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*Research article*

## **Sustainable valorization of fruit waste: Extraction, characterization, and application of pectin in jam**

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**Abstract:** In this study, we aimed to extract and evaluate pectin from apple (*Malus domestica*), lemon (*Citrus limon*), and lime (*Citrus aurantiifolia*) peels using microwave-assisted extraction (MAE) and ultrasound-assisted extraction (UAE) techniques with different extractants. The effects of microwave power and irradiation time on pectin yield were investigated in MAE. The impacts of temperature and sonication time were assessed for UAE. The extracted pectin was subsequently used to prepare java plum (*Syzygium cumini* L.) jam. The highest pectin yield (18.25%) was obtained from lime peel using MAE at 500 W for 3 minutes, while UAE yielded 11.1% from lemon peel at 65 °C for 45 minutes. The extracted pectin was characterized as high-methoxy pectin, with a degree of esterification ranging from 50.13% to 61.63%. Fourier transform infrared spectroscopy confirmed its structure, showing a peak at 1700–1770 cm<sup>-1</sup> for polygalacturonic acid and characteristic fingerprints in

the 1600–1750  $\text{cm}^{-1}$  region. Scanning electron microscopy revealed that UAE-derived apple peel pectin had a ruptured surface, whereas MAE-derived lime peel pectin exhibited a porous structure, indicating a higher degree of hydrolysis. Jams prepared with the pectin showed total soluble solids of 65.1°, 67.2°, and 68.1° Brix for apple pectin jam (APJ), lemon pectin jam (LPJ-a), and lime pectin jam (LPJ-b), respectively. The LPJ-b exhibited the highest viscosity (75.1 cP) and received the best sensory acceptability score. MAE was more effective than UAE, providing a higher yield in a shorter time. Furthermore, the jam formulated with MAE-extracted lime pectin demonstrated superior physicochemical and organoleptic properties.

**Keywords:** pectin; fruit waste; peel; ultrasound-assisted extraction; microwave-assisted extraction; jam; scanning electron microscopy

## 1. Introduction

Agri-business generates a significant amount of waste during its production process. In response to this global challenge, the United Nations has implemented Sustainable Development Goals (SDGs) that aim to cut agro-industrial waste by 50%. Consequently, there has been increased interest in using agro-based waste to recover high-value products like pectin. Pectin is a complex polysaccharide made up of  $\alpha$ -(1-4)-linked monosaccharides. It originates in plant cell walls as protopectin, which is insoluble in ne-way analysis of variance (ANOVA) and is converted into water-soluble pectin through hydrolysis. Due to its functional properties, pectin is commonly used in jams and jellies as a gelling agent. Similarly, in baked goods, it serves as a fat substitute due to its oil-binding capacity. Moreover, adding pectin to functional beverages enhances their flavor, mouthfeel, and overall acceptability. Pectin is a major component of waste from fruits; for instance, about 60-70% of citrus peel is produced after citrus fruit processing. Indeed, citrus peel is a rich source of high-methoxyl pectin, with common sources including sweet orange, mandarin, bitter orange, lemon, grapefruit, and lime [1]. Thus, after separating the juice from the fruit, a large amount of peel is generated as waste [2].

Structurally, commercial pectin is composed of  $\geq 65\%$  galacturonic acid monomers and is classified as high-methoxy (HMP) or low-methoxyl pectin (LMP) based on its degree of esterification (DE). HMP has a DE of  $>50\%$ , while LMP has a DE of  $<50\%$ . Within the plant cell wall, pectin comprises approximately 65% of the structure, and rhamnogalacturonan II constitutes approximately  $>30\%$  of pectin content. It has a more complex structure than rhamnogalacturonan I [3]. Critically, the degree of esterification directly affects the gelling capability of pectin, a property that is gaining much attention [4]. High methoxy pectin forms a gel at an acidic pH ( $\sim 3.8$ ) in the presence of high sugar content ( $>55\%$ ), and these gels are often more stable than those of LM pectin. Alternatively, another gel-forming mechanism for LMP occurs with the addition of divalent cations in an acidic medium ( $\text{pH} < 3.0$ ) [5].

Apple (*Malus domestica*) is one of the most cultivated fruits globally and belongs to the family Rosaceae. Apple peel is an abundant source of pectin [6]. Apple pomace, a waste product from cider industries consisting of apple peels, seeds, and flesh, is a key source. It has been reported that almost 57–60% of pectin can be extracted from 'Granny Smith' apple peel [7]. Here, we uniquely utilize local varieties of the apple for pectin extraction.

Many researchers have investigated the extraction of pectin from other plants, such as

pomegranate, passion fruit, citrus, and melon peels [8]. However, conventional extraction methods often require high energy input and produce large amounts of wastewater. Therefore, novel technologies are being used for the extraction of pectin from fruit waste. Among them, the most promising alternative to conventional extraction methods is ultrasound- and microwave-assisted extraction. Microwave-assisted extraction (MAE) has been widely utilized for pectin extraction from apple pomace, orange peel, passion fruit rind, and pistachio husk [9]. Many studies reported that MAE is the best alternative to conventional extraction, as it requires the shortest processing time and gives a high yield [10]. To illustrate, passion fruit peel pectin, which is previously characterized as HMP, can be extracted using MAE. The combination of MAE with alkaline extraction significantly affects the yield and recovery of pure pectin. Specifically, an extraction time of 7 min at 376 W microwave power gave the highest (14.2%) recovery rate [11]. Similarly, the sugar industry produces sugar beet flakes as a by-product, which could be utilized as a source of pectin. Researchers employed MAE and pulsed ultrasound-assisted extraction alongside commercial methods to recover pectin from this industrial by-product and observed a higher content of galacturonic acid (85 g/100 g) in the final product. One of the major applications of pectin is in jam production. Jam is a popular food often consumed for breakfast with bread or chapati. It has a low cost and high organoleptic appeal. Moreover, it is prepared from a single fruit or a combination of more than two fruits. According to standards, it must contain at least 45% fruit pulp and 68% total soluble solids (TSS). These properties contribute to its long shelf life and year-round availability [12].

