# EXTRACTION OF PECTIN FROM PEANUT SHELL WASTE WITH HEATING IN COMBINATION WITH ULTRASONIC-ASSISTED EXTRACTION

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**ABSTRACT:** The extraction of pectin from peanut shell waste with heating in combination with ultrasonicassisted extraction (UAE) was studied. Powder of peanut shell was extracted at a temperature of 70, 80 and 90 °C for 2 hr. It was shown that the pectin extraction at 80 °C had the highest amount of pectin at 1.56±0.07 % compared to other treatments, and the chemical characteristics of the extracted pectin were similar to commercial pectin. Then, the pectin extraction condition at 80 °C for 2 hr was combined with UAE at 40kHz/1500W for 10, 20, and 30 min. Results shown that pectin extraction at 80 °C for 2 hr in combination with UAE at 40kHz/1500W for 10 min had the highest amount of extracted pectin at 1.61±0.04 % with 88.64± 2.69 % degree of esterification, 52.12±1.46 % galacturonic acid, 9.40 % moisture and 13.50 % ash which were the most similar properties to commercial pectin compared to other treatments. Therefore, pectin extraction with heating in combination with UAE method appeared to be an increasing pectin extraction when compared to the conventional method.

Keywords: Extraction, Peanut shell, Pectin, Ultrasonic wave

# 1. INTRODUCTION

Peanut (Arachis hypogaea L.) is an important oil and protein crop in the world. It belongs to Leguminosae. Peanut crops used for oil production, roasted peanut and snack product, etc. [1] However, the amount of waste is generated in the process of harvest or oil extraction, which is only a few wastes of peanut is used as animal feed and treated as a fertilizer. It regarded as agriculture waste. Peanut shell waste derived from the harvested groundnut, such as the kernels are used to roasted snack peanuts and peanut oil, which generated a shelled peanut kernel. Sobolev and Cole [2] reported that a little peanut shell makes the cattle feed or extract of phenolic compounds. So, the more efficient utilization of peanut shells as a renewable raw material can provide for the extraction of compounds.

Pectin is a polysaccharide in the cell walls and middle lamellae of many plants and fruits and associated with cellulose, hemicellulose, and lignin structures [3]. Pectin forms the most complex of polysaccharides, which pectin backbone composed of  $(1\rightarrow 4)$ -  $\alpha$  -D-galacturonic acid molecules [4], [5]. For example, plants of *Lupinus* genus had pectin contents around 1.5%–7%, or peach (*Persica vulgaris* L.) had pectin contents up to 10%. [6]. As well as from the agricultural and food industries to obtain pectin contents with over 15% on dry basis by-products [7]. The extraction method for pectin is a new trend for the revalorization of agricultural residues. Pectin is one of the most important and widely used in the food industry. It is used as a gelling, stabilizing, encapsulating agents, and a thickening agent such as jellies and jams [8]. The pectin content has resulted in different extraction methods. It depended on the raw material, the extraction methods. and further chemical purification treatments. Usually, The conventional extraction technique was a regular solvent extraction based on stirring and heating. It was extracted by acidic solvent at high temperature [6]., microwaveassisted extraction (MAE), ultrasound-assisted extraction (UAE), and enzymatic extraction. Moreover, the extracted pectin investigated the combination of two or more techniques.

UAE is a process that highly efficient in terms of acoustic energy and solvents to extract compounds from various plant matrices. It can be relatively short times and obtain the yield quantity and high purity [9]. The mechanism of UAE based on the interaction of the ultrasonic waves and molecules of the solvent, allowing cavitation and cell disruption by ultrasonic waves, may enhance the mass transfer across the cell membranes [6]. The increasing extraction yield is fragmentation mechanisms that result in reducing particle size and increasing the surface area of the mass transfer [10]. The standard frequencies of ultrasonic waves comprised between 20 kHz and 100 kHz.

Furthermore, the high ultrasonic power induces the greater shorten that results in the fragmentation of material [11]. High efficiencies recently reported for the UAE of pectin from several agricultural wastes, such as pomegranate peel [12], passion fruit peel [13], and *Opuntia ficus indica* cladodes [14]. However, this study about the peanut shell waste for a renewable raw material can provide for the extraction of pectin. Thus, this research was to determine the extraction of pectin from peanut shell waste with heating in combination with ultrasonicassisted extraction (UAE).

