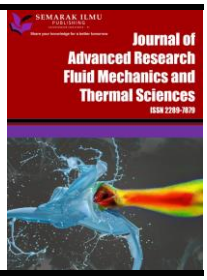
SEMARAK ILMU PUBLISHING

Journal of Advanced Research in Fluid Mechanics and Thermal Sciences

Journal homepage:
https://semarakilmu.com.my/journals/index.php/fluid_mechanics_thermal_sciences/index
ISSN: 2289-7879



Ultrasound-Assisted Extraction of Pectin from 'Saba' Banana Peel Waste: Optimization and Rheology Study

Joel Padilla Rivadeneira[1,2,*], Tao Wu[1], Katherine Ann Tamolang Castillo-Israel[2]

[1] Department of Food Science, The University of Tennessee, Knoxville, 37996, Tennessee, United States
[2] Institute of Food Science and Technology, College of Agriculture and Food Science, University of the Philippines Los Baños, 4031, College, Laguna, Philippines

ARTICLE INFO

Article history:
Received 30 March 2022
Received in revised form 6 July 2022
Accepted 20 July 2022
Available online 13 August 2022

Keywords:
Banana peel; pectin; gel; ultrasound-assisted extraction

ABSTRACT

This study utilized 'saba' banana peel waste as a source of pectin for gelling and thickening applications. Peel wastes were dried, powdered, mixed with acidified solvent, and subjected to an ultrasound-assisted extraction. Significant factors for the extraction of pectin were screened and optimized using Response Surface Methodology. The predicted optimum extraction conditions were 60% amplitude, pH 5, and 20 minutes with predicted and actual yields of 4.6% and 4.8%, respectively. The gelling ability of the ultrasound-extracted pectin (UEP) was evaluated, and results showed that the strength of the gel is directly proportional to the concentration of UEP. Moreover, the concentration of acidifier also affects gel strength – 1% glucono-delta-lactone being the most appropriate concentration of acidifier. UEP was also applied to a commercial orange juice and subjected to an in vitro digestion. Results showed that UEP increased the beverage's apparent viscosity during different digestion stages.

1. Introduction

'Saba' is a popular banana variety in the Philippines. It is processed into various food products such as chips, flour, preserve, jams, and juice – producing a significant amount of waste that usually ends up in landfills. These underutilized peel wastes contribute to the emission of methane, a greenhouse gas [1]. To mitigate the adverse environmental effects, banana peel waste can be processed or reused for other purposes. It was reported that banana peels may be used as bio-flocculant for water clarification [2]. In addition, banana peel can also be utilized to source high-value ingredients (such as pectin) and vitamins (such as ascorbic acid) that are in demand in the food industry [3].

This study focused on the extraction of pectin from 'saba' banana peel waste. Pectin is a heteropolysaccharide with a backbone made of α-1,4-galacturonic acid residues partially esterified with acetic acid or methyl alcohol at the carboxylic acid portion. It comprises 8-14% of the cell wall and is considered polymolecular and polydiverse [4]. In fruit peels, pectin is found in the middle

* *Corresponding author.*
E-mail address: jprivadeneira@up.edu.ph

https://doi.org/10.37934/arfmts.98.1.106117

lamellae and primary cell wall together with cellulose and hemicellulose. At least 300 galacturonic acid units linked by α-1,4 glycosidic linkages make up pectin [5].

Pectin is commonly used as a gelling agent in fruit applications in the food industry. Depending on the type of pectin, the mode of gelation varies. For high-methoxy pectin, gel formation is initiated by hydrogen bonding between the free carboxyl groups on the pectin molecules and between neighboring molecules' hydroxyl groups. Meanwhile, for low-methoxy pectin, a junction zone is created between unbranched non-esterified galacturonan blocks covalently bound together by coordinated calcium ions [6,7]. Aside from pectin's gelling properties, it also acts as a beverage thickener. The thickening property of pectin is influenced by its interaction with the fluid's components such as carbohydrates, proteins, pigments, and minerals. The market for pectin on a global scale attained a volume of around 80,000 tons in 2020. Furthermore, it is expected to reach around 104,000 tons by 2026 due to the increasing demand for natural and organic products [8].

Conventionally, the extraction of pectin involves the use of water, acid, base, or enzyme in combination with heat. Meanwhile, the more advanced methods use microwave, ultrasound, or pulse electric fields. This study chose ultrasound technology, among different advanced methods, because of its simplicity of operation and extraction efficiency. Also, ultrasound-assisted extraction (UAE) of pectin has already been done for mango peel, jackfruit, grapefruit peel, orange peel, dragon fruit peel, tomato waste, jujube waste, and grape pomace [9-16].

This study aims to develop an eco-efficient extraction process by optimizing the parameters for the UAE of pectin using response surface methodology (RSM) and evaluate the extracted pectin for its gelling and thickening properties.

2. Methodology

2.1 Raw Material

Fresh samples of 'saba' banana peel were obtained from a banana chips processing plant in Santa Marinduque, Philippines. The sample was manually sliced (approximately 2x2 cm^2) and then dried at 50°C for 48 h. The dried peel was then powdered (Oster®, Classic Series Accurate Blend™, Boca Raton, Florida) and stored at 25°C. The chemicals and other reagents used, unless otherwise stated, were analytical reagent grade and were purchased from Fisher Scientific, USA.