*Syzygium cumini* L. (java plum) is a large, versatile tropical tree belonging to the family Myrtaceae, commonly known as jamun in Pakistan. It can reach a height of 30 meters and has a life span of around 100 years. Native to Southeast Asia, from Pakistan to Australia, the plant has huge economic importance, as not only are its fruits useful, but also its seeds, leaves, and bark possess medicinal properties. Traditionally, it has been utilized to cure cough, constipation, diarrhea, and cardiovascular diseases (CVD). The deep color of java plum fruit indicates that it is a good source of anthocyanins and other micronutrients, such as vitamins and minerals [13].

Several studies demonstrate the successful application of waste-derived pectin. For example, pectin extracted from orange peel using HCl yielded about 15%. When the extracted pectin was utilized in jam making, it received a sensory score of 7.1 for overall acceptability. Similarly, pectin isolated from mango peel had a yield that ranged between 14% and 28% under different conditions. When used in pineapple jelly production, it scored 7.71 points for taste and 7.5 for overall acceptability [14]. Beyond food, pectin from dried citrus peel has been utilized as a binding agent in the pharmaceutical industry.

Therefore, we hypothesized that MAE and UAE could yield a higher amount of pectin within the shortest extraction time from underutilized fruit wastes. Specifically, we focus on the valorization of local varieties of apple (Kala Kulu; *M. domestica*), lemon (Kagzi Nimbu; *Citrus limon*), and lime (Kaghzi Lime; *Citrus aurantiifolia*), investigating how regional growing conditions influence pectin yield and quality. The valorization of this waste can be beneficial for bioactive extraction. The effect of different extraction methods and their conditions will be investigated to maximize the yield and to evaluate any structural changes. The locally extracted pectin will then be used to formulate and optimize Java plum jam to investigate its functional properties. Overall, by focusing on regional resources and traditional applications, we aim to promote sustainability, support local agriculture, and fill gaps in knowledge.

## 2. Materials and methods

### 2.1. Materials

The apples (Kala Kulu; *M. domestica*), lemons (Kagzi Nimbu; *Citrus limon*), and limes (Kaghzi Lime; *Citrus aurantiifolia*) used in the experiment were procured from a local market in Faisalabad, Punjab, Pakistan. After being removed, the peels were soaked in a water bath at ~90-95 °C for 4-5 min for thermal enzyme inactivation.

### 2.2. MAE procedure

Dried peel powder (3 g) was mixed with 90 mL of 0.5 M hydrochloric acid (HCl) as the extraction solvent, at a solid-to-solvent ratio of 1:30 (g/mL). HCl was selected as a strong mineral acid known for high pectin extraction efficiency, while citric acid was chosen as a weak organic acid suitable for food-grade applications. The mixture was placed in a microwave oven (ME711K, Suwon, South Korea). Extractions were performed at microwave powers of 360 W and 500 W for irradiation times of 1 min and 3 min, respectively. After irradiation, the solution was allowed to cool to ambient temperature and then centrifuged at 1300 rpm for 25 min. The supernatant was then precipitated with a 1:1 volume of 90% ethanol and left overnight at 4 °C. The resulting pectin precipitate was separated by vacuum filtration and washed with ice-cold 70% ethanol (v/v) to remove impurities. The purified pectin was dried in an oven (redLINE RF 115, Deutschland, Germany) at 60 °C. The dried pectin was weighed, stored in a desiccator, and the yield was calculated. The same extraction procedure was repeated using a 0.5 M citric acid solution as the extractant for all samples [2].

### 2.3. UAE procedure

Pectin was extracted using a modified method described by Gharibzahedi et al., [15]. Three g of powdered fruit peel was suspended in 30 mL of distilled water. The pH of the suspension was adjusted to 2.0 using 1N HCl. The extraction was carried out in an ultrasonic water bath (Isolab, Wertheim, Germany) maintained at 65 °C or 80 °C, and the extraction times were 15 min or 45 min. Following sonication, the solution was allowed to cool to ambient temperature and then centrifuged at 1700 rpm for 15–20 min at 4 °C. The supernatant was mixed with an equal volume of 95% ethanol before being left overnight at 4 °C. The colloidal mixture was then filtered under vacuum. The retentate (pectin) was washed with 95% ethanol and then dried in an oven (redLINE RF 115, Deutschland, Germany) at 60 °C. The dried pectin was weighed, and the yield was calculated.

### 2.4. Determination of pectin yield

The pectin yield (%) was calculated on a dry weight basis using the following formula, as described by Gharibzahedi et al. [15]:

$$\text{Yield (\%)} = \frac{\text{weight of dried pectin (g)}}{\text{weight of dried peel powder (g)}} \times 100 \quad (1)$$

## 2.5. DE

The DE was determined using the titrimetric method described by Zioga et al., [16]. Approximately 1 g of dried pectin was moistened with 2 mL of ethanol and then dissolved in 20 mL of distilled water. A homogeneous mixture was prepared by stirring, and three drops of phenolphthalein were added as an indicator. The solution was then titrated with 0.1N NaOH solution until a permanent pale pink endpoint was reached; the volume used was recorded as A. Subsequently, 10 mL of 0.1N NaOH was added to the same sample flask, and the mixture was left to react for 15–20 min to hydrolyze the methyl esters. Then, 10 mL of 0.1N HCl was added to the mixture, followed by continuous stirring until the pink color vanished. Finally, the solution was titrated again with 0.1N NaOH until the pale pink color reappeared and persisted; this volume was recorded as B.

The DE% was calculated using the following formula (2):

$$DE(\%) = \frac{B}{A+B} \times 100 \quad (2)$$

## 2.6. Morphological characterization

The morphological characteristics of the dried samples were examined using scanning electron microscopy (SEM) (JSM-7610 F plus, JEOL, Japan). Dried powdered pectin was mounted on a sample stub using double-sided conductive carbon tape and subsequently coated with a thin layer of gold via ion sputtering. The system was operated at an acceleration voltage of 15 kV, and images were captured at 100× magnification [17].

## 2.7. Fourier transform infrared spectroscopy

The functional groups of the extracted fruit peels pectin were analyzed using Fourier Transform Infrared Spectroscopy (FT-IR) (PerkinElmer, FTIR/FIR/NIR Spectrometer Frontier-ATR, USA) following the method described by Li et al. [18]. Dried pectin powder was thoroughly mixed with dry potassium bromide powder at a ratio of 1:100 (w/w) and compressed to form transparent pellets. Spectra were acquired in the mid-infrared region from 4000 to 400  $\text{cm}^{-1}$ . All samples were scanned 240 times with a resolution of 4  $\text{cm}^{-1}$  and a scan speed of 1  $\text{cm/s}$ .

