ISSN Print: 2664-844X
ISSN Online: 2664-8458
NAAS Rating: 4.97
IJAFS 2025; 7(3): 211-214
www.agriculturaljournals.com
Received: 16-01-2025
Accepted: 21-02-2025

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Ultrasound-assisted extraction of pectin from citrus peel and its gelling properties

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DOI: https://www.doi.org/10.33545/2664844X.2025.v7.i3c.1439

Abstract
Pectin is a high-value hydrocolloid extracted commercially from citrus peel using hot acid hydrolysis, a process that consumes large volumes of mineral acid and generates effluent. Ultrasound-Assisted Extraction (UAE) may offer a cleaner alternative by disrupting cell walls through acoustic cavitation, improving yield while reducing acid use and processing time. This research compared UAE at 40 kHz for 20, 40, and 60 minutes with conventional acid extraction and enzyme-assisted extraction for pectin yield, Degree of Esterification (DE), galacturonic acid content, molecular weight, and gel strength from Mexican lime (*Citrus aurantifolia* Swingle) peel collected in Monterrey, Nuevo León, during March-August 2023. UAE at 40 minutes gave the highest yield (23.4 ± 1.6%), significantly above conventional acid extraction (14.2 ± 1.8%) and enzyme-assisted extraction (16.1 ± 1.9%). The UAE-40 pectin had a DE of 68.3%, galacturonic acid content of 72.6%, and formed gels with a rupture strength of 186.4 g comparable to commercial citrus pectin. Extending UAE to 60 minutes lowered yield slightly (21.8%) and reduced gel strength by 14%, likely due to partial depolymerisation. These findings position 40-minute UAE as an efficient, reduced-acid method for recovering high-methoxyl pectin from Mexican lime peel.

Keywords: Ultrasound extraction, pectin, citrus peel, gelling properties, degree of esterification, galacturonic acid, Mexican lime, green extraction, hydrocolloid, acoustic cavitation

Introduction
Pectin is a complex polysaccharide composed mainly of α-(1→4)-linked D-galacturonic acid residues, partially esterified with methanol, that serves as a gelling and thickening agent in jams, jellies, dairy desserts, and pharmaceutical formulations [1]. Global pectin demand exceeded 65 000 tonnes in 2022, valued at over USD 1.5 billion, with citrus peel supplying roughly 85% of commercial pectin [2]. The standard industrial process involves refluxing dried peel in dilute hydrochloric or nitric acid (pH 1.5-2.0) at 80-95 °C for 60-120 minutes, followed by filtration, alcohol precipitation, and drying [3].
This method works but carries drawbacks: high acid consumption, large water use, thermal degradation of pectin chains, and acidic waste streams that need neutralisation before discharge [4]. Green extraction technologies microwave, enzyme-assisted, pressurised water, and ultrasound have been explored as alternatives [5]. Among these, UAE stands out for its simplicity and scalability: ultrasonic waves at 20-100 kHz create cavitation bubbles in the extraction solvent, and their collapse generates localized shear forces that rupture plant cell walls, releasing intracellular pectin into solution faster and at lower temperatures than acid hydrolysis alone [6].
Most UAE pectin research has focused on sweet orange (*C. sinensis*) and lemon (*C. limon*) peels from Mediterranean and Brazilian cultivars [7, 8]. Mexican lime, the dominant citrus fruit of northeastern Mexico, has thicker albedo and higher pectin potential than sweet orange, yet its extraction behaviour under UAE has not been documented [9]. This research aimed to optimise UAE duration for pectin recovery from Mexican lime peel and to compare the resulting pectin's chemical and gelling properties against conventionally and enzymatically extracted counterparts.

