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Study of the Chemical Properties of Pectin Extracted from Residues of Some Plant Sources

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ABSTRACT

The study was conducted in the laboratories of the College of Agriculture, Tikrit University. The study was aimed at studying the chemical properties of pectin extracted from the residues of some plant sources using citric acid and ammonium oxalate and to estimate the mineral contents. The highest percentage of pectin yield through citric acid was 64.16, 62.5, 58.33, 45.83, 33.33 and 24.70 for the peels of eggplant, orange, pomegranate, pomelo, pumpkin and watermelon, respectively. As for extraction with ammonium oxalate, the percentage of pectin extracted from eggplant peels was 37.45, orange 35.29, pomegranate 30.58, pomelo 31.76, pumpkin 27.45 and watermelon 20.8. It indicated that the best extraction solution was citric acid. The estimation of ash content in pectin samples is indicator of pectin purity. The moisture content was 1.50, 1.75, 5.78, 1.39, 4.16 and 4.89% and ash was 1.88, 2.67, 1.96, 1.65, 2.9, and 4.9%, respectively. The reason for the discrepancy in the ash content of the pectin extracted from the peels of the samples under study was due to the difference in the extraction conditions as well as the difference in the type of the primary sample used in the extraction. The equivalent weight was 849.5, 1744.2, 1052, 612.51, 580.12 and 1250 g, and the methoxyl content was 6.22, 9.72, 6.15, 7.3, 7.44 and 5.39%. The per cent content of anhydronic acid was 55.89, 74.5, 59.14, 66.41, 77.3 and 50.52, while the degree of esterification was 63.18, 74.07, 59.03, 62.40, 54.64 and 60.57. In conclusion, extraction with citric acid gave the highest percentages of pectin production from the peels of the studied samples compared with ammonium oxalate extraction. Further, the nutritional importance of the peels of fruits and vegetables was confirmed.

Key words : Pectin, moisture, methoxyl, equivalent weight, anhydronic acid

INTRODUCTION

Fruit and vegetable peels are a good source of dietary fiber and active substances as well as antioxidants (Arun *et al*, 2015). By-products of vegetables and fruits are an important source of phenolic compounds, minerals, sugars, tannins and other important compounds. Pectin is one of these compounds of high biological and industrial value in the peels of fruits and vegetables, and it is a food ingredient characterized by a high nutritional value and is used as a fixing and gelling agent in many food industries and is usually found in plant cell walls. It has been used as a gelling agent and tonic, especially in the manufacture of jams, jelly, marmalade and confectionery products (Devi *et al*, 2014). The pectin extraction process is a physico-chemical process that goes through several stages, starting with the acid extraction step, followed by the alcohol precipitation process, which is affected by several factors, including pH, time, type of solvent used and temperature. In view of the aforementioned importance of the peels of multiple fruits and vegetables, attention was directed towards studying the nutritional importance of these and tried to extract pectin to study its chemical properties along with determining content of mineral elements. Therefore, this research aimed at studying the chemical properties of pectin extracted from the residues of some plant sources using citric acid and ammonium oxalate and to estimate the mineral contents.

MATERIALS AND METHODS

The samples were collected in October 2020 from the local market of Anbar Governorate, which included eggplant, watermelon, pumpkin, orange, pomelo and pomegranate. These were kept at a temperature of 50°C until the peels were completely dry. The dry peels

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were crushed for homogeneous fineness, and kept in labelled and sealed polyethylene bags until use.

The pectin was extracted from the samples of the peels by two methods. In the first method (by citric acid) 1 g of dry peel powder was added to 30 ml of 5.0 N citric acid and the mixture was heated at a temperature of 90°C for 90 min using a magnetic stirrer. The mixture was filtered through a boring cloth (Karim, 2018).

In the second method (by ammonium oxalate); 5 g of dry peel powder was added to 120 ml of 2% ammonium oxalate in 250 ml flasks. The mixture was heated at a temperature of 90°C for 90 min, using a magnetic stirrer. The mixture was filtered using a boring cloth.

A similar volume of 98% ethanol was added to each extracted pectin and kept in the refrigerator. After 60 min it was filtered through Whatman No. 1 filter paper to dry at a temperature of 50°C. Each pectin sample was crushed and preserved in labelled and tightly closed plastic containers until use. The pectin percentage was calculated as :

% Pectin = 100 x [(weight of dried pectin) /(weight of dry peels)]

Moisture was estimated by placing 0.1 g of pectin in an oven at a temperature of 130°C until a constant weight was obtained to calculate % weight loss (Karim , 2018).