## 2.8. Preparation of jam

Jam was prepared using java plum (*Syzygium cumini*, locally known as jamun) as the raw material according to the method of Bukya & Madane [19]. Fresh, ripe fruits were purchased from a local market in Faisalabad, Pakistan, and washed thoroughly with tap water. After seed removal, the fruit pulp was prepared manually. The formulation consisted of 1 kg jamun pulp, 1 kg sugar, and 7 g pectin. Initially, 500 g of sugar was added to the pulp, and the mixture was heated over low flame with constant stirring. Heating continued for 12–16 min, with the remaining sugar added gradually during this process. Total soluble solids (TSS) were monitored using a refractometer. When TSS reached 62 °Brix, pectin and citric acid (as a preservative) were incorporated. Heating was then discontinued, and the mixture was cooled to room temperature. The prepared jam was stored in sterilized glass jars and maintained at room temperature for 15 days. APJ, LPJ-a, and LPJ-b were prepared following the same procedure.

## 2.9. Physicochemical analysis of jam

The jam underwent comprehensive physicochemical analysis according to AOAC [20] methods. Parameters determined included pH, total soluble solids (TSS), moisture content, and ash content. Viscosity was measured at 25 °C using a Brookfield DV-E viscometer. Samples were poured into the viscometer cup, and readings were taken after inserting the T-shaped spindle. Titratable acidity was assessed by diluting 2 g of sample with 10 mL of water. A 2 mL aliquot of this dilution was mixed with 10 mL of water, and 2–4 drops of phenolphthalein indicator were added. The solution was titrated against 0.1N NaOH, and calculations were performed using Equation 3.

$$\text{Citric acid (\%)} = \frac{\text{Volume of alkali used}}{\text{Volume of sample}} \times 0.007 \times 100 \quad (3)$$

## 2.10. Evaluation of organoleptic characteristics

Organoleptic evaluation was performed using a 9-point hedonic scale following the method described by Gemechu et al. [21]. A ten-member sensory panel (5 males and 5 females), aged 20–40 years, was trained according to standard sensory evaluation protocols. Prior to testing, all samples were coded with random three-digit numbers to ensure unbiased assessment. Evaluations were conducted in a specialized sensory analysis room maintained at 20–22 °C, with controlled lighting and minimal environmental distractions. Panelists were provided with distilled water and unsalted crackers for palate cleansing between samples. All sensory responses were recorded and subsequently analyzed statistically.

## 2.11. Statistical analysis

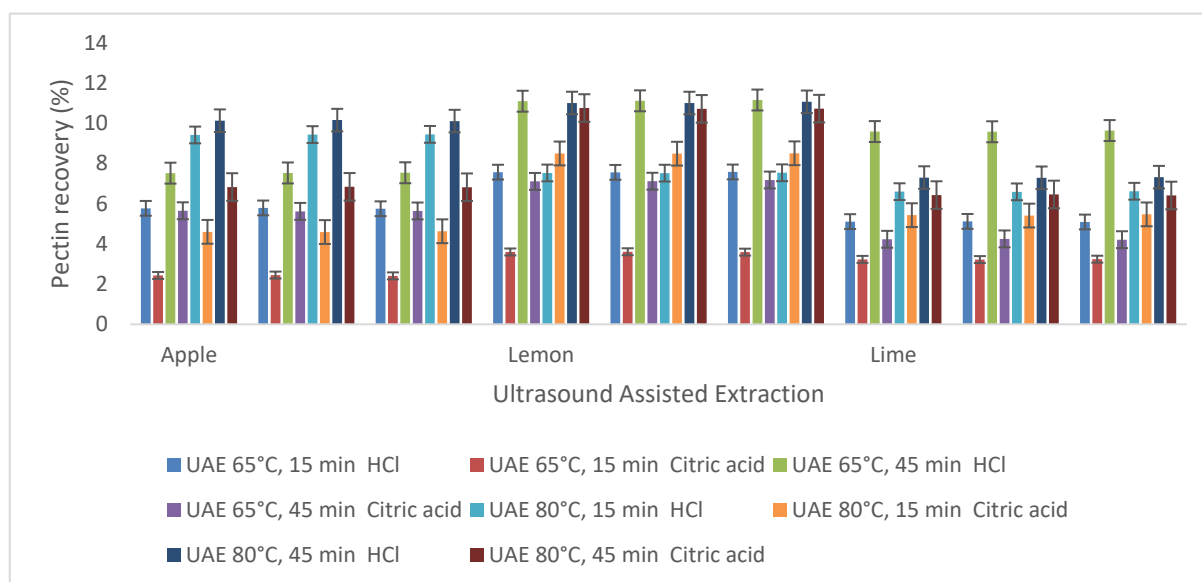
All experimental data were arranged in a completely randomized design (CRD) and analyzed using Statistix 8.1 software (Analytical Software, Tallahassee, FL, USA). Results are expressed as mean  $\pm$  standard deviation of three independent replicates. Data were subjected to one-way analysis of variance (ANOVA), and means were separated using Tukey's Honestly Significant Difference (HSD) test at a 5% probability level ( $p < 0.05$ ) to determine significant differences among treatment means, following the statistical methods described by Montgomery [22].

# 3. Results and discussion

## 3.1. Ultrasound-assisted extraction

Pectin can be extracted using numerous methods, with UAE being a particularly suitable technique for achieving higher yields. The pectin yields obtained under various UAE conditions are presented in Figure 1. In this study, extraction was performed at temperatures of 65 and 80 °C for 15 and 45 min, respectively, using a constant power (360–500 W) and a dried peel-to-solvent ratio of 1:30 g/mL. Temperature and extraction time are two critical factors influencing pectin yield. The highest lemon peel pectin yield (11.1%) was achieved at 65 °C using HCl as the solvent for 45 min. In contrast, the lowest yield (2.43% from apple peel) was obtained with citric acid at 15 min. Increasing the temperature enhances extraction by reducing solvent viscosity and surface tension; however, elevating

the temperature beyond 80 °C inhibited pectin recovery in our experiments. The mechanism of UAE involves the creation of cavitation bubbles in the liquid solvent. The implosion of these bubbles disrupts plant cell walls, facilitating the release of pectin. Consequently, a gradual increase in extraction time enables greater solvent penetration and typically results in higher pectin recovery. Nonetheless, prolonging the time beyond 45 minutes can lead to the degradation of the pectin polysaccharide structure, reducing the yield.



