

## Effect of microwave-assisted extraction on the yield and quality of apple pomace and lemon peel pectins

<sup>1,2\*</sup>Zarei, M., <sup>3</sup>Ahmadi Zenouz, A., <sup>2</sup>Saari, N., <sup>2</sup>Ghanbari, R., <sup>4</sup>Nikkhah, M. and <sup>1</sup>Vaziri, M.

<sup>1</sup>Department of Food Science and Technology, College of Agriculture and Natural Resources, Sanandaj Branch, Islamic Azad University, Sanandaj, Kurdistan, Iran

<sup>2</sup>Department of Food Science, Faculty of Food Science and Technology, Universiti Putra Malaysia, 43400 UPM, Serdang, Selangor, Malaysia

<sup>3</sup>Department of Food Science and Technology, Faculty of Agriculture, Tabriz University, Tabriz, Iran

<sup>4</sup>Institute of Technical and Vocational Higher Education of the Ministry of Jihad-e-Agriculture, Tehran, 1457896681, Iran

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### Abstract

Microwave assisted extraction treatments showed the higher pectin yields of 10.07% and 8.83% in pretreated samples by microwave and 9.4% and 8% in the extraction of dried after microwave treatment in lemon peel and apple pomace samples, respectively. Lemon peel pectin in pretreated samples by microwave and extraction of dried after microwave treatment showed the higher degree of esterification 71.8% and 70%, respectively, while apple pomace revealed 68% and 65.4% in same treatments. Furthermore, lemon peel pectin exhibited the highest galacturonic content of 74.5% in extraction of pretreated samples by microwave, while apple pomace pectin indicated the higher galacturonic acid content of 70.5% and 70% in both extraction of dried after microwave treatment and extraction of dried samples. Texture analysis of jellies prepared by various extracted pectin indicated the highest fracturability in the microwave-assisted drying treatment of 33 N and 32.5 N for apple pomace and lemon peel pectin, respectively.

### Keywords

Pectin extraction  
Microwave  
Lemon peel  
Apple pomace

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### Introduction

Pectin is an important structural component which is widely used in various fields especially in the food industries as a gelling agent, thickener, texturizer, emulsifier, and stabilizer in some products such as jams, jellies and preserves. Pectins are primarily a polymer of D-galacturonic acid, which the polygalacturonic acid chain is partly esterified with methyl groups and the free acid groups may be partly or fully neutralized with sodium, potassium or ammonium ions (Jarvis, 1984; May, 1990; Thakur *et al.*, 1997; Mohnen, 2008; Pauly and Keegstra, 2016.). The ratio of esterified galacturonic acid (GalA) groups to total GalA groups is termed as the degree of esterification (DE). There can be a wide range of DEs depending on species, tissue, and maturity. In general, tissue pectins range from 60 to 90% DE. The pectins are classified based on the DE to high methoxyl (HM) pectin and low methoxyl (LM) pectin which are either the conventionally demethylated or the amidated molecule (May, 1990; Sriamornsak,

2003; Mohnen, 2008).

The joint FAO/WHO committee on food additives recommended pectin as a safe additive with no limit on acceptable daily intake except as dictated by good manufacturing practice (Thakur *et al.*, 1997). Various physical and chemical methods have been adopted for disintegration of plant tissues and inactivation of pectolytic enzymes in the process of pectin extraction ( Pagan and Ibarz, 1999; Pagán *et al.*, 2001; Kratchanova *et al.*, 2004). These techniques include pulsating hydrodynamic action with turbulent recirculation (Panchev *et al.*, 1988), which leads to increase the yield, reduced processing time and production costs, and microwave-assisted extraction (MAE) that is one of the important techniques for extraction valuable compounds from vegetal materials. MAE has been investigated by many researchers, which their findings showed that the it can lead to a considerable increase in the yield and quality of extracted pectin. MAE is one of the preliminary treatments in the food industries that have been applied. In this technique, fruit wastes are

\*Corresponding author.  
Email: [mzarei.mail@gmail.com](mailto:mzarei.mail@gmail.com)

exposed to microwaves and some of the physical and chemical properties of their pectin and pectinases could be improved in comparing with controls (Kratchanova *et al.*, 1996; Fishman *et al.*, 2006). The most important characteristic of microwaves is volumetric heating, which means that materials can absorb microwave energy directly and internally. This fact leads to higher penetrative power, faster heating rates, higher thermal efficiency and shorter processing times compared to conventional technologies (Benlloch-Tinoco *et al.*, 2012). Many studies have been conducted on the yield and quality of extracted pectin from different fruit sources but there are few studies, which have applied the extracted pectins into a food product system such as jellies to analyze their texture profile.

