarelli *et al*., 2019). Watermelon and honeydew showed similar yields to tomato, while papaya had a significantly lower yield. This variation may reflect species differences in fruit peel composition (Suleria *et al*., 2020), particularly differences in polysaccharide components, which may influence hydrolysis efficiency and subsequent cutin recovery (Mellinas *et al*., 2022).

In addition to extraction yield, the moisture-related properties of the CREs were evaluated through water sorption and hydrophobicity (Table 1). Water sorption was lowest in tomato (8.50 ± 1.96%), followed by honeydew (16.46 ± 2.57%), watermelon (20.38 ± 0.72%), and papaya (30.90 ± 1.44%). These differences may be associated with variations in hydrophilic polysaccharide components, which contribute to water interaction within plant cuticles (Heredia *et al*., 2024). The notably higher water sorption of papaya CRE may partly reflect its polysaccharide composition, as papaya peel has been reported to be rich in pectic polysaccharides (Koubala *et al*., 2014; Maran and Prakash, 2015). Furthermore, papaya peel has been reported to have a higher calcium pectate-producing potential than watermelon, indicating differences in pectic components between these fruit sources *(Begum et al*., 2017). These compositional differences may partly contribute to the greater water sorption observed for papaya CRE.

While water sorption measures long-term moisture absorption, hydrophobicity shows the ability of the surface to repel water, as shown in Table 1. Tomato, watermelon, and honeydew showing similar values, while papaya had the lowest value, consistent with its higher water sorption. This difference may be explained by surface wax affecting hydrophobicity, while internal porosity and polysaccharide composition affect water sorption (Philippe *et al*., 2020). Despite higher water sorption, watermelon and honeydew showed similar hydrophobicity to tomato, suggesting their potential as alternative sources of CRE.

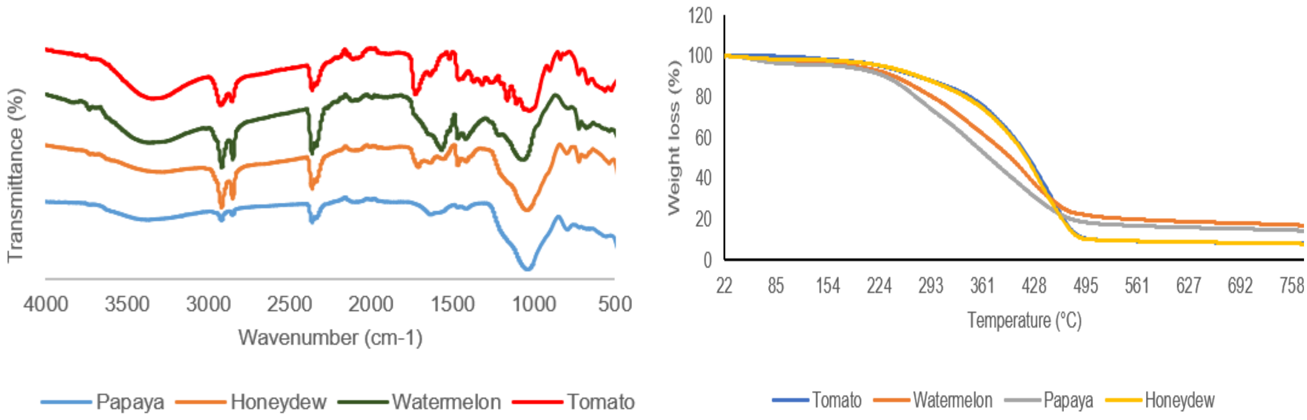
Table 1. Properties of cutin-rich extracts from different fruit peels.

Cutin-rich extract	Yield (%)	Water sorption (%)	Hydrophobicity (%)	TPC (mg GAE/g)	DPPH (%)	ABTS (%)
Tomato	22.33 ± 0.44[a]	8.50 ± 1.96[c]	17.99 ± 2.86[a]	48.21 ± 1.13[c]	16.61 ± 0.41[c]	85.81 ± 0.21[c]
Watermelon	21.07 ± 0.84[a]	20.38 ± 0.72[b]	17.50 ± 1.93[a]	71.23 ± 0.91[a]	19.41 ± 0.80[a]	88.64 ± 0.17[a]
Honeydew	20.59 ± 0.58[a]	16.46 ± 2.57[b]	14.51 ± 0.66[a]	26.15 ± 0.90[d]	18.27 ± 0.27[b]	76.80 ± 0.21[d]
Papaya	15.45 ± 0.81[b]	30.90 ± 1.44[a]	6.23 ± 2.86[b]	54.30 ± 1.35[b]	13.05 ± 0.57[d]	87.77 ± 0.18[b]

Values are given as mean ± standard deviation (n=3). Means in the same column with different superscript letters are significantly different ($p < 0.05$).