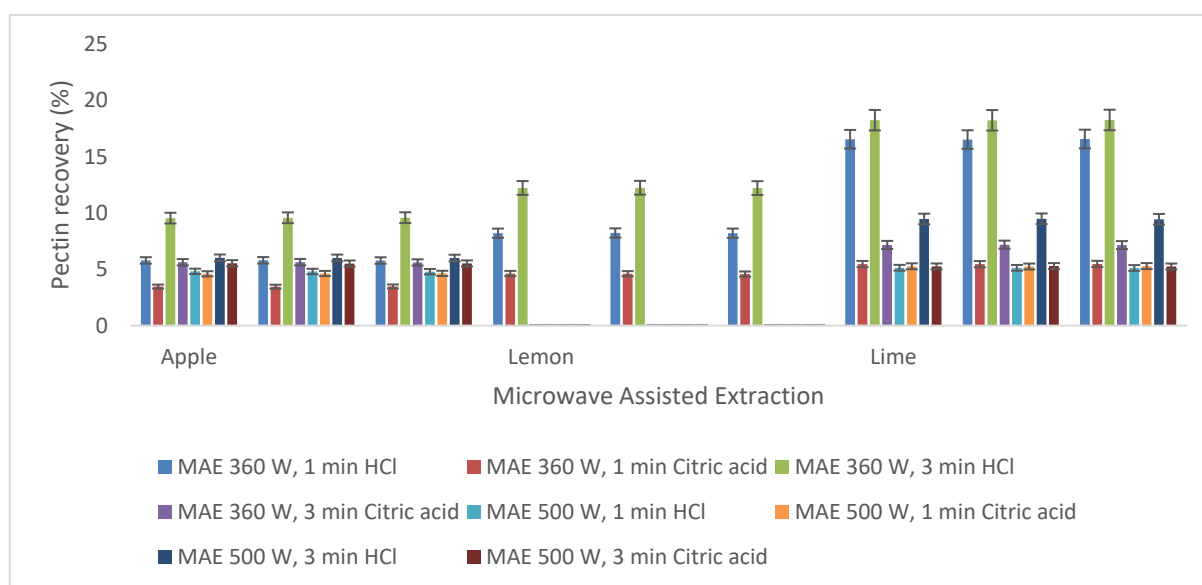
**Figure 1.** Pectin yield (%) from UAE at different extraction conditions.

These findings align with other research. Lasunon and Sengkhamparn [23] reported efficient pectin extraction from industrial tomato waste using UAE at 80 °C for 20 min. Furthermore, our results are consistent with the high yields reported for other fruits under optimized UAE conditions. For example, yields of 11.9% from kiwifruit, 13.97% from fig [15], and 15.21% from tomato waste have been documented. Similarly, a study on *Hylocereus polyrhizus* peels revealed a 31.4% yield of high-methoxyl pectin at 65 °C for 71 min. The high yield of 21.5% from jackfruit using acetic acid and 9.18% from apple pomace [10] further demonstrates the efficacy of UAE. Notably, a very high yield (57–60%) was extracted from Granny Smith apple peel by Villamil-Galindo and Piagentini [7], underscoring the potential of UAE as a highly effective extraction method.

### 3.2. Microwave-assisted extraction

MAE was performed across power and time ranges, with the results detailed in Figure 2. The highest pectin yield was obtained from lime peel (18.25%) using hydrochloric acid (HCl) as the solvent at 360 W for 3 minutes. Under similar solvent conditions but at a shorter duration (360 W for 1 min), lemon peel yielded 8.21% pectin. However, pectin extraction from lemon peels was not feasible at higher microwave powers. This was attributed to the development of an excessively thick solution consistency, which prevented effective centrifugation under the required conditions. As the power increased from 360 W to 500 W, the absorbed microwave energy caused pectin degradation. This breakdown of the molecular network adversely affected pectin recovery. When citric acid was used as

the solvent, the highest yield from lime peel was 7.17%. Apple peel yielded 9.57% pectin with HCl as the solvent. Consistently, increasing the microwave power led to a decrease in yield across all conditions. Microwave-induced rapid heating and ultrasound-induced cavitation differentially facilitate the hydrolysis of protopectin and the release of soluble pectin. These findings align with other studies. Maran & Prakash [24] reported similar trends during pectin extraction from *Carica papaya* L. peel. Rivadeneira et al. [25] achieved a 12.8% yield from banana peel at pH 3 using HCl, resulting in low-methoxyl pectin (LMP) with a particle size of 300  $\mu\text{m}$ . Lasunon & Sengkhampan [23] observed that the pectin yield from tomato waste increased from 9.43% to 22.85% as the extraction time was extended to 10 minutes. Conversely, other sources like pineapple peel, while rich in HMP, yielded a lower percentage (2.79% w/w) under MAE. Wongkaew et al. [26] reported a 13.85% yield of LMP from mango peel at a higher power of 700 W. In conclusion, MAE and UAE are more effective than conventional extraction methods, producing notable yields. A key advantage of MAE is its significantly shorter extraction time, which reduces energy and solvent consumption.