Therefore, this study was aimed to evaluate effects of microwave heating (2450 MHz) on pectin extraction and quality of extracted pectin from fruit wastes (lemon peels, apple pomace) as well as the fracurability of jellies prepared with the produced pectins.

## Materials and Methods

Apple pomace from fresh apples (*Malus pumila* var. Golden delicious) and lemon peel from Citrus lemonia were used in this study. Apple was stored in the refrigerator (1-2°C) and lemons were stored in a cold storage (5°C). Lemon peels were used to extract the pectin after removal and cutting to pieces around 0.5 cm<sup>3</sup>. Apples first were grated and pressed using a muslin cloth and residue was used to extract the pectin (apple pomace). One part of samples immediately subjected to microwave for 10 min at 2450 MHz in a microwave oven and then pectin was extracted. Another part of samples was subjected to microwave for 10 min, dried in a conventional laboratory oven and then its pectin was extracted.

### Extraction methods

Four treatments were used to extract the pectin from lemon peel and apple pomace in this study as follows:

#### Extraction without treatment - control

Lemon peel and apple pomace (200 g) were placed in 1000 mL boiling water for 10 mins then drained on muslin cloth. After that the fruit materials were placed in a beaker (3 L) and soaked in 1.8L of water. The pH was adjusted to 1.5 using 0.5N hydrochloric acid. The samples were heated to 80-82°C and the extraction was completed by continuous stirring for 1 h. The resulting hot mass was filtered through muslin

cloth. After cooling, the filtrate was coagulated using an equal volume of 96% ethanol and left for 1h. The coagulated pectin was filtered using muslin cloth and washed once with 70% ethanol and finally with 96% ethanol repeatedly until there was no sugar left by reacting the pectin with 5% naphthol-ethanol. If the color of the mixture was purple, it indicated that there was sugar present. The extracted pectin was dried in an oven at 50°C until reaching to constant weight.

#### Extraction of pretreated samples by microwave

Fresh lemon peel and apple pomace were subjected to the microwave treatment for 10 min at 2450 MHz in a microwave oven. Then extraction procedure was carried out as described in section Extraction without treatment – Control.

#### Extraction of dried samples-control

A 200 g of fresh material (apple pomace and lemon peel) was dried in a conventional laboratory oven at 60°C. Pectin exaction of dried samples was performed by adding water to the lemon peel or apple pomace at ratios of 1:50 and 1:15, respectively. The extraction was carried out as described in section Extraction without treatment – control.

#### Extraction of dried after microwave treatment

Fresh samples were subjected to the microwave treatment in a microwave oven at 2450 MHz first and then dried in a normal oven at 60°C until reaching to a constant weight. Extraction of pectin was carried out as described in section Extraction without treatment – Control.

#### Methods of analysis

The extracted pectins were dried in an oven at 50°C. The yield of pectin was expressed as the weight of dried pectin extracted from the peels and pomace (w/w). Degree of esterification (DE) of extracted pectin was determined using titration with NaOH before saponification and back titration with HCl after saponification (Schultz.T.H, 1965). The anhydrouronic acid content (AUAC) of the initial material was determined by the method of Gee *et al.* (1958) with a minor modification. The ash content of samples was evaluated at 660°C using the ashing method for 8 h (Kalapathy and Proctor, 2001). Gel fracturability was evaluated using an Instron (model 1140, High Wycombe, Bucks, UK) with speed of 20 , loadcell= 50 N and a probe designed and made by authors as shown in Figure 1:

#### Jellies preparation

The jellies were formulated using 0.1% of the

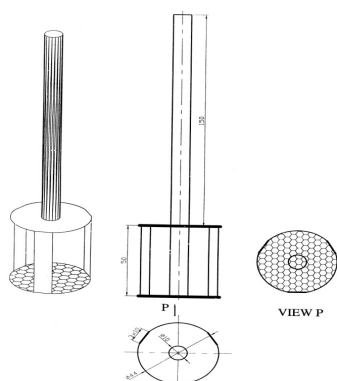


Figure 1. Specifications of probe designed for fracturability determination

extracted pectin in order to measure the fracturability. Briefly, extracted pectins were mixed with sugar at a ratio of 1:4, respectively, dispersed in hot water (60°C - 70°C) and stirred using a magnetic stirrer. The mixture was heated and stirred continuously to ensure the pectin was completely dissolved. The remaining sugar was added when the temperature reached to about 60°C - 70°C and heating stopped when the Brix of mixture reached to 62°Brix (refractometer reading). The mixture was poured into glass containers, and then stored at 4°C until further analysis.

#### Statistical design and analysis

Completely randomized design with four treatments and three replicates was used in this study. Statistical analysis was performed by MSTATC software (Version 2.1). Comparisons of treatment means were performed by Duncan's multiple range test (DMRT) at the significance level of  $p \leq 0.05$ .

## Results and Discussion

### Yield

The yield is defined as: Yield of extracted pectin =  $(P/B_0) \times 100$ , where

P is the amount of extracted pectin in g,  $B_0$  is the initial amount of sample. As shown in Figure 2, four treatments were used to extract the pectin from apple pomace and lemon peel. Lemon peel and apple pomace pretreated by microwave showed the highest pectin yields of 10.07% and 8.83%, respectively followed by dried lemon peel and apple pomace pretreated by microwave with the pectin yields of 9.4% and 8%, respectively. Higher pectin yield of samples treated with microwave heating could have been a result of being disrupted by heat thus enhances the yields of pectin.

The results showed that pretreatments of lemon peel and apple pomace increased the pectin extracted

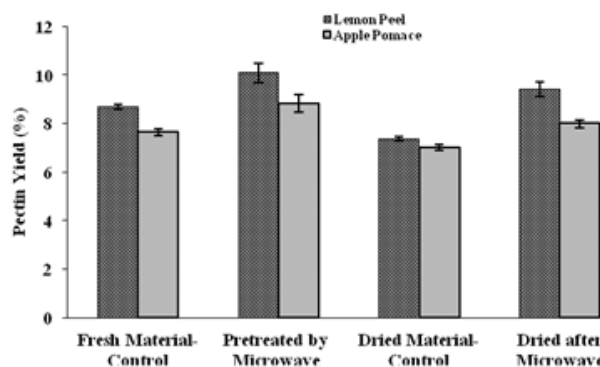


Figure 2. Effect of extraction methods on yield and pectin quality in apple pomace and lemon peel

from fresh and dried materials as well that could be attributed to the specific action of the non-gradient heating method leading to partial disintegration of the plant tissue, thus facilitating the penetration of the extracting agent as well as the partial hydrolysis of the protopectin. These effects are obviously specific to the type of fruit tissue tested. Thus, the extractable pectin rate obtained with various materials were different, but in all cases the microwave treatment favorably affected on the pectin extraction. However, in the lemon peel, extracted pectin yield percentage was higher in comparison to apple pomace. These results are in agreement with the results of the previous studies (Kratchanova *et al.*, 1994; Kratchanova *et al.*, 1996; Bagherian *et al.*, 2011).