2.2 General Procedure for the Ultrasound-assisted Extraction of Pectin

Following the solid-liquid ratio (SLR) in the experimental design, and with a final volume of 100 mL, the powdered sample was mixed with 0.003 M sulfuric acid in a 100-mL beaker. The final mixture was manually mixed and then sonicated (Sonics®, VibraCell™, Newton, Connecticut) at a given pressure amplitude and length of time. During ultrasonication, the system was submerged in a cold-water bath (approximately 10°C). The ultrasonicated mixture was filtered using a Miracloth (Calbiochem®, Millipore Corp., Billerica, Massachusetts), and the resulting filtrate was centrifuged (Thermo Fisher Scientific, Sorvall Lynx 6000, Osterode, Germany) at 15,000 x g for 15 min. The filtrate underwent another round of filtration and centrifugation to remove any remaining solid particles. Dropwise addition of an equal volume of 95% ethanol was performed to precipitate the pectin. The mixture was centrifuged and the ultrasound-extracted pectin (UEP) was recovered by filtration using a Miracloth. UEP was lyophilized (Labconco, FreeZone™, Kansas City, Missouri), and the yield was calculated using the formula

$$\%\ \text{Yield, UEP} = \frac{\text{mass of pectin}}{\text{mass of dried peel}} \times 100 \quad (1)$$

2.3 Optimization of Ultrasound-assisted Extraction of Pectin

2.3.1 Screening for significant extraction parameters

A two-factorial design was used to screen the significant parameters for the ultrasound-assisted extraction of pectin. Four factors: amplitude (50 to 60%), SLR (5 to 10%), time (10 to 30 min), and the presence of acid (yes/no) were evaluated. Table 1 shows the experimental design (using coded values) for screening significant UAE parameters.

Table 1
Coded factorial design for screening parameters for the ultrasound-assisted extraction of pectin from 'saba' banana peel waste (SLR: Solid-liquid ratio)

Run	Amplitude	SLR	Time	Presence of acid
1	-1	-1	-1	-1
2	+1	-1	-1	+1
3	-1	+1	-1	+1
4	+1	+1	-1	-1
5	-1	+1	+1	+1
6	+1	+1	+1	-1
7	-1	-1	+1	-1
8	+1	-1	+1	+1

2.3.2 Optimization of significant extraction parameters

The experimental design used for the optimization of ultrasound-assisted extraction parameters was a Central Composite Design (CCD) wherein three parameters: amplitude (50 to 60%), pH (2 to 3), and time (10 to 30 min), were evaluated. SLR, previously identified as a non-significant parameter, was standardized at 8% (w/v). Table 2 shows the experimental design (using coded values) for optimizing UAE parameters.

Table 2
Central composite design for optimizing parameters for the ultrasound-assisted extraction of pectin from 'saba' banana peel waste

Run	Amplitude	pH	Time	Run	Amplitude	pH	Time
1	-1	-1	-1	11	0	-1	0
2	+1	-1	-1	12	0	+1	0
3	-1	+1	-1	13	0	0	-1
4	+1	+1	-1	14	0	0	+1
5	-1	-1	+1	15	0	0	0
6	+1	-1	+1	16	0	0	0
7	-1	+1	+1	17	0	0	0
8	+1	+1	+1	18	0	0	0
9	-1	0	0	19	0	0	0
10	+1	0	0	20	0	0	0

2.3.3 Optimization of significant extraction parameters

Design Expert 10 (Stat Ease, Inc.) was used to run statistical tests for both the factorial and optimization experiments. For the factorial experiment, the significant main effects of the UAE parameters were chosen from the half-normal probability plot of the effects. To further support the result, the magnitudes of effects were obtained from the resulting Pareto chart. The significant

parameters were identified using the generated analysis of variance (ANOVA). For the optimization experiment, the same software analyzed the responses using multiple regressions, and the resulting significance coefficients were evaluated using F-Test. The variance of the developed model was analyzed to determine the model's acceptability. Upon generating a valid model, the optimum combinations of extraction parameters were determined using numerical optimization.

2.3.4 Validation of the predicted optimum UAE parameters

The predicted optimum condition was validated by performing ultrasound-assisted extractions in triplicates. The yields obtained were compared with the predicted value.

2.4 Rheology of Pectin Gel

The rheological properties of the pectin gel were measured by a rotational rheometer (Physical MCR 301, Anton Paar, Graz, Austria) using a parallel plate (50 mm) geometry with a fixed 1 mm-gap was used. The temperature was set to 25°C.

To evaluate the effect of the concentration of an acidifier on the rheology of gel, 2% UEP solutions were prepared. The solutions were adjusted to pH 5 and then added with calcium citrate such that the final ion concentration was 2% (w/v). The resulting mixture was stirred to allow the dissolution of the solids. To acidify the solution, varied concentrations (0.5, 1, 2%) of glucono-delta-lactone (GDL) were added. The solutions were stored at 10°C for 12 h.

To evaluate the effect of the pectin concentration on the rheology of the gel, 0.5, 1.0, and 2.0% UEP and LMP were hydrated for 4 h. The solutions were adjusted to pH 5 and then added with calcium citrate such that the final ion concentration was 2% (w/v). The resulting mixture was stirred to allow the dissolution of the solids. To acidify the solution, 2% GDL was added. The solutions were stored at 10°C for 12 h.

The same gelling protocols were also performed using commercial low-methoxy pectin (LMP). The effect of the concentrations of GDL and pectin on the rheology of pectin gels were evaluated by performing a frequency sweep (0.01 to 1.0 Hz) at the identified gel's LVR (0.5% strain).

2.5 In Vitro Digestion of Pectin-fortified Orange Juice

The effect of UEP on the viscosity of an orange juice during in vitro digestion was tested following the protocol used for dietary fibers, but with slight modifications [17]. Fifteen grams of Simply® Orange juice, containing 2% pectin (w/v), was transferred to a 125-mL Erlenmeyer flask containing 4 x 1-cm glass beads. Two percent was used as the standard concentration of pectin following the standard protocol used by the Institute of Food Science and Technology, University of the Philippines Los Baños, in product development.