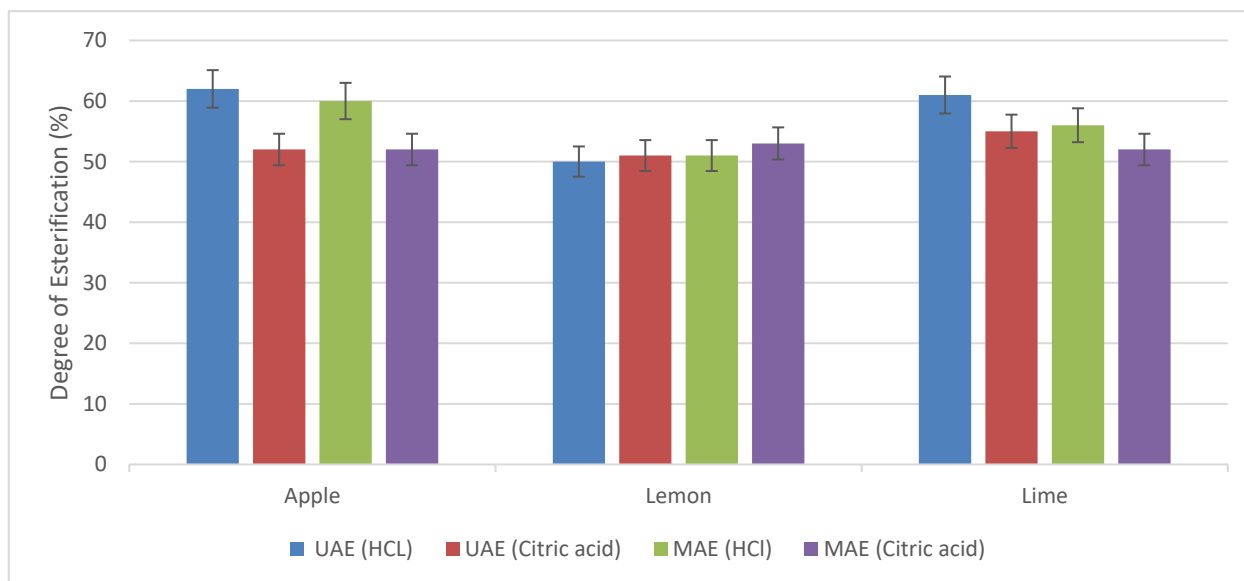


**Figure 2.** Pectin yield (%) from MAE at different extraction conditions.

### 3.3. Degree of esterification

Pectin quality was determined by its DE. Pectin polymers with a DE greater than 50% are classified as HMP, while those with less than 50% are termed low-methoxy pectin (LMP). The DE values for our samples, listed in Figure 3, ranged from 50.13% to 61.83%, indicating that all extracted samples qualify as HMP. This classification is significant as the DE directly affects the gelling characteristics of pectin. High-DE pectin forms gels in high-sugar environments, with gelation time decreasing as DE increases. In contrast, low-DE pectin gels in the presence of calcium ions within a broader pH range of 2.3 to 6.1 [27]. Our results are consistent with the broader literature, though variations exist based on the source and extraction method. For instance, UAE-extracted sisal pectin using HCl had a DE of 44.35%, classifying it as LMP. MAE-extracted pectin from jackfruit waste showed a DE of 68.43%, which was lower than that from conventional extraction [28]. Other studies have reported HMP with varying DE values: *Hylocereus polyrhizus* peels (56.10%), Konjan peel (84.5%),

banana peel (76.93%), mango peel (56.67%, and pineapple peel (63.93%). Lai et al. [29] concluded that unripe banana peel is a promising alternative to citrus fruit for pectin production.



**Figure 3.** DE (%) of pectin samples extracted from apple, lemon, and lime peels by UAE and MAE utilizing HCl and citric acid as solvents.

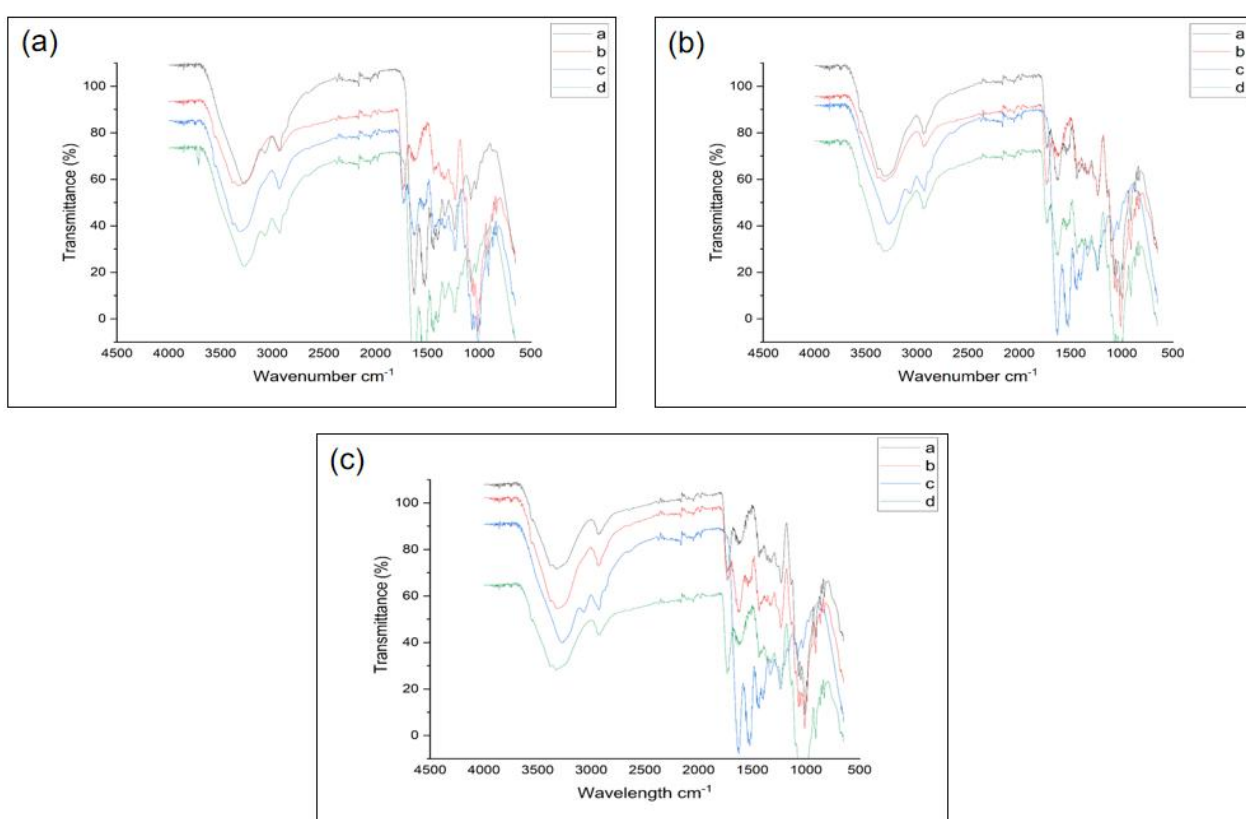
### 3.4. Influence of pH on pectin yield