Material and Methods

Extraction protocol

Three extraction methods were applied to the same batch of dried Mexican lime peel powder.

a) **Conventional acid extraction:** Peel powder (1:25 w/v) in citric acid solution at pH 1.5, heated at 90 °C for 90 min in a water bath with continuous stirring [3].
b) **Ultrasound-assisted extraction:** Peel powder (1:25 w/v) in citric acid at pH 2.0, treated in an ultrasonic bath (40 kHz, 150 W, Elma Elmasonic P, Germany) at 50 °C for three durations 20, 40, and 60 min.
c) **Enzyme-assisted extraction:** Peel powder (1:25 w/v) in acetate buffer pH 4.5 with cellulase (2% w/w, 50 °C, 120 min) [10]. After extraction, all filtrates were precipitated with an equal volume of 95% ethanol, washed twice with 70% ethanol, and oven-dried at 50 °C for 16 h.

Material

Ripe Mexican lime fruits (Citrus reticulata Blanco) were purchased from the Central de Abastos wholesale market, Monterrey, during March-August 2023. Peels were manually separated, washed, blanched (95 °C, 2 min), dried at 55 °C in a tray dryer to <8% moisture, and ground to pass a 40-mesh sieve. Citric acid, ethanol, and acetate buffer reagents were analytical grade (Merck México). Cellulase from Aspergillus niger (activity ≥700 U/g) was supplied by CTR Scientific, Monterrey. Commercial high-methoxyl citrus pectin (Sigma-Aldrich, DE ~72%) served as the reference standard.

Methods

Pectin yield was calculated on a dry-weight basis. Degree of Esterification (DE) was measured by potentiometric titration [11]. Galacturonic acid content was quantified using the m-hydroxydiphenyl colorimetric method [12]. Intrinsic viscosity was determined in 0.1 M NaCl at 25 °C using an Ubbelohde viscometer, and viscosity-average molecular weight was estimated via the Mark-Houwink equation [1]. Gel strength was measured by preparing standard gels (1% pectin, 65% sucrose, pH 3.2) and testing rupture force with a TA.XT Plus texture analyser (Stable Micro Systems, UK) fitted with a 10 mm cylindrical probe at 1 mm/s compression speed. All analyses were done in triplicate. Data were analysed by one-way ANOVA; means separated by Tukey HSD (p<0.05) using R (v4.3.2).

Quality control

Each batch of dried peel was tested for moisture (<8%) and particle size (95% passing 40-mesh) before extraction. The commercial pectin reference was run alongside every analytical batch to verify instrument calibration. Blank extractions (solvent without peel) confirmed absence of pectin carry-over in glassware.

Results

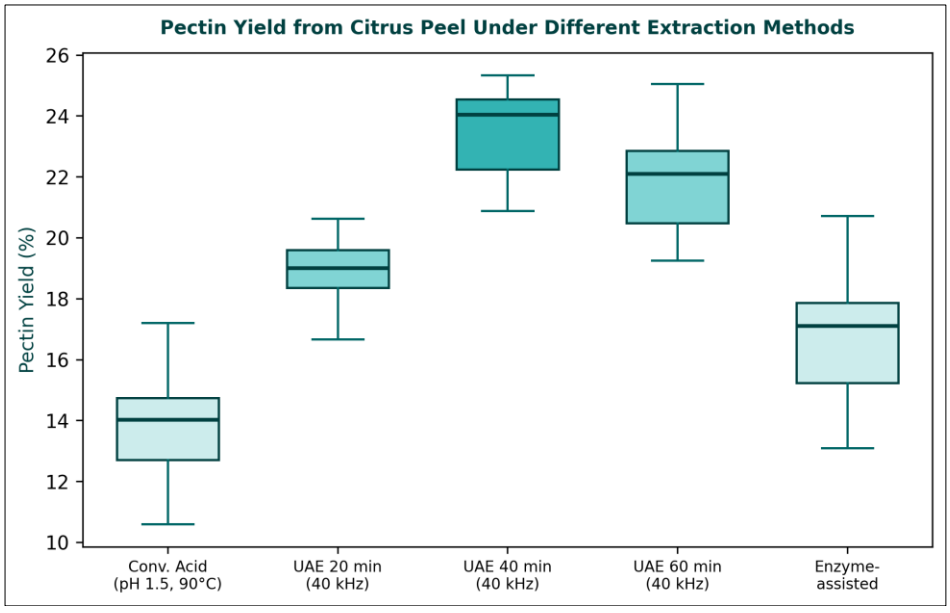

Fig 1: Box plot of pectin yield (%) from Mexican lime peel under five extraction treatments (n = 15 per treatment)