To simulate gastric digestion, the pectin-fortified sample was added with 7 mL of simulated gastric fluid (SGF: 0.2% NaCl (w/w) in 0.7% HCl (w/v)) was added. SGF also contained pepsin such that the latter's final concentration was 3.2 mg/mL. The final solution was adjusted to pH 2.0 using 0.1 to 0.5 M HCl. The solution was then incubated (37°C) in a shaking water bath at a speed of 175 rpm for 2 h to imitate gastric digestion. The viscosity at physiological shear rates was measured right before the incubation, and after 1 and 2 h of *in vitro* digestion [18].

To the solution used in gastric digestion, 2 mL of 750 mM $CaCl_2$, 4.6 mL of simulated bile fluid (SBF: containing 8 mg/mL bile salts), and 12 mL of simulated intestinal fluid (SIF: 5 mg/mL pancreatin dissolved in 0.5 M sodium phosphate buffer; pH 7.8) were added. The incubation with shaking was

continued for another 2 h. The viscosity of the solution was measured after the first hour and at the end of the digestion.

3. Result

3.1 Optimization of Ultrasound-assisted Extraction

3.1.1 Screening of extraction parameters

The factorial experiment was performed to screen and identify the significant parameters for the UAE of pectin from 'saba' banana peel waste. Results showed that the yield of crude pectin ranged from 1 to 3% relative to dry weight, or more than 13% of the total pectic content of the substrate [19]. Based on the analysis of variance (ANOVA), the significant parameters ($p<0.05$) were extraction time and 2-way interaction between ultrasound amplitude and acidity of the solvent. The effects of these parameters can be visualized in Figure 1. Among the significant parameters, extraction time has the highest effect on the yield of pectin. Time, amplitude, and acidity were considered for optimization in the next part of the study.

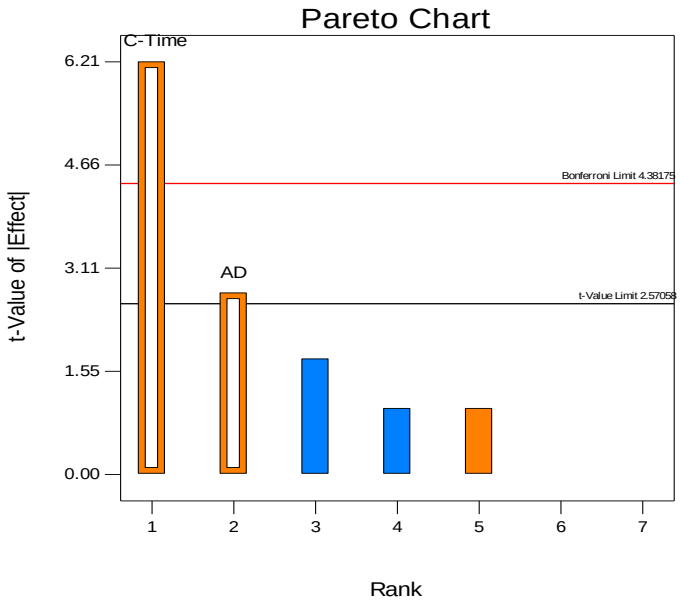


Fig. 1. Effect of the ultrasound-assisted extraction parameters on the yield of pectin from 'saba' banana peel waste (A: amplitude, C: time, and D: presence of acid)

The amplitude is the maximum variation occurring in an acoustic variable. In this study, the amplitude was used as the measure of ultrasound intensity, wherein higher amplitude means the greater formation of cavitation bubbles. The bubbles are a result of a series of expansion and compression in the medium, wherein the earlier pulls the molecules apart while the latter puts them together [20-23]. Subsequently, the biological cells and cellular membranes are disrupted, allowing the solvent to interact with the internal portions of the plant material and extract the pectic substance. Since the application of ultrasound also produces heat, the extraction system was submerged in an ice bath to eliminate the effect of temperature.

The solvent used in the study was diluted HCl. Its acidic nature initiated the hydrolysis of pectin from the protopectin [24]. Depending on the pH, the effectiveness of the solvent may increase or decrease the yield of pectin. The hydrolysis, as well as the extraction of pectic substances, is also

affected by the length of exposure to ultrasound. The time factor is essential as it dictates the completeness of the reaction. An appropriate extraction time provides an allowance for the cavitation to disrupt the plant material and the solvent to extract the pectic substances [25].

3.1.2 Optimization of extraction parameters

A CCD was used to generate the combinations of significant UAE parameters due to its ability to provide a relatively high-quality prediction over the entire design space. In addition, it doesn't require the use of points outside the original range [26]. Results showed that the range of pectin yield was 3 to 5%. Upon fitting the results in a second-order equation, a quadratic model was generated. Based on ANOVA, all three individual factors were significant ($p<0.05$). The squares of pH and time were also significant ($p<0.05$), while all the interactions between any of the factors did not significantly ($p<0.05$) affected the yield of pectin.

The RSM (Figure 2) shows that the pectin yield increased, regardless of the levels of other parameters, as the amplitude increased within the set range. The increase in amplitude translates to increased cavitation in the solvent system – disrupting the cellular structure and allowing the exposure of pectic substances. While this study shows a linear trend in the increase of pectin yield as affected by amplitude, it is also possible that beyond the range used, the number of bubbles will increase, and the transmission of energy to the solvent will be inefficient [27]. For both pH and time, regardless of the levels of other parameters, the pectin yield increased up to a certain point, then started to decrease. Hence, the acidity and time of extraction were most effective at around pH 2.5 and 20 minutes, respectively. The exposure of the plant material for a long time can cause the structural destruction and decomposition of pectin, which may reduce the extraction yield [28].