The effect of pH on pectin yield was investigated across a range of values, with the maximum yield recorded at a pH of 2.00. In an acidic medium, insoluble protopectin is hydrolyzed and converted into its soluble form. However, it was observed that the yield gradually decreased as the pH was reduced further below this optimum point. Conversely, as the pH increased beyond the optimal acidic range, pectin precipitation was inhibited, leading to lower recovery. This trend is consistent with findings from other sources. A study on pomegranate peel, for instance, reported that pectin recovery decreased as the pH increased above 1-2, which was identified as the optimal range [30]. The proposed mechanism is that the solvent hydrolyzes insoluble pectin into a soluble form at highly acidic pH. In contrast, a study on passion fruit peel revealed that the yield did not differ significantly within a broad pH range of 0.3 to 5.0 [31]. Similarly, the highest yield from navel orange peel was achieved at a highly acidic pH, and a blend of banana and papaya peels yielded maximum pectin at pH 2 [32]. These results collectively demonstrate that a highly acidic environment is generally optimal for pectin extraction, although the optimum pH can vary depending on the source material.

### 3.5. FT-IR analysis

Fourier transform infrared (FT-IR) spectroscopy was performed to identify the functional groups present in the extracted pectin samples. The spectra for apple, lemon, and lime pectin, shown in Figures 4 (a, b, and c), were recorded in the range of 400–4000  $\text{cm}^{-1}$  and confirmed their classification as HMP. The analysis revealed key characteristic peaks. A broad absorption band between 3600–3400  $\text{cm}^{-1}$  was observed, indicative of O-H stretching vibrations in polyhydroxy compounds. Peaks in the range of

2929–2980  $\text{cm}^{-1}$  corresponded to C-H stretching vibrations. Critically, a strong peak between 1740–1750  $\text{cm}^{-1}$  was identified, which is characteristic of the ester carbonyl (C=O) stretching of methyl-esterified galacturonic acid units. This is a definitive marker for HMP. An additional peak near 1600–1630  $\text{cm}^{-1}$  was assigned to the asymmetric stretching of free carboxylate ( $\text{COO}^-$ ) groups. Furthermore, the region between 800–1220  $\text{cm}^{-1}$ , is known as the "fingerprint region", for carbohydrates [33], showing spectral patterns consistent with pectin polysaccharide chains. The presence of these characteristic peaks, particularly the strong ester carbonyl band at  $\sim 1745 \text{ cm}^{-1}$ , confirms that the primary structure of all extracted pectin samples is polygalacturonic acid. Rhamnogalacturonan I is a heterogeneous, branched structure composed of repeating sugar units. The Rhamnogalacturonan II is strongly anionic, consists of eight to ten  $\alpha$ -(1 $\rightarrow$ 4)-d-galacturonic acid residues and among them one may carry a methyl ester group [34].

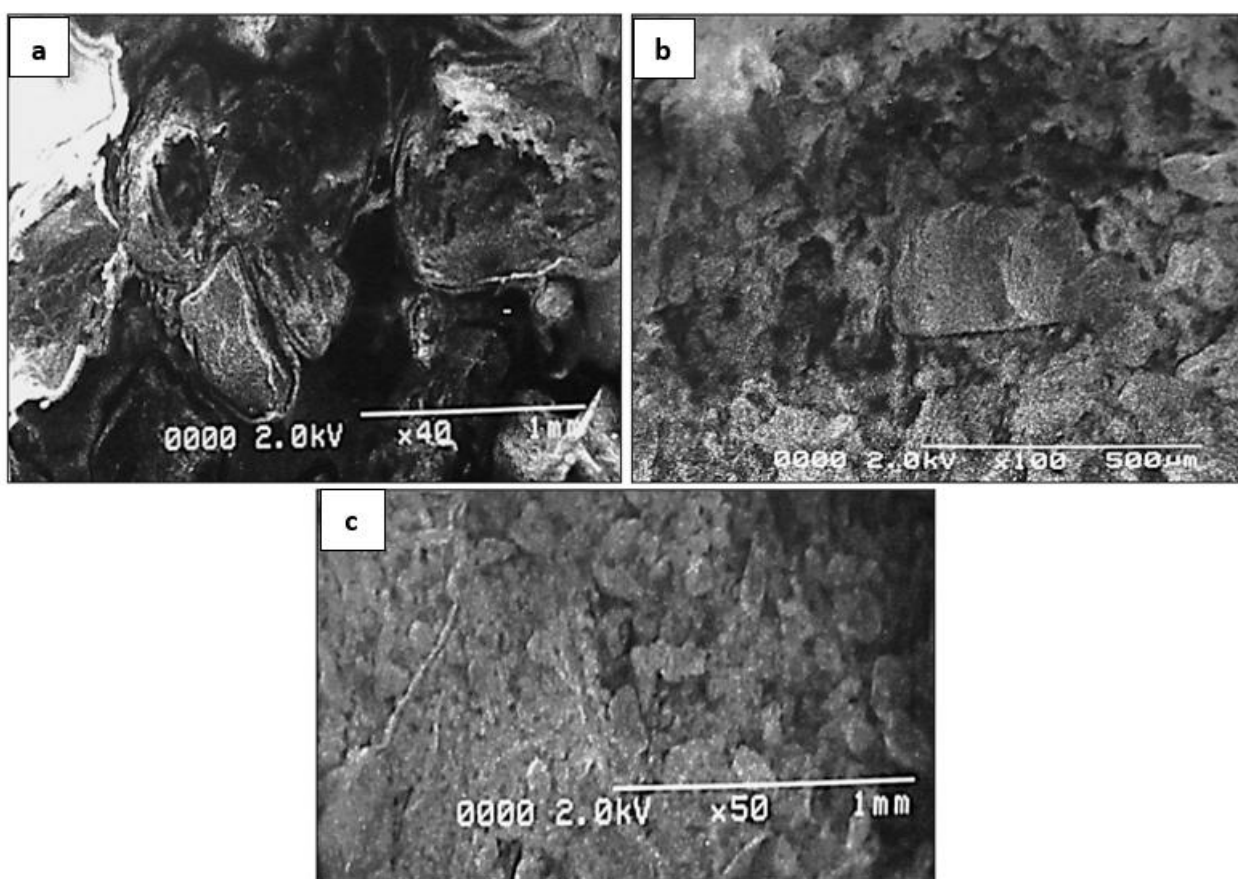


**Figure 4.** (a) FT-IR spectra of apple, lemon and, lime pectin samples; (b) FT-IR spectra of lime pectin samples; (c) FT-IR spectra of lemon pectin samples (a = UAE at 65 °C, HCl, extraction time 45 min; b = UAE at 65 °C, citric acid, extraction time 45 min; c = MAE at 360 W, 1 min, HCl, d = MAE at 360 W, 1 min, citric acid).