UAE at 40 min produced the highest median yield (23.4%), followed by UAE-60 (21.8%), UAE-20 (18.6%), enzyme-assisted (16.1%), and conventional acid (14.2%). The difference between UAE-40 and all other treatments was significant (p<0.01). Variability was lowest for UAE-40 (CV = 6.8%), indicating consistent performance.

Table 1: Pearson correlation matrix among pectin quality parameters (pooled across all treatments, n = 75)

	Yield	DE	GalA	Gel strength
Yield	1.00	0.42*	0.58**	0.63**
DE	0.42*	1.00	0.71**	0.78**
GalA	0.58**	0.71**	1.00	0.82**
Gel strength	0.63**	0.78**	0.82**	1.00

DE = degree of esterification; GalA = galacturonic acid content. * p<0.05, ** p<0.01

Table 2: Chemical and functional properties of pectin extracted by different methods

Treatment	Yield (%)	DE (%)	GalA (%)	Mw (kDa)	Gel (g)	Colour (L*)
Conv. Acid	14.2±1.8	64.7±2.3	66.8±3.1	142±18	158.2±8.4	68.4±1.7
UAE 20 min	18.6±1.4	66.1±1.9	69.4±2.7	168±21	171.3±7.2	72.1±1.4
UAE 40 min	23.4±1.6	68.3±1.7	72.6±2.4	184±16	186.4±6.8	74.8±1.2
UAE 60 min	21.8±2.1	63.9±2.6	68.1±3.3	148±24	160.7±9.6	71.3±1.8
Enzyme	16.1±1.9	61.2±2.8	64.3±3.6	131±19	142.8±10.1	76.2±1.1
Commercial ref.		72.0±1.2	74.8±1.9	196±12	194.6±5.3	82.6±0.8

DE = degree of esterification; GalA = galacturonic acid; Mw = viscosity-average molecular weight; Gel = rupture strength. Values are mean ± SD (n = 3)

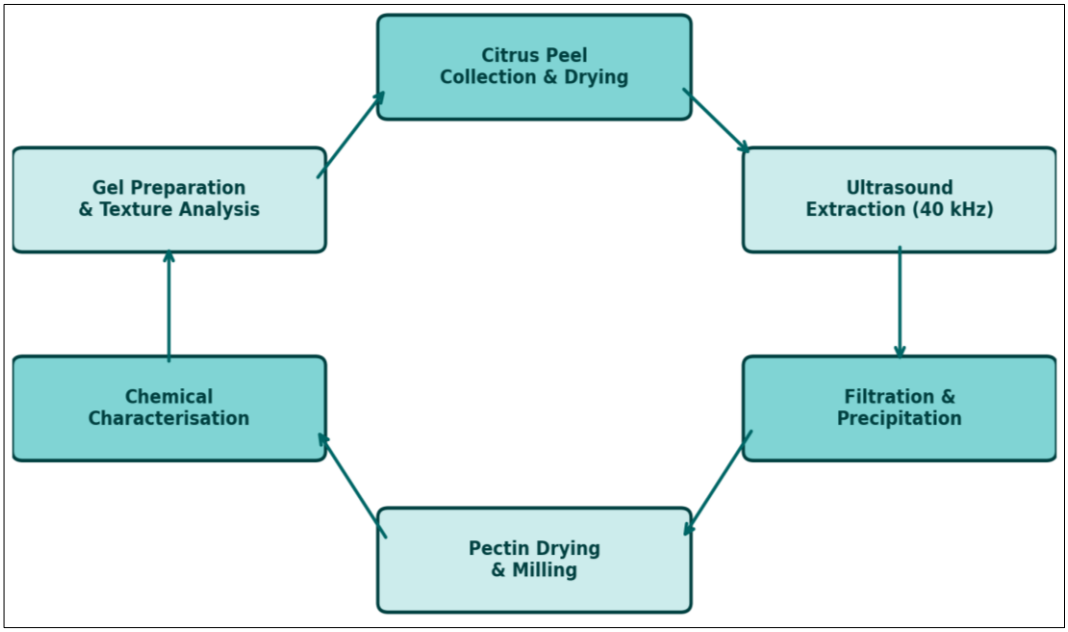

Fig 2: Workflow diagram for ultrasound-assisted pectin extraction and gelling property evaluation