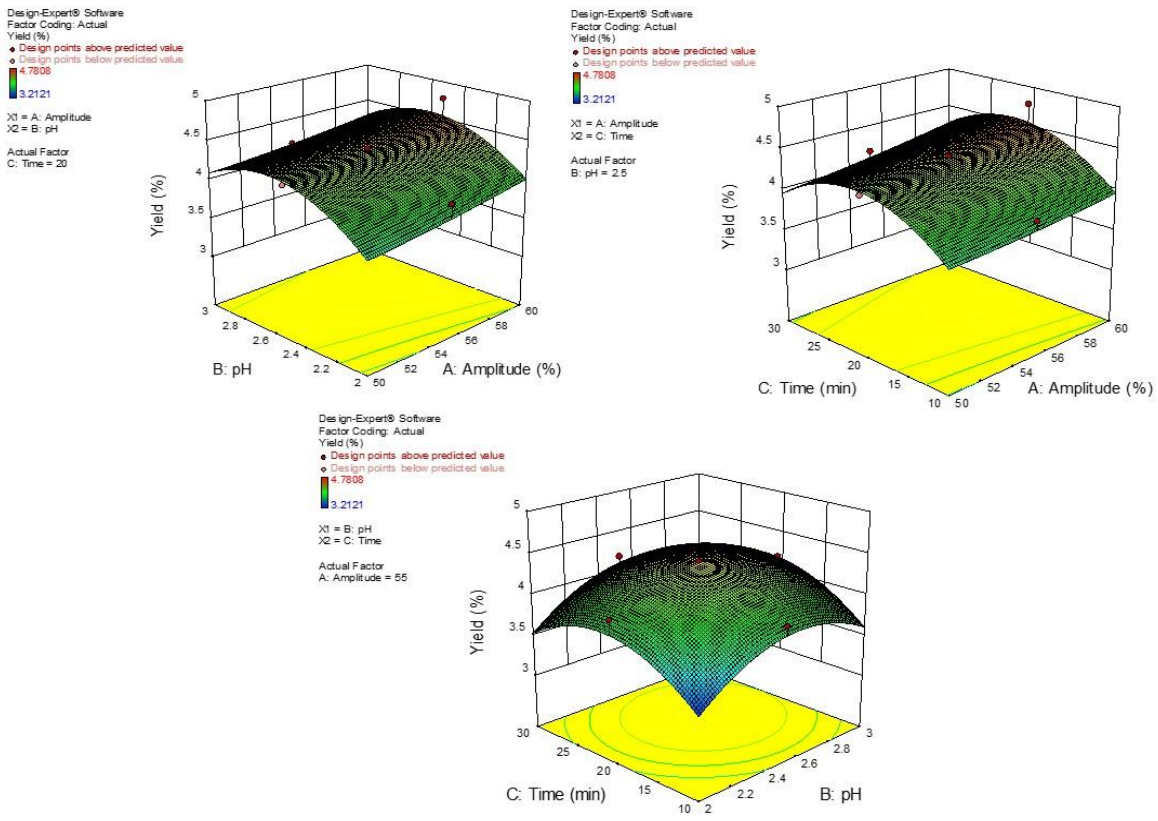


Fig. 2. Response surface model for the ultrasound-assisted extraction of pectin from 'saba' banana peel waste

The model's equation in terms of coded factors is:

$$\% \text{ Yield} = +4.41 + 0.13\text{A} + 0.16\text{B} + 0.13\text{C} - 0.39\text{B}^2 - 0.43\text{C}^2 \quad (2)$$

The model (desirability=0.97) predicted that the optimal yield can be obtained by subjecting dried 'saba' banana peel waste (submerged in diluted acid (pH 2.5)) to UAE for 20 minutes using 60% amplitude. The SLR and temperature were maintained at 8% and 195°C, respectively. The predicted yield for the said combination of parameters was 4.56%. The validation procedure, done in triplicates, resulted in 4.83 $\pm$ 0.02% or a 6% increase from the predicted yield. The amount of pectin extracted is 91% of the crude fiber content (5.5%) [19].

The yield in this study was lower than that of sisal waste (39%), grape pomace (32%), and grapefruit (18%) [28-30]. While the yield was lower compared to that of other plant materials, 4.83% is already more than 60% of the theoretical pectic content (7.5%) of dried banana peel [19]. Comparing the yield from that of the microwave-assisted extraction, UAE produced a lower yield but higher quality pectin. Preliminary tests showed that UEP has 69% purity while microwave-extracted pectin has 26% [19]. The UEP's purity was within the acceptable limit (>65%) for food use as set by the Joint FAO/WHO Expert Committee on Food Additives and the European Commission [31].

3.2 Gel Properties

Gelling is one of the most widely-used applications of pectin. Depending on the type of pectin, the mode of gelation differs. For instance, high-methoxy pectin requires an acidic environment and a sugar concentration of around 60% for gelation to happen. At low pH, the repulsion is low enough to reduce the distance between pectin chains and form a hydrogen bond and, eventually, a gel network. Moreover, sufficient hydrophobic interactions must be achieved to stabilize the molecular network, hence, the use of high-sugar content to lower the water activity of the system [32]. In contrast, for low-methoxy pectin, gelation requires higher pH and the presence of a cation. The most acceptable explanation is that the cation serves as a bridge between carboxylic groups from two pectin, forming an ionic linkage. Hydrogen bonds are also formed within the molecule, which contributes to the strength of the gel. The said mechanism is illustrated by an 'egg-box' model, wherein two oxygen atoms from one pectin molecule and three from another chain are incorporated in the calcium ion's coordination shell. While other positive ions may be used as a bridge, this study used calcium due to its large ionic radius that is big enough to coordinate with many oxygen atoms from different pectin chains [33].

Preliminary tests showed that UEP is low-methoxy in nature, hence, a cation is required to initiate the gelling process. This study used calcium citrate as the source of Ca^{2+}. Calcium citrate's low solubility in water was improved by the addition of GDL, an acidulant that slowly dissociates to gluconic acid at low temperatures [34]. The gradual decrease in pH protonated the free carboxylate ions, reducing both the surface charge and repulsion between the polymers. The whole method combines the acid-induced gelation and the 'egg-box' model, which resulted in slow but homogenous gelation of pectin.