### 3.6. Morphological characterization

The surface morphology of the pectin samples was analyzed using SEM to explore the impact of the extraction method. The micrographs (Figure 5 A, B, C) revealed clear differences in the microstructure of each sample. The apple peel pectin extracted via UAE exhibited a ruptured surface,

likely caused by localized heating effects. Similarly, the lemon peel sample appeared highly degraded with a ruptured cell wall, attributable to the intense pressure generated by ultrasonic cavitation. In contrast, the lime peel pectin extracted via MAE using citric acid showed a highly porous and flaky structure, suggesting a higher degree of hydrolysis. This porosity is often due to a sudden increase in internal pressure from microwave heating, leading to vaporization and the formation of small bubbles or hollow openings on the surface. These observations align with other studies. A rough, ruptured, and flaky morphology is commonly reported for pectin extracted using MAE and UAE, resulting from the rapid and intense energy input of these methods [35]. Conversely, pectin from unripe fruits or that used in films is often smooth and compact, indicating minimal structural degradation [36]. In conclusion, UAE and MAE significantly alter the native morphological appearance of pectin, inducing micro-fractures, porosity, and surface ruptures that reflect the intense physical forces involved in these advanced extraction techniques.



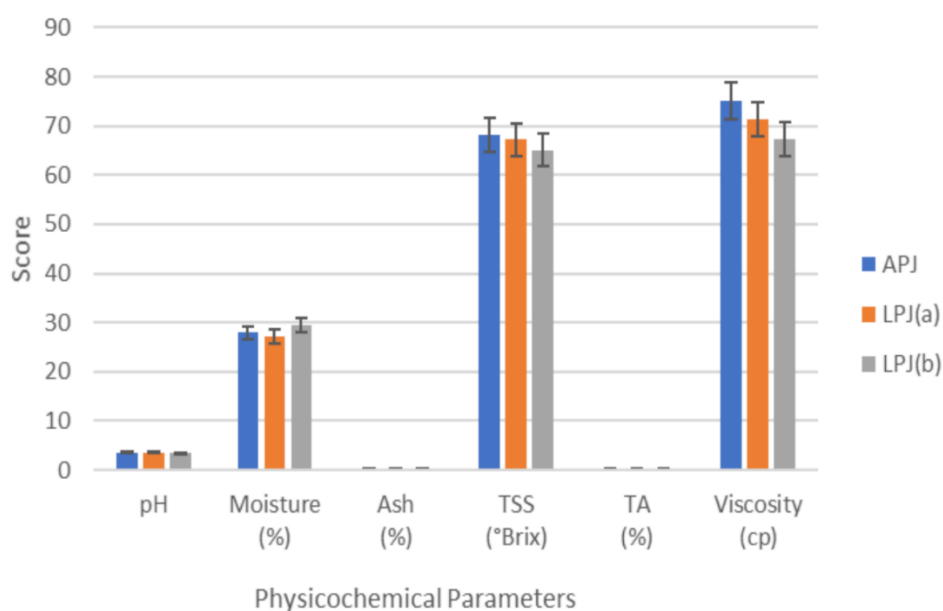
**Figure 5.** SEM images of apple, lemon and lime pectin samples respectively: 15kV, 100x magnification. (a) apple peel (UAE at 65 °C 45 min, HCl), (b) lemon peel (UAE at 65 °C, 15 min, HCl), and (c) lime peel (MAE at 360W, 3 min, citric acid).

### 3.7. Physicochemical characteristics of java plum jam

The prepared jams were subjected to physicochemical analysis, and the results are presented in

Figure 6. The jams were formulated using the same raw material but with different types of pectin extracted via MAE from apple, lemon, and lime peels. The pH values of the APJ, LPJ-a, and LPJ-b were 3.6, 3.5, and 3.4, respectively. These values are within the acceptable range for jam (3.2-3.5) as per international standards. A lower pH enhances shelf stability by inhibiting microbial growth. Furthermore, a pH between 3.2 and 3.4 was found to be optimal for gel formation. The total soluble solids (TSS) were 65.1, 67.2, and 68.1 °Brix for APJ, LPJ-a, and LPJ-b, respectively, which also fall within the required range of 65-70 °Brix. TSS is known to increase with the storage period. According to the Codex Alimentarius, fruit jams must contain 60-62% total solids.

The moisture contents of the APJ, LPJ-a, and LPJ-b were 29.4%, 27.1%, and 27.9%, respectively. All values were below the 30% threshold stipulated by international standards, which is critical for minimizing the risk of mold growth. These findings are consistent with those of Rana et al. [12], who reported moisture contents between 26.7% and 28.5% for mixed fruit jam. The ash content, which represents the total mineral content, was 0.35%, 0.33%, and 0.32% for APJ, LPJ-a, and LPJ-b, respectively. These results are comparable to the ash content range of 0.34-0.45% reported for mango jam by Bekele et al. [37].