Similarly, in a study performed by Kratchanova *et al.* (1996) the extraction of pectin from fruit materials pretreated in an electro-magnetic field of super-high frequency led to higher pectin yield. It seems that the endogenous enzymes of the peels will be inactivated and the plant tissue will be disintegrated after exposing to the microwave frequencies. In another study, response surface methodology (RSM) was used to optimize the effects of processing parameters of extraction on the yield of pectin (Fishman *et al.*, 1999; Wang *et al.*, 2007). Moreover, microwave-assisted extraction of pectin from orange skin has also been investigated by scanning electron microscopy (SEM) and atomic force microscopy (AFM) (Zhongdong *et al.*, 2006). Their findings showed that microwave can increase the yield of extracted pectin in comparing to control samples. In another study, the effect of microwave power and heating time on the yield and quality of extracted pectin from grapefruit was investigated by Bagherian *et al.* (2011), which their findings showed that the highest total amount of pectin yield was obtained at 6 min of extraction at 900W. It could be seen clearly that pectin yield increases with increasing microwave power and heating time.

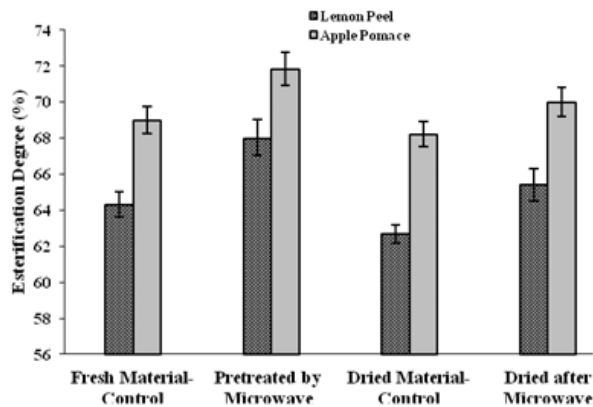


Figure 3. Effect of extraction methods on degree of esterification in apple pomace and lemon peel

#### Degree of esterification

The polygalacturonic acid chain is partly esterified with methyl groups and the free acid groups may be partly or fully neutralized with sodium, potassium or ammonium ions. The ratio of esterified GalA groups to total GalA groups is termed as the degree of esterification (DE). Pectin might be formed initially in a highly esterified form, undergoing some deesterification after they have been inserted into the cell wall or middle lamella. There can be a wide range of DEs dependent on species, tissue, and maturity. In general, tissue pectins range from 60 to 90% DE (Sriamornsak, 2003).

The effect of extraction methods on the degree of esterification (DE) of the extracted pectin from apple pomace and lemon peel are presented in Figure 3. The values of DE varied from 68.2% to 71.8% for apple pomace and 62.7% to 68% for lemon peel which are in the range of commercially available food-grade high methoxyl pectin (Table 1). The high methoxyl pectins are those with more than 60% degree of esterification. It could be seen that among all the treatments, microwave assisted methods showed higher DE values compared to the control (conventional methods). These results support the findings of Kratchanova *et al.* (1994), Hosseini *et al.* (2016) and Košťálová *et al.* (2016).

More significant decrease in DE was observed during drying of untreated materials in the laboratory drying-oven at 60°C. This could be attributed to the fact that the slow increase in temperature may activate the naturally present fruit pectin esterase and thus, catalyze demethylation of pectin. In the microwave assisted methods, the heating occurs rapidly in the whole bulk of the material and, this inactivates the pectolitic enzymes more quickly. Thus, the microwave treatment of the fresh fruit

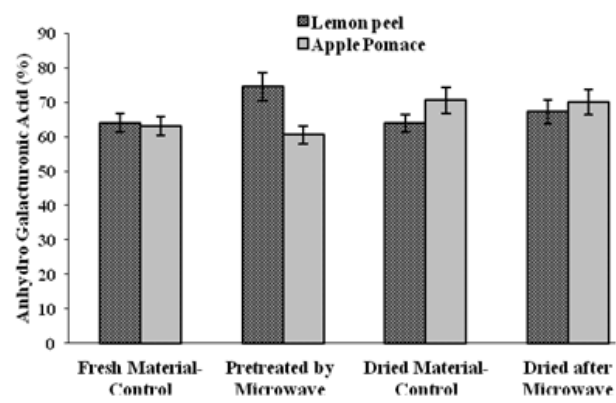


Figure 4. Effect of extraction methods on anhydro galacturonides in apple pomace and lemon peel

material exerted a favorable influence with regard to the quality of the pectic substances in the raw material (Kratchanova *et al.*, 1994; Kratchanova *et al.*, 1996).