For the effect of GDL's concentration, results (Figure 3) showed that visually, all concentrations resulted in the formation of a UEP gel. At these conditions, there is a synergistic effect between the ionic bond formed by calcium ions and the negative portions of pectin and the hydrogen bond formed by the neighboring protonated carboxyl groups of pectin. Similarly, the same concentrations of GDL resulted in a gel formation of the commercial LMP.

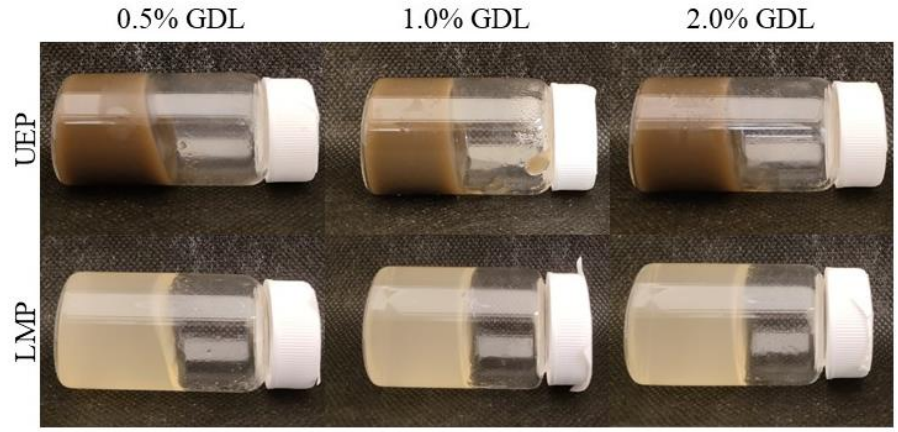


Fig. 3. Physical appearance of the gel formed by ultrasound-extracted pectin from 'saba' banana peel waste (UEP) and commercial low-methoxy pectin (LMP) (2% pectin, 2% calcium citrate, 2% GDL, pH 5)

The frequency sweep (Figure 4) of gels using varied concentrations of GDL resulted in lower loss modulus (G") relative to their corresponding storage modulus (G'). Hence, the gels possess an elastic characteristic and will remain as a gel when applied with up to 1 Hz of 0.5% strain. This property of the gel has a good implication when it comes to handling and storage. The gels won't be easily disrupted with small strains. In terms of gel strength, the use of 1.0% GDL in acidifying calcium citrate produced the strongest UEP gels.

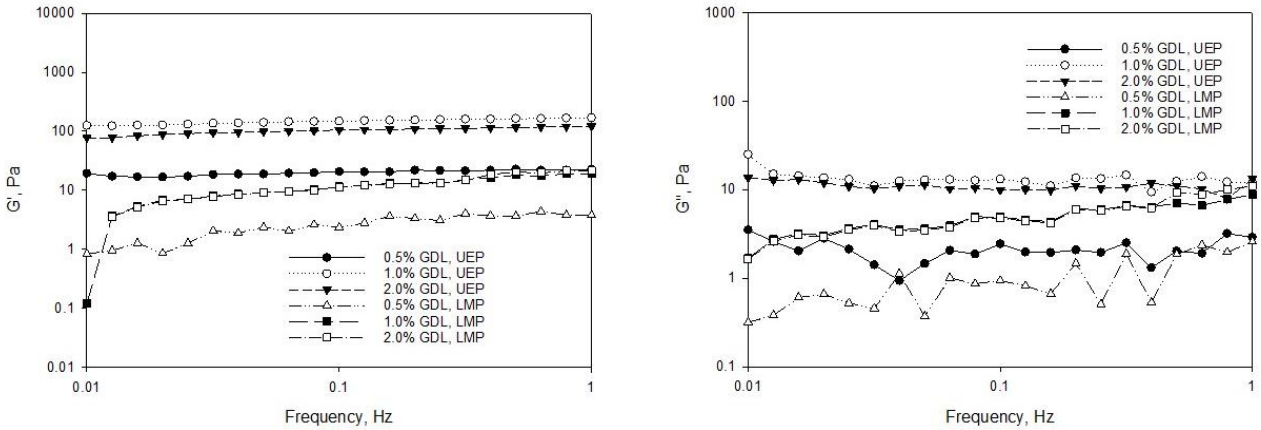


Fig. 4. Effect of varying the concentration of glucono-delta-lactone (GDL) on the rheological behaviour of gel formed by ultrasound-extracted pectin from 'saba' banana peel waste (UEP) and commercial low-methoxy pectin (LMP) (2% pectin, 2% calcium citrate; strain=0.5%)

Results (Figure 5) also showed that there is a direct relationship between the gel strength and the concentration of UEP. The gel formed by 2% UEP was the strongest gel despite the application of varied frequencies of 0.5% strain. In addition, all the gels formed by varying concentrations of pectin resulted in G" lower than their corresponding G'. Hence, the structure of all gel samples will be intact upon the application of 0.5% strain.