The ash content was estimated by placing 0.1 g of pectin in an oven at a temperature of 550°C until a grayish-white ash was obtained (Karim, 2018).

Pectin equivalent weight was measured according to the method of Kamble *et al.* (2017), by adding 5 ml of ethanol to 0.5 g of pectin in a 250 ml beaker, and completing the volume to 100 ml by adding NaCl solution (prepared by dissolving 0.1 g of NaCl in 100 ml of distilled water) with the electrode of the pH meter placed in the solution. 1 to 2 drops of methyl red was added in a solution of 0.1 N NaOH until pH 7.5 was reached. The contents of the flask were stirred well and left at room temperature for 30 min. Then, the equivalent weight was calculated as :

Pectin equivalent weight = 1000 x [(weight of sample)/(normality x quantity in ml of alkali)]

The method described by Kamble *et al.* (2017) was used to estimate the methoxyl aggregates of the pectin samples prepared from the peel of the samples under study and prepared in the previous step after calculating their equivalent weight, by adding 25 ml of 0.25 N hydrochloric acid solutions to each flask. After leaving it for 30 min, then stir well and rubbed the mixture against 0.1 N sodium hydroxide solution until pH 7.5 was reached. The volume of the consumed base was recorded and the methoxyl content was calculated as :

Methoxyl content = [(3.1) x (ml of alkali) x (normaility of alkali)]/[weight of sample]

The percentage of anhydronic acid (AUA) in pectin samples was calculated according to the method mentioned by Kamble *et al.* (2017), as :

% AUA = [(176 x 0.1 Z x 100) / (W x 1000)] + [(176 x 0.1 Y x 100] / (W x 1000)]

Where,

Z = ml (titer) of NaOH from the equivalent weight determination

Y = ml (titer) of NaOH from the methoxyl content determination

W = weight of sample

RESULTS AND DISCUSSION

The results in Fig. 1 show the percentages of the pectic material yield from the peels of the samples under study, using different solutions that included ammonium oxalate and citric acid at a temperature of 90°C for 90 min. The citric acid was distinguished by extracting highest pectin %, which amounted to 64.16, 62.5, 58.33, 45.83, 33.33 and 24.70 for the peels of eggplant, orange, pomegranate, pomelo, pumpkin and watermelon, respectively. As for extraction with ammonium oxalate, the percentage of pectin extracted from eggplant peels was 37.45, orange 35.29, pomegranate 30.58, pomelo 31.76, pumpkin 27.45 and watermelon 20.8.

It showed that the best extraction solution was citric acid. This was confirmed by Kazemib *et al.* (2019) though the percentage obtained was lower than what was found in this study for eggplant peels (29.17), whereas the percentage extracted from pumpkin peels was higher than

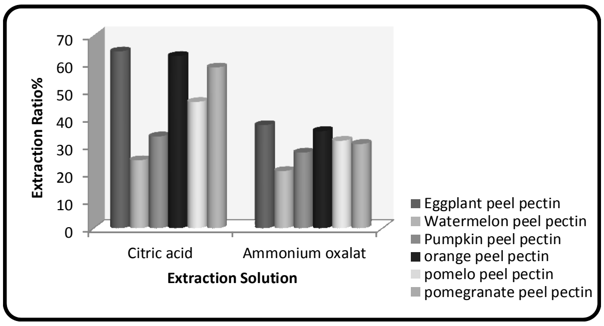

Fig. 1. Effect of using different solutions on the percentages of pectin yield from the peels of the samples under study.

what Hamed *et al.* (2017). Citric and nitric acid amounting to (6.80 and 7.72%), respectively, while Don (2019) found that the percentage of pectin produced upon extraction with citric acid and hydrochloric acid was 7.30 and 11.04%, respectively. The percentage of pectin in watermelon peels ranged from 17.6-17.4%. The percentage of pectin extracted from orange peels in this study was higher than what was obtained by Alamineh (2018), reaching 14.3%. The percentage of pectin extracted from pomelo was higher than that obtained by Roy *et al.* (2018), which amounted to 16.07%, while the percentage of pectin extracted from pomegranate peels in this study was higher than what was obtained by Cheikhrouhou *et al.* (2017) which was 10.1-6.8%, respectively, when they studied pectin extracted from pomegranate peels.