# 2. MATERIALS AND METHODS

### 2.1 The Plant Materials

Peanut shell (*Arachis hypogaea* L.) collected from peanut farms in Mae-Tha, Lampang Province, Thailand, and transported to the Laboratory at Lampang Rajabhat University. The peanut shell was immersed in hot air oven at 55°C for 24 hr and then dried peanut shell were ground until obtaining a fine powder using a grinding machine and stored at room temperature until use.

# 2.2 Determination of Heating For Pectin Extraction

The extraction process was performed using the following condition by Pagan [15]. The powder of peanut shell 33 g added to 100 ml of distilled water after that, pH of solution was adjusted to 2.0 using 0.25 M HCl. The extraction by using the thermostatic bath at three different temperatures (70, 80, and 90°C) for 120 minutes and stir every 30 minutes. After heating, each treatment filtered with a white cloth, and then the solution was precipitated using 95% ethanol (solution/ethanol ratio 1:3). And then, the precipitate was filtered using vacuum filtration. The precipitate was later rinsed three times using 70% ethanol. Subsequently, it was dried at 40°C using an oven until constant weight. The extraction yield was calculated as follows:

Extraction yield (%) =  $\frac{Pectin weight (g)}{Sample weight (g)} X 100$ 

## **2.3 Determination of Heating in Combination** With Ultrasonic-Assisted Extraction For Pectin Extraction

The powders of peanut shells were extracted with the optimal condition determined from conventional heating. The powder of peanut shell 33 g was added to 100 ml of distilled water and pH 2.0 (0.25 M HCl). The pectin extraction condition at 80 °C for 2 hr was combined with UAE [14] at the frequency of 40 kHz and the power of intensity of 1500 W for 10, 20, and 30 min. After the time extraction, each treatment was cooled at room

temperature for 120 minutes and adjusted to 2.0 using 0.25 M HCl. Then, the solution was filtered with a white cloth, and then the solution was precipitated using 95% ethanol (solution/ethanol ratio 1:3). And then, the precipitate was filtered using vacuum filtration. The precipitate was later rinsed three times using 70% ethanol. Subsequently, it was dried at 40°C using an oven until constant weight. The extraction yield was calculated

# 2.4 Physiochemical Analyses of the Extracted Pectin

#### 2.4.1 Degree of esterification

The degree of esterification (DE) of pectin was determined by Rangana [16]. A dried sample of pectin (0.5 g) was solubilized in 100 ml of water without  $CO_2$  with 2 ml of ethanol. After the sample completely dissolved, five drops of phenolphthalein were added. The solution was titrated with 0.5 NaOH, and the volume of NaOH dissolved was recorded as the initial titer. Then, 10 ml of 0.5 NaOH was added and allowed to stand for 15 minutes. And then, 10 ml of 0.5 HCl was added, and the sample was shaken until the pink color disappeared. Five phenolphthalein was added and titrated with 0.5 NaOH to pink color, and the volume of NaOH dissolved was recorded as the final titer. The DE was calculated from the following equation:

$$DE\% = \frac{\text{final titer}}{\text{initial titer} + \text{final titer}} X \ 100$$

#### 2.4.2 Galacturonic acid

Galacturonic acid (GA) was determined by Rangana [16]. A dried sample of pectin (0.01 g) was solubilized in 100 ml of 0.05 N NaOH and allowed to stand for 25 minutes. The sample 2 ml was diluted with 100 ml of distilled water and then sampling 2 ml immersed into the test tube with 1 ml of 0.1% carbazole. Then, 12 ml of 98%  $H_2SO_4$  was added and allowed stand for 25 minutes, and absorbance was measured at 525 nm with UV-VIS spectrophotometer. The GA content was expressed as GA% (w/w), obtained from a calibration curve of galacturonic acid standard solutions (Fig. 1).