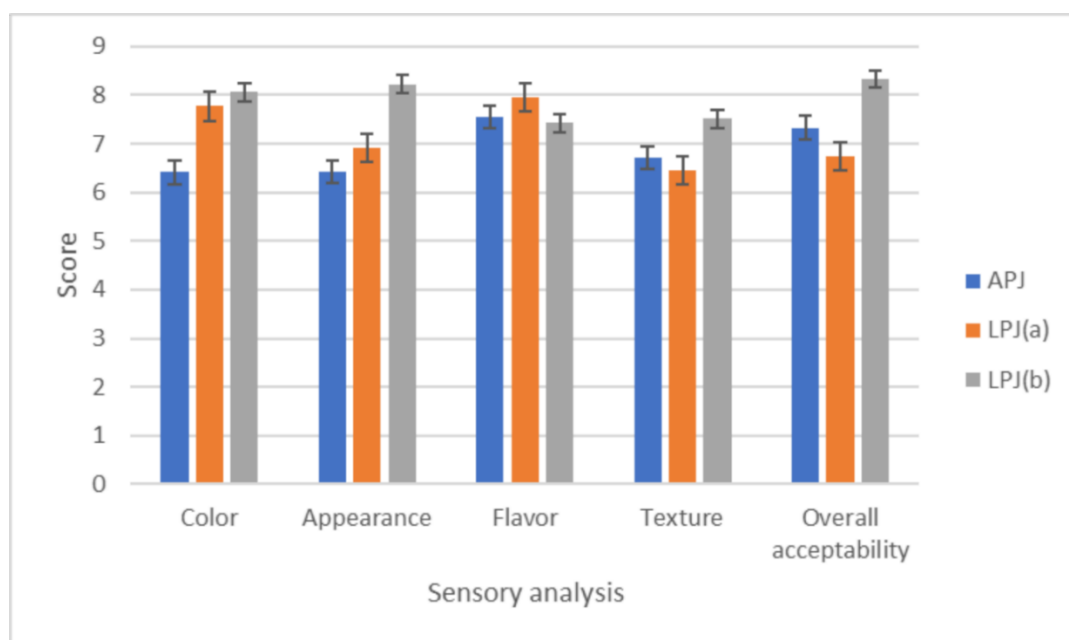
**Figure 6.** Graph representing the mean physicochemical analysis of APJ, LPJ-a, and LPJ-b. Values represent mean  $\pm$  standard deviation,  $n = 3$ ,  $p < 0.05$ .

Viscosity, defined as the internal friction of a moving liquid, is a key textural property. LPJ-b exhibited the highest viscosity (75.1 cP), followed by LPJ-a (71.3 cP) and APJ (65.3 cP). All samples fell within the acceptable viscosity range for jams. Viscosity is directly influenced by pectin concentration, sugar content, and temperature. For instance, high sugar levels contribute to the formation of a rigid gel, and pectin is pseudoplastic [38]. Awolu et al. [39] also demonstrated that temperature significantly affects the viscosity of jam. The titratable acidity (TA) values for APJ, LPJ-a, and LPJ-b were 0.34%, 0.31%, and 0.35%, respectively. TA is a crucial parameter for shelf life, as higher acidity retards microbial growth. It is important to note that lower acidity can result in poor gel formation, while excessive acidity may cause syneresis (weeping). These findings are consistent with

the TA range of 0.21–0.41% reported by Mozakkin et al. [40], with the observation that TA can increase during storage.

### 3.8. Organoleptic evaluation

The prepared java plum jams were subjected to sensory evaluation by trained panelists using a 9-point hedonic scale. The results, presented in Figure 7, indicated significant differences ( $p < 0.05$ ) in color, appearance, flavor, texture, and overall acceptability among the samples. Lime pectin jam (LPJ-b) received the highest scores for overall acceptability (8.0–8.2), followed by lemon pectin jam (LPJ-a) and APJ. Specifically, panelists preferred the flavor of jams made with lemon peel pectin and highly rated the texture of jams made with lime peel pectin, giving LPJ-b a high score of 7.5 for this attribute. These sensory results are supported by previous studies on non-traditional jams, which consistently report high consumer acceptance. The scores for color and appearance align with those reported for rose petal jam [41]. Furthermore, the overall acceptability scores are consistent with those of high-pressure processed strawberry jam (7.5–8.0) [42] and jams made from carrot-apple blends. The high score for texture in LPJ-b is a critical finding, as texture and spreadability are key determinants of consumer preference, a fact also noted by Onyekwelu [43] in a study on orange jam. It can be concluded from the panelists' scores that all jam samples were sensorially acceptable, with LPJ-b being the most preferred variant.



**Figure 7.** Graph representing the mean sensory analysis of apple pectin jam (APJ), lemon pectin jam (LPJ-a), and lime pectin jam (LPJ-b). Values represent mean  $\pm$  standard deviation,  $n = 3$ ,  $p < 0.05$ .

## 4. Conclusion

In this study, we successfully demonstrated the extraction and characterization of pectin from

apple, lemon, and lime peels using UAE and MAE extraction methods with HCl and citric acid as solvents. The extraction parameters, including time, temperature, and microwave power, were found to have a significant impact on the pectin yield. The highest pectin yield (18.25%) was obtained from lime peel using MAE. Furthermore, yields were generally higher with HCl as the solvent compared to citric acid, which is attributed to HCl's stronger acidic nature, promoting more effective hydrolysis. Characterization based on the DE% confirmed that all extracted pectin samples were HMP, with DE values exceeding 50%. This was further supported by FT-IR spectroscopy, which showed characteristic peaks confirming the presence of polygalacturonic acid. SEM analysis revealed that the extraction method significantly altered the surface morphology of the pectin. The application of the extracted pectin in java plum (jamun) jam demonstrated its excellent functional properties. Moreover, the addition of pectin significantly improved the jam's physicochemical properties and sensory profile. Jam formulated with lime peel pectin (LPJ-b) received the highest overall acceptability score (8.3) in sensory evaluation. We conclude that MAE is a more efficient extraction method than UAE for obtaining high yields of pectin in a shorter time. Apple, lemon, and lime peels are valuable sources of HMP. These findings indicate that the extracted pectin has significant potential for use in the food industry as a gelling and thickening agent, as well as in other fields for various applications, promoting the valorization of fruit processing waste. In future research, researchers should focus on the industrial-scale application of this valorization, including optimization through hybrid techniques, such as microwave-assisted enzymatic extraction, and a thorough assessment of its economic and environmental viability.

### Use of AI tools declaration

The authors declare they have not used Artificial Intelligence (AI) tools in the creation of this article.

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### Conflict of interest

The authors declare no conflicts of interest.

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