The results shown in Fig. 2 show the percentages of moisture in the pectin samples extracted from the peels of the plants under study. It was noted that the moisture content of pectin extract using ammonium oxalate was 7.65, 8.12, 2.59, 7.92, 6.49 and 2.19% for each of eggplant, watermelon, pumpkin, orange, pomelo and pomegranate, respectively, and the moisture content of pectin samples extracted using citric acid was 1.50, 1.75, 5.78, 1.39, 3.16 and 2.89% for each of the peels of eggplant, watermelon, pumpkin, orange, pomelo and pomegranate, respectively. Kazemib *et al.* (2019) reported that the moisture content of pectin extracted from eggplant peels using citric acid was 7.7%. while Abidin *et al.* (2020) mentioned that the moisture content of the pectin extracted from the husks of soybeans using sulfuric acid was 5.64%. Hamed *et al.* (2017) indicated that in the pectin of pumpkin peels the moisture content when extracted with nitric and citric acid was 5.45 and 5. 42%, respectively, and Guzel and Akpinar (2019) found that the moisture content of orange peel pectin was 10.18%. Liew *et al.* (2019) reported that the moisture content of pomelo peel pectin was 10.18%, whereas Guzel and Akpinar (2019) found the moisture content of pectin extracted from pomegranate peels as 10.31%.

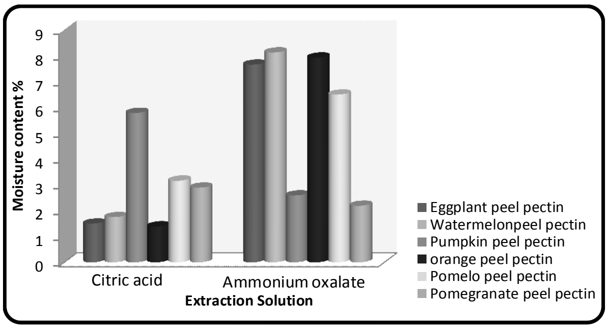

Fig. 2. Moisture percentages of pectin extracted from the peels of the samples under study using citric acid and ammonium oxalate.

The estimation of ash content in pectin samples is an indicator of pectin purity. The results showed (Fig. 3) that the percentage of ash for pectin samples extracted from ammonium oxalate was 6.08 and 7.57, 6.03, 3.31, 1.90 and 2.72% for each of eggplant, watermelon, pumpkin, orange, pomelo and pomegranate, respectively. As for the ash percentage of pectin samples extracted using citric acid was 1.88, 2.67, 1.96, 1.65, 1.9 and 2.9% for each of the eggplant, watermelon, pumpkin, orange, pomelo and pomegranate, respectively.

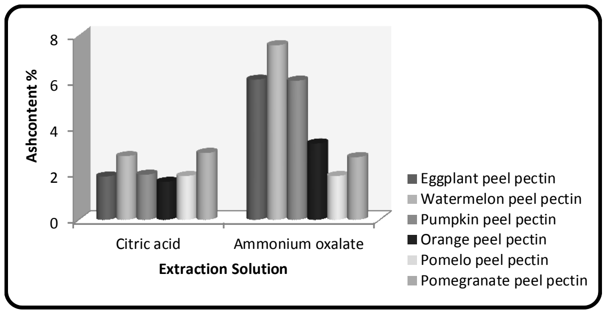

Fig. 3. Percentages of ash in pectin extracted from the peels of the samples under study with citric acid and ammonium oxalate.

Kazemib *et al.* (2019) found that the pectin extracted from eggplant peels using citric acid was 9.13%, whereas Perez *et al.* (2021) observed ash percentage of 1.24 when they studied the pectin from the peels extracted using citric acid. Further, Balsslsse and Fahlou (2018) found ash percentage of 2.41%, and Guzel and Akpinar (2019) in orange peels was 1.19%. Roy *et al.* (2018) estimated the ash content of pomelo peel pectin as 2.82, and Guzel and Akpinar (2019) reported the ash content of pomegranate peel pectin extracted using citric acid was 1.15%. The reason for the discrepancy in the ash content of the pectin extracted from the peels of the samples under study was due to the difference in the extraction conditions as well as the difference in the type of the primary sample used in the extraction, but all of them were within the minimum permissible level of 10%.

Fig. 4 shows the values of the equivalent weight of the pectin extracted from the peels of the samples under study. The equivalent weight of the pectin extracted using citric acid was 849.5, 1744.2, 1052, 612.51, 580.12 and 1250 g for each of eggplant, watermelon, pumpkin, orange, pomelo and pomegranate, while the values of the equivalent weight when extracted using ammonium oxalate were eggplant peels 705.9, watermelon 1599, pumpkin 880.3, orange 601.3, pomelo 552.2 and pomegranate 1140 g. Sari *et al.* (2018) indicated that the equivalent weight of pectin from watermelon peels ranged between 2007.7-1249.7 g, Don (2019) found the equivalent weight values of pumpkin peel pectin