Structural and Thermal Properties

FTIR analysis was used to identify the functional groups in the CREs (Figure 1a). All samples showed a broad band at 3600–3000 cm^{-1}, corresponding to O–H stretching from polysaccharides and residual carboxylic acids (Mellinas *et al*., 2022). Bands at 3000–2800 cm^{-1} were assigned to CH_2 stretching from the aliphatic chains of fatty acids, waxes, and cutin (Eslami *et al*., 2024; Mellinas *et al*., 2022). Bands between 1600 and 1750 cm^{-1} were related to C=O groups, while the band around 1100 cm^{-1} was associated with C–CO–O groups (Ben *et al*., 2020). Bands at 1600–1450 cm^{-1} were related to aromatic C=C groups, while other bands were associated with phenolic and flavonoid groups (Manrich *et al*., 2017). The FTIR spectra showed characteristic functional groups associated with cutin, including aliphatic chains and ester groups, in all CREs.



(a) (b)

Figure 1. Characterization of cutin-rich extracts (CREs) from tomato, watermelon, honeydew, and papaya peels: (a) Fourier transform infrared (FTIR) spectra and (b) thermogravimetric analysis (TGA) curves

The thermal stability of CREs was evaluated using TGA by monitoring weight loss as a function of temperature *(Eslami et al*., 2024; Singha *et al*., 2021), as shown in Figure 1b. All samples exhibited an initial weight loss below 150 °C, primarily attributed to the evaporation of moisture and other volatile constituents. Among them, tomato CRE demonstrated the greatest thermal stability, with its main degradation phase occurring between 200 and 400 °C due to the breakdown of its polymer structure, in agreement with previous reports (Eslami *et al*., 2024; Mellinas *et al*., 2022). Watermelon showed moderate thermal stability, with major degradation between 250 and 400 °C, consistent with previous findings by Chaudhari & Singhal, (2015). Honeydew showed a similar degradation pattern to tomato, while papaya degraded earlier and had the lowest thermal stability.

Antioxidant Properties

Antioxidant activity is an important functional property of CREs because phenolic compounds can help reduce oxidative reactions (Nabilah *et al*., 2021). The TPC results for the CREs are presented in Table 1. Watermelon CRE showed the highest TPC (71.23 ± 0.91 mg GAE/g), followed by papaya, tomato, and honeydew. The high TPC of watermelon is consistent with its reported phenolic content. (Mellinas *et al*., 2022; Neglo *et al*., 2021).

Antioxidant activity was assessed using DPPH and ABTS assays, which vary in their responsiveness to different antioxidant compounds (Neglo *et al*., 2021). As shown in Table 1, among the samples tested, watermelon exhibited the highest DPPH scavenging activity (19.41 ± 0.80%), followed by honeydew, tomato, and papaya. The higher activity of watermelon was consistent with its high TPC, as phenolic compounds contribute to antioxidant activity, in agreement with previous findings on watermelon peel (Neglo *et al*., 2021; Suleria *et al*., 2020). Similarly, watermelon showed the highest ABTS activity (88.64 ± 0.17%), followed by papaya, tomato, and honeydew (Table 1), consistent with its high TPC and DPPH activity, as well as previous studies on watermelon peel (Mellinas *et al*., 2022; Neglo *et al*., 2021). Papaya showed higher ABTS than DPPH activity, suggesting that the assays respond differently to antioxidant compounds.

CONCLUSION

The properties of CREs varied depending on the fruit peel source, with tomato showing better moisture resistance and thermal stability, while watermelon showed stronger antioxidant activity with similar yield and hydrophobicity. Watermelon peel is a promising alternative source of CRE for sustainable bio-based applications, although further studies are needed to evaluate its composition and potential applications.

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