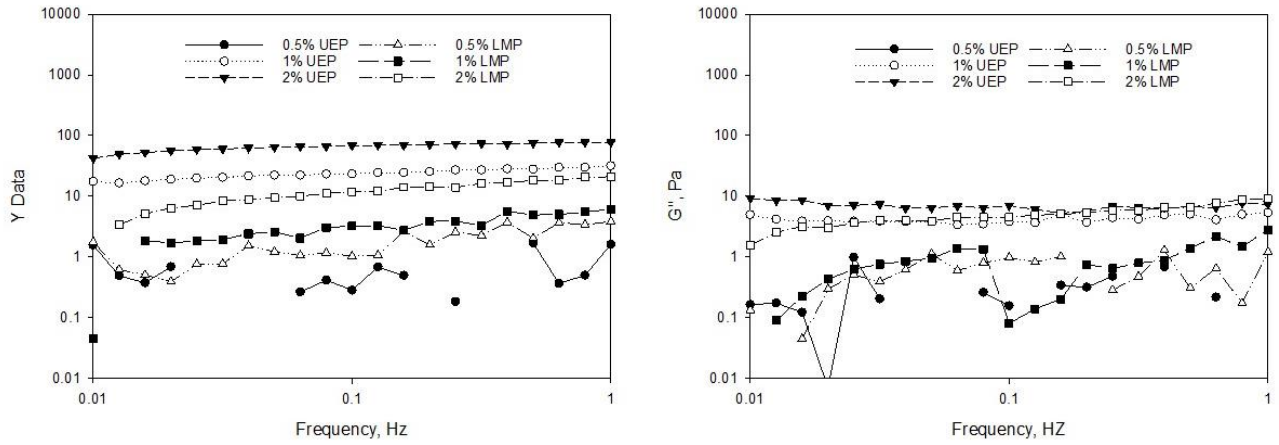


Fig. 5. Effect of varying the concentration of ultrasound-extracted pectin (UEP) on the rheological behavior of gel formed by ultrasound-extracted pectin from 'saba' banana peel waste (UEP) and commercial low-methoxy pectin (LMP) (0.5% calcium citrate; strain=0.5%)

3.3 Thickening Properties

For the rheology testing of pectin-fortified commercial orange juice, the viscosity was measured at physiological shear rates: 10 and 50 s^{-1}, as shown in Figure 6 [12]. Before digestion, UEP and LMP increased the orange juice's apparent viscosity, with the latter having a higher effect. In the first hour of the gastric phase, the viscosity was the same for the UEP and LMP-fortified orange juice 10 s^{-1}. The effect of UEP, however, was lower than that of LMP at 50 s^{-1}. During the second hour of gastric digestion, the same trend was observed on the viscosity of orange juice at 10 s^{-1}. At the higher shear rate, UEP had no more effect on the viscosity of the orange juice, while LMP further increased the juice's viscosity. In the remaining hours of in vitro digestion, the viscosities of the UEP- and LMP-fortified commercial orange juices were higher than that of the control at the lower shear rate but with no difference at the higher shear rate. Therefore, the effectiveness of UEP in increasing the viscosity of the commercial orange juice was only applicable at a low-shear rate in vitro digestion. This finding is in agreement with the results of a related study wherein commercial brands of pectin were found to increase the viscosity of orange juice [35].

A study correlating the viscosity of food with satiety suggested that fullness during a meal is higher for highly viscous food [36]. Hence, aside from the improvement of the texture of the juice, the addition of UEP could increase the level of fullness during meals. For health-conscious individuals, this could be a potential diet option in maintaining a healthy body weight.

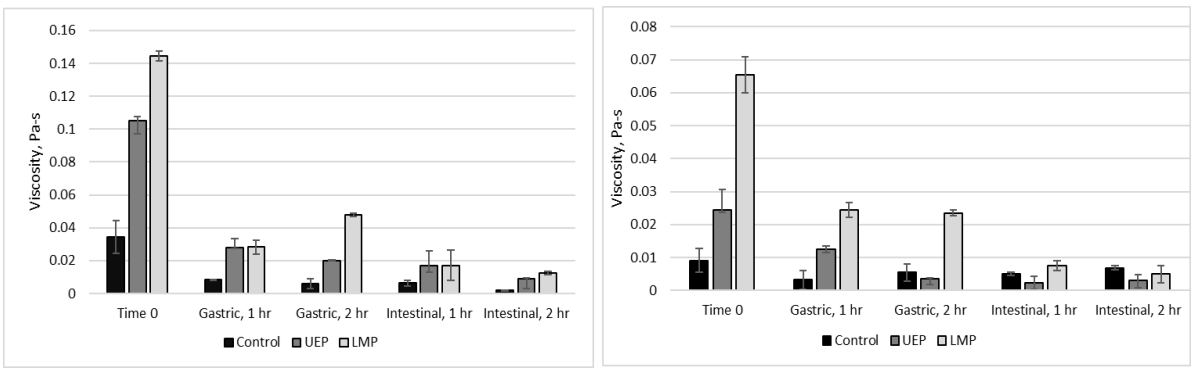


Fig. 6. In vitro digestion viscosity of orange juice with 2% pectin at physiologically relevant shear rates (A: ~10 s^{-1}, B: ~50s^{-1}). Data are mean ± standard deviation of three replications

4. Conclusion

'Saba' banana peel waste was used as a raw material for the extraction of pectin, a high-value food ingredient that is normally used as a gelling agent and a thickener in the food industry. Ultrasound-assisted extraction of pectin was performed wherein parameters were screened for significance and then optimized to maximize the pectin yield. The identified significant parameters (and corresponding optimum conditions) were amplitude (60%), time (20 min), and pH (2.5), with an actual yield of 4.8% relative to dried banana peel waste.

The ultrasound-extracted pectin (UEP) was evaluated for its gelling property. UEP was found to be low-methoxy in nature; hence, a cation source is needed for gelling. Calcium citrate, with the aid of an acidifier (glucono-delta-lactone (GDL)), was used to facilitate gelling. The slow release of calcium ions in the pectin solution resulted in a soft but strong gel that can withstand an application of at most 1 Hz of 0.5% strain. On the effect of the acidifier, the use of 1.0% GDL in acidifying calcium citrate produced the strongest UEP gel. In addition, the concentration of UEP was found to be directly proportional to gel strength.

UEP was also evaluated for its thickening property. It was applied to a commercial orange juice and then subjected to an in vitro digestion. UEP increased the apparent viscosity of the orange juice in the first two hours of in vitro digestion at the low physiological shear rate (10s-1). An increased viscosity can promote the feeling of satiety, one of the major concerns in food product development, particularly those that are intended for the health-conscious market.