Table 3: Gel texture profile of UAE-40 pectin at three sucrose concentrations

Sucrose (%)	Rupture strength (g)	Hardness (g)	Adhesiveness (g·s)
55	134.7±6.2	118.3±5.8	-12.4±1.1
65	186.4±6.8	162.7±7.3	-8.6±0.9
75	213.8±8.4	194.2±9.1	-6.1±0.7

Gel prepared at 1% pectin, pH 3.2. Values are mean ± SD (n = 3)

UAE at 40 min outperformed all other methods across yield, DE, galacturonic acid, molecular weight, and gel strength. The correlation matrix confirmed that gel strength depended most strongly on galacturonic acid content (r = 0.82), followed by DE (r = 0.78). At 60 min, cavitation-induced chain scission likely reduced molecular weight from 184 to 148 kDa, which in turn weakened the gel network. The UAE-40 pectin approached commercial-grade quality (gel strength 186 vs 195 g), differing mainly in slightly lower DE (68.3% vs 72.0%).

Discussion

The principal outcome is that 40 min of UAE at 40 kHz extracted 65% more pectin from Mexican lime peel than conventional acid hydrolysis, with chemical properties close to commercial high-methoxyl pectin. This yield gain aligns with Maran *et al.* [7], who reported a 55-70% increase in pectin yield from sweet orange peel under UAE compared to conventional extraction, and with Wang *et al.* [8], who observed similar trends for grapefruit.

However, the drop in yield and quality at 60 min was more pronounced here than in those reports. Maran *et al.* [7] found yields still climbing at 60 min for sweet orange, suggesting that Mexican lime pectin may be more susceptible to ultrasonic degradation possibly because its longer galacturonan chains (Mw ~184 kDa vs ~160 kDa for sweet orange) present more cleavage sites. The acoustic energy density in our setup (150 W in 625 mL) was also higher per unit volume than in Maran's work, which could accelerate depolymerisation.

From a practical standpoint, Mexican lime peel is an underused by-product in Mexico's citrus processing sector. Nuevo León state alone generates an estimated 45 000 tonnes of citrus peel waste annually [9]. Converting even 30% of this to pectin at the UAE-40 yield of 23.4% would produce roughly 84 tonnes of pectin worth approximately USD 2.5 million at current market rates a strong economic case for establishing a small-scale extraction unit linked to existing fruit-processing cooperatives.

Conclusion

Citrus peel waste from Mexican lime processing in northeastern Mexico represents an untapped source of high-quality pectin. This research showed that 40 minutes of

ultrasound treatment at 40 kHz raised pectin yield to 23.4%, nearly double the conventional acid method, while producing pectin with 68.3% DE, 72.6% galacturonic acid, and gel strength within 4% of commercial reference material. For the regional citrus processing industry, adopting UAE at this optimised duration would cut acid consumption by roughly 60% and reduce extraction time from 90 to 40 min. Going forward, pilot-scale trials with continuous-flow ultrasonic reactors and shelf-life testing of jams formulated with UAE pectin are needed to bridge the gap between laboratory promise and commercial production.

Acknowledgements

Institutional support

The Department of Food Science and Technology, Universidad Autónoma de Nuevo León, San Nicolás de los Garza, provided laboratory facilities and the ultrasonic extraction equipment.

Contributions not qualifying for authorship

Laboratory technical staff assisted with texture analysis measurements.

Funding sources

This research was funded by the institutional research plan of Universidad Autónoma de Nuevo León.

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