#### Total anhydro galacturonides (TAG)

Pectins are primarily a polymer of D-galacturonic acid (a homopolymer of [1, 4]  $\alpha$ -D-galactopyranosyluronic acid units with varying degrees of carboxyl groups methylesterified) and rhamnogalacturonan (heteropolymer of repeating [1, 2]  $\alpha$ -L-rhamnosyl-[1-L]  $\alpha$ -D-galactosyluronic acid disaccharide units), making it an  $\alpha$ -D-galacturonan. The molecule is formed by L-1, 4-glycosidic linkages between the pyranose rings of D-galacturonic acid units. These uronic acids have carboxyl groups, some of which are naturally present as methyl esters and others which are commercially treated with ammonia to produce carboxamide groups.

As it can be seen in Figure 4, the percentage of the anhydrogalacturonides in all samples that were pretreated with microwave was higher than other samples, however these amounts were not significantly different for all samples. The anhydro galacturonic acid percentage for lemon peel pectin (74.5%) was higher than apple pomace pectin (70.5%). Thus there is a significant difference between the samples of lemon peel and apple pomace pectins, the sample which pretreated directly using microwave showed the highest amount of anhydro galacturonic acid percentage in lemon peel. However, this result for apple pomace was not similar and microwave assisted treatment showed the lowest anhydro galacturonic acid percentage. However, dried material after microwave treatment showed higher GaA% in apple pomace. These results are in agreement with the results of Kratchanova *et al.* (1994). These findings could be attributed to enhance the degree of penetration of reagents in the fruit tissue and speeding up the inactivation of pectinases

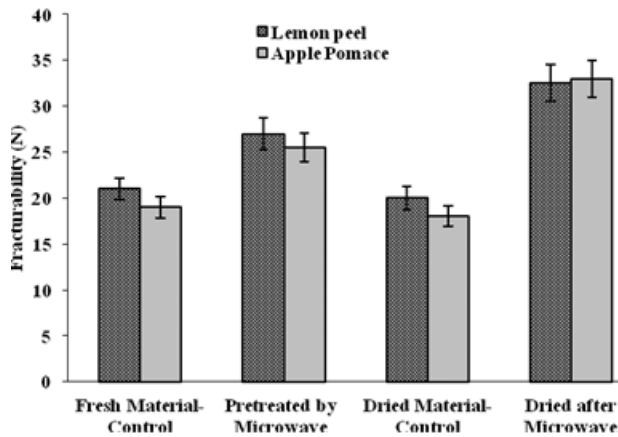


Figure 5. Effect of extraction methods on fracturability (N) in apple pomace and lemon peel

with microwave methods in comparing with other methods.

#### *Fracturability of jellies prepared by extracted pectins*

Fracturability, also called brittleness, is determined by the measure (%) of the plunger path into the jelly when it breaks. Texture analysis of Jellies prepared from extracted pectins showed that fracturability of jellies which were prepared from extracted pectin in pretreatment with microwave was higher than other methods. Pretreatment of fresh or dried material using microwave heating increased yield and quality of isolated pectins (Figure 5). Effectiveness of microwave heating pretreatment in lemon peel was higher than apple pomace samples. As shown in Figure 5, the dried samples after assisting with microwave showed the higher values of 33 N and 32.5N in apple pomace and lemon peel, respectively followed by the samples pretreated directly by microwave. Results showed that the fracturability of control samples in both cases were significantly lower than the samples, which pretreated using microwave. In both samples (lemon peel and apple pomace), the microwave dried samples showed the highest fracturability. Therefore they revealed a significant difference in comparison with the other treatments.

In conclusion, results showed that microwave could be an effective technology to employ in pretreatment and drying of apple pomace and lemon peel derived from fruit processing factories as pectin source with high quality. In fact, based on the present study, microwave drying improve the quality and yield of pectin in comparison with traditional drying methods. Moreover, microwave speeds up the drying of apple pomace and lemon peel probably due to the fast inactivation of pectinases.

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