Acknowledgement
This study was funded by the Department of Science and Technology's Accelerated Science and Technology Human Resource and Development Program (DOST-ASTHRDP) and the Department of Science and Technology's Research Enrichment Program (DOST-REP).

References

[1] Castillo-Israel, K. A. T., S. F. Baguio, M. D. B. Diasanta, R. C. M. Lizardo, E. I. Dizon, and M. I. F. Mejico. "Extraction and characterization of pectin from Saba banana [Musa'saba'(Musa acuminata x Musa balbisiana)] peel wastes: A preliminary study." *International Food Research Journal* 22, no. 1 (2015): 190-195.

[2] Chong, Kian Hen, and Peck Loo Kiew. "Potential of banana peels as bio-flocculant for water clarification." *Progress in Energy and Environment* 1 (2017): 47-56.

[3] Said, K. A. Mohamad, Z. Radzi, I. Yakub, and M. A. Mohamed Amin. "Extraction and Quantitative Determination of Ascorbic Acid from Banana Peel Musa Acuminata 'Kepok'." *Journal of Advanced Research in Applied Mechanics* 7, no. 1 (2015): 12-20. https://doi.org/10.31436/iiumej.v17i1.576

[4] Walter, Reginald. *The chemistry and technology of pectin*. Academic Press, 2012.

[5] Hamid Akash, Muhammad Sajid, Kanwal Rehman, and Shuqing Chen. "Natural and synthetic polymers as drug carriers for delivery of therapeutic proteins." *Polymer Reviews* 55, no. 3 (2015): 371-406. https://doi.org/10.1080/15583724.2014.995806

[6] Harholt, Jesper, Anongpat Suttangkakul, and Henrik Vibe Scheller. "Biosynthesis of pectin." *Plant Physiology* 153, no. 2 (2010): 384-395. https://doi.org/10.1104/pp.110.156588

[7] Sundar Raj, A. Allwyn, S. Rubila, R. Jayabalan, and T. V. Ranganathan. "A review on pectin: Chemistry due to general properties of pectin and its pharmaceutical uses." *Open Access Scientific Reports* 1, no. 12 (2012): 1-4.

[8] Expert Market Research. "Market Report and Forecast 2022-2027." *EMR*. Accessed June 28, 2022. https://www.expertmarketresearch.com/reports/pectin-market.

[9] Banerjee, Jhumur, R. Vijayaraghavan, Amit Arora, Douglas R. MacFarlane, and Antonio F. Patti. "Lemon juice based extraction of pectin from mango peels: waste to wealth by sustainable approaches." *ACS Sustainable Chemistry & Engineering* 4, no. 11 (2016): 5915-5920. https://doi.org/10.1021/acssuschemeng.6b01342

[10] Xu, Shu-Ying, Jun-Peng Liu, Xuesong Huang, Li-Ping Du, Fu-Lin Shi, Rui Dong, Xiao-Ting Huang, Kai Zheng, Yang Liu, and Kit-Leong Cheong. "Ultrasonic-microwave assisted extraction, characterization and biological activity of pectin from jackfruit peel." *LWT* 90 (2018): 577-582. https://doi.org/10.1016/j.lwt.2018.01.007