# 2.4.3 Determination of moisture content and ash content

The moisture content and ash content were determined by the standard test method (ASTM D3173-95). For moisture content, a dried sample of pectin (1 g) was immersed into the crucible, close with a cover, and inserted into the furnace chamber, which maintained the temperature at 150 for 3 hr until constant weight. The moisture content was calculated as follows:

Moisture (% wb) = 
$$A-B$$
 X 100

A where: A = weight of sample used (g) B = weight of sample after heating (g)

For ash content, a dried sample of pectin (1 g) was immersed into the crucible, close with a cover, and inserted into the furnace chamber, which maintained the temperature at 650 for 3 hr until constant weight. The ash content was calculated as follows:

Ash (% wb) = weight of sample used (g) 
$$X 100$$
  
weight of sample after heating (g)

#### 2.5 Statistical analysis

All experiments were replicated three times and evaluated with the regression procedure using SPSS version 17. Differences among treatments performed using Duncan's Multiple Range test ( $P \le 0.05$ ).

### **3. RESULTS AND DISCUSSION**

### **3.1 Determination of Heating for Pectin** Extraction

The yield of pectin extraction revealed in Fig. 2. The result showed that the amount of pectin extracted significantly obtained the highest yield at 80°C of 1.56 yield% (w/w). Meanwhile, the temperature at 70°C and 90°C had a yield of 1.19 and 0.72 yield% (w/w), respectively. The high extraction yields were obtained at high temperatures and low pH values due to the separating of pectin and other cell wall materials. As well as Oliveira [17] reported that the extraction of banana fruit had been considered an excellent source of pectin, which was used to determine the effect of pH 2.0–4.5 and the temperature between 70–90 °C for 120–240 minutes under stirring at 150 rpm.

In the same way, Korish [18] reported that Citrullus lanatus var. Colocynthoides watermelon obtained the optimum yield (16.7%) with acidic water solution (HCl, pH 2.0) at 85 °C for 60 min. In the other agricultural residues, peels of yellow passion fruit and dragon fruit gave pectin yield of 14.2% and 12.6% at pH 2.4 and the extraction times for 58 and 65 min, respectively [19]. Physiochemical analysis of heating for the extracted pectin as represented in Table 1. The result showed that DE and GA content of pectin extraction had nonsignificant when compared to commercial pectin. DE content at 80°C obtained the highest amount of DE of 70.41 %, which the degree of methylesterification containing up to 50% was pectin with high DM. It was used as a gelling component in heat-resistant bakery jams, fruit preservatives, and juices and jellies [20].

While GA content of all treatments was similar

to commercial pectin, which is related to the characteristic of the ability of pectin to form gels. Moisture contents were similar to all treatments, and ash content had significantly, and at 80°C had the highest when compared to all treatments.

# **3.2 Determination of Heating in Combination** With Ultrasonic-Assisted Extraction for Pectin Extraction

Pectin extraction with heating in combination with UAE represented Fig. 3, and Fig. 4. The yield of pectin extraction showed the highest yield at 80°C in combination with UAE for 10 minutes of 1.67 yield% (w/w) while, UAE for 20 minutes UAE for 30 minutes had the yield of 1.37 and 1.21 yield% (w/w), respectively. Xu [21] found that the longer time of extraction occurred, the yield slightly decreased. The extraction with heating in combination with UAE could result in the enhanced ability of pectin extraction. Wang [9] reported that the pectin extraction of grapefruit peel obtained the optimum time was 29.95 minutes at the power intensity of 12.56W and the temperature at 66.71°C, as well as Moorthy [12], found that the maximum extraction yields of pomegranate peel were determined at 61.9 °C for 28.3 minutes resulting in yield extraction of 24.2%. The extraction temperature and sonication time are essential for UAE.

On the other hand, the extraction conditions of sisal fibers showed the optimal extraction condition was 61 W for 26 min and temperature at 50 °C resulting in pectin yield of 29.3% [22]. Therefore, UAE destroyed the cell wall by allowing cavitation and enhance the mass transfer across the cell membranes [6]. The characteristic of pectin as represented in Table 2. The result showed that the amount of DE and GA significantly obtained the highest DE% at UAE 10 minutes gave the amount of DE of 88.64 %, which was high DM. Moreover, GA% in UAE 30 minutes was similar to UAE 10 and 20 minutes. Moisture contents were similar to all treatments, and ash content had significantly, and UAE 20 minutes had the highest when compared to all treatments.

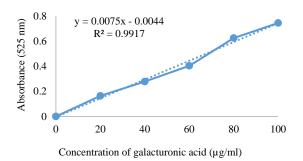


Fig. 1 Standard curve of galacturonic acid

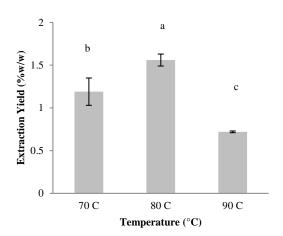


Fig. 2 Yield of pectin extracted at different temperatures. The result followed by error bar represented standard deviations. Values with the same letter on the same color bars represented no significance differences (\*P≤0.05)

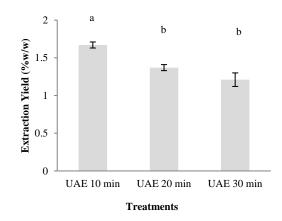


Fig. 3 Yield of pectin extracted with heating at 80°C and UAE. The result followed by error bar represented standard deviations. Values with the same letter on the same color bars represented no significance differences (\*P≤0.05)

Table 1 Characteristics of	pectin were extracted at	different temperatures.
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Characteristics	Commercial pectin	70 °C	80 °C	90 °C
DE%	83.10±4.63 <sup>a</sup>	29.35±4.82 <sup>b</sup>	$70.41 \pm 9.23^{a}$	66.37±10.15 <sup>a</sup>
GA (% w/w)	$30.36 \pm 8.33^{a}$	$24.85 \pm 0.54^{a}$	$24.18 \pm 0.38^{a}$	$24.18 \pm 0.69^{a}$
Moisture (%wb)	$9.10 \pm 0.55^{a}$	7.00±0.32 <sup>a</sup>	$8.00\pm0.87^{a}$	8.30±0.52 <sup>a</sup>
Ash (% wb)	$8.70{\pm}0.19^{a}$	$13.00\pm0.72^{a}$	28.20±0.13°	$20.10\pm0.46^{b}$

\*mean value followed by different superscript in the same row differs significantly by Duncan's new multiple range test ( $P \le 0.05$ ).

Table 2 Characteristics of	bectin were extracted with heating at 80°C and UAE at different times.

Characteristics	Commercial pectin	UAE 10 min	UAE 20 min	UAE 30 min
DE%	83.10±4.63 <sup>a</sup>	$88.64 \pm 2.69^{a}$	$62.25 \pm 5.82^{b}$	79.35±5.21 <sup>a</sup>
GA(% w/w)	$30.36 \pm 8.33^{b}$	$57.12 \pm 1.46^{a}$	56.96±4.14 <sup>a</sup>	$58.58 \pm 0.87^{a}$
Moisture(%wb)	9.10±0.03ª	9.40±0.11ª	9.30±0.12 <sup>a</sup>	12.70±0.55ª
Ash(% wb)	8.70±0.32 <sup>a</sup>	13.50±0.72 <sup>a</sup>	27.80±0.13 <sup>b</sup>	24.10±0.55 <sup>b</sup>

\*mean value followed by different superscript in the same row differs significantly by Duncan's new multiple range test ( $P \le 0.05$ ).

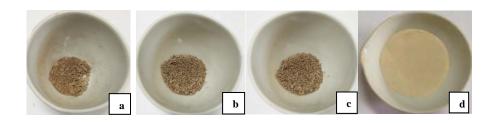


Fig. 4 Pectin was extracted from peanut shell waste with heating at 80°C and UAE at different times (a) UAE 10 min (b) UAE 10 min (c) UAE 10 min (d) commercial pectin.

### **4. CONCLUSION**

The extraction of pectin from peanut shell waste with heating in combination with ultrasonicassisted extraction (UAE) appeared to be an increasing pectin extraction. The pectin extraction at 80 °C had optimal condition, and the pectin extraction condition at 80 °C for 2 hr was combined with UAE at 40kHz/1500W for 10 min had the amount of extracted pectin, degree of esterification, galacturonic acid, moisture and ash content which were the most similar properties to commercial pectin compared to other treatments.

### 5. ACKNOWLEDGMENTS

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