[11] Wang, Wenjun, Xiaobin Ma, Yuting Xu, Yongqiang Cao, Zhumao Jiang, Tian Ding, Xingqian Ye, and Donghong Liu. "Ultrasound-assisted heating extraction of pectin from grapefruit peel: Optimization and comparison with the conventional method." *Food Chemistry* 178 (2015): 106-114. https://doi.org/10.1016/j.foodchem.2015.01.080
[12] Yousuf, Owais, Anupama Singh, N. C. Shahi, Anil Kumar, and As K. Verma. "Ultrasound assisted extraction of pectin from orange peel." *Bulletin of Environment, Pharmacology and Life Sciences* 7, no. 12 (2018): 48-54.
[13] Lin, Chua Bee, Ng Yee Kai, and Ameena Ali. "Ultrasound assisted extraction of pectin from dragon fruit peels." *Journal of Engineering Science and Technology* 13 (2018): 65-81.
[14] Grassino, Antonela Ninčević, Mladen Brnčić, Dražen Vikić-Topić, Sunčica Roca, Maja Dent, and Suzana Rimac Brnčić. "Ultrasound assisted extraction and characterization of pectin from tomato waste." *Food Chemistry* 198 (2016): 93-100. https://doi.org/10.1016/j.foodchem.2015.11.095
[15] Bai, Feng-Qi, Jie Wang, and Jie Guo. "Optimization for ultrasound-microwave assisted extraction of pectin from jujube waste using response surface methodology." *Advance Journal of Food Science and Technology* 7, no. 3 (2015): 144-153. https://doi.org/10.19026/ajfst.7.1283
[16] Minjares-Fuentes, R., A. Femenia, M. C. Garau, J. A. Meza-Velázquez, S. Simal, and C. Rosselló. "Ultrasound-assisted extraction of pectins from grape pomace using citric acid: A response surface methodology approach." *Carbohydrate Polymers* 106 (2014): 179-189. https://doi.org/10.1016/j.carbpol.2014.02.013
[17] Fabek, Hrvoje, Stefanie Messerschmidt, Valentin Brulport, and H. Douglas Goff. "The effect of in vitro digestive processes on the viscosity of dietary fibres and their influence on glucose diffusion." *Food Hydrocolloids* 35 (2014): 718-726. https://doi.org/10.1016/j.foodhyd.2013.08.007
[18] Borwankar, Rajendra P. "Food texture and rheology: a tutorial review." *Rheology of Foods* (1992): 1-16. https://doi.org/10.1016/B978-1-85166-877-9.50005-5
[19] Rivadeneira, Joel P., Tao Wu, Quincy Ybanez, Adeliza A. Dorado, Veronica P. Migo, Fidel Rey P. Nayve, and Katherine Ann T. Castillo-Israel. "Microwave-assisted extraction of pectin from "Saba" banana peel waste: Optimization, characterization, and rheology study." *International Journal of Food Science* 2020 (2020). https://doi.org/10.1155/2020/8879425
[20] Hashemi, Seyed Mohammad Bagher, Amin Mousavi Khaneghah, and Hamid Akbarirad. "The effects of amplitudes ultrasound-assisted solvent extraction and pretreatment time on the yield and quality of Pistacia khinjuk hull oil." *Journal of Oleo Science* 65, no. 9 (2016): 733-738. https://doi.org/10.5650/jos.ess15252
[21] Li, Haizhou, Lester Pordesimo, and Jochen Weiss. "High intensity ultrasound-assisted extraction of oil from soybeans." *Food Research International* 37, no. 7 (2004): 731-738. https://doi.org/10.1016/j.foodres.2004.02.016
[22] Luque-Garcıa, J. L., and MD Luque De Castro. "Ultrasound: a powerful tool for leaching." *TrAC Trends in Analytical Chemistry* 22, no. 1 (2003): 41-47. https://doi.org/10.1016/S0165-9936(03)00102-X
[23] Supardan, Muhammad Dani, Anwar Fuadi, Pocut Nurul Alam, and Normalina Arpi. "Solvent extraction of ginger oleoresin using ultrasound." *Makara Journal of Science* 15, no. 2 (2012): 163-167. https://doi.org/10.7454/mss.v15i2.1066
[24] Sandarani, M. D. J. C. "A review: different extraction techniques of pectin." *Journal of Pharmacognosy & Natural Products* 3, no. 3 (2017): 1-5.
[25] Adetunji, Lanrewaju Ridwan, Ademola Adekunle, Valérie Orsat, and Vijaya Raghavan. "Advances in the pectin production process using novel extraction techniques: A review." *Food Hydrocolloids* 62 (2017): 239-250. https://doi.org/10.1016/j.foodhyd.2016.08.015
[26] National Institute of Standards and Technology. "Comparisons of response surface designs." *NIST*. Accessed June 28, 2022. https://www.itl.nist.gov/div898/handbook/pri/section3/pri3363.htm.
[27] Filgueiras, A. V., J. L. Capelo, I. Lavilla, and C. Bendicho. "Comparison of ultrasound-assisted extraction and microwave-assisted digestion for determination of magnesium, manganese and zinc in plant samples by flame atomic absorption spectrometry." *Talanta* 53, no. 2 (2000): 433-441. https://doi.org/10.1016/S0039-9140(00)00510-5
[28] Maran, J. Prakash, and B. Priya. "Ultrasound-assisted extraction of pectin from sisal waste." *Carbohydrate Polymers* 115 (2015): 732-738. https://doi.org/10.1016/j.carbpol.2014.07.058
[29] Minjares-Fuentes, R., A. Femenia, M. C. Garau, J. A. Meza-Velázquez, S. Simal, and C. Rosselló. "Ultrasound-assisted extraction of pectins from grape pomace using citric acid: A response surface methodology approach." *Carbohydrate Polymers* 106 (2014): 179-189. https://doi.org/10.1016/j.carbpol.2014.02.013
[30] Bagherian, Homa, Farzin Zokaee Ashtiani, Amir Fouladitajar, and Mahdy Mohtashamy. "Comparisons between conventional, microwave-and ultrasound-assisted methods for extraction of pectin from grapefruit." *Chemical Engineering and Processing: Process Intensification* 50, no. 11-12 (2011): 1237-1243. https://doi.org/10.1016/j.cep.2011.08.002
[31] Müller-Maatsch, Judith, Mariangela Bencivenni, Augusta Caligiani, Tullia Tedeschi, Geert Bruggeman, Montse Bosch, Janos Petrusan, Bart Van Droogenbroeck, Kathy Elst, and Stefano Sforza. "Pectin content and composition

from different food waste streams." *Food Chemistry* 201 (2016): 37-45. https://doi.org/10.1016/j.foodchem.2016.01.012

[32] Dea, I. C. M., and J. K. Madden. "Acetylated pectic polysaccharides of sugar beet." *Food Hydrocolloids* 1, no. 1 (1986): 71-88. https://doi.org/10.1016/S0268-005X(86)80009-1

[33] Flutto, L. "Pectin: properties and determination." *Encyclopedia of Food Sciences and Nutrition* (2003): 4440-4449. https://doi.org/10.1016/B0-12-227055-X/00901-9

[34] Yuliarti, Oni, Amanda Low Sok Hoon, and Sin Yee Chong. "Influence of pH, pectin and Ca concentration on gelation properties of low-methoxyl pectin extracted from Cyclea barbata Miers." *Food Structure* 11 (2017): 16-23. https://doi.org/10.1016/j.foostr.2016.10.005

[35] Logan, Kirstyn, Amanda J. Wright, and H. Douglas Goff. "Correlating the structure and in vitro digestion viscosities of different pectin fibers to in vivo human satiety." *Food & Function* 6, no. 1 (2015): 62-70. https://doi.org/10.1039/C4FO00543K

[36] Marciani, Luca, Penny A. Gowland, Robin C. Spiller, Pretima Manoj, Rachel J. Moore, Paul Young, and Annette J. Fillery-Travis. "Effect of meal viscosity and nutrients on satiety, intragastric dilution, and emptying assessed by MRI." *American Journal of Physiology-Gastrointestinal and Liver Physiology* 280, no. 6 (2001): G1227-G1233. https://doi.org/10.1152/ajpgi.2001.280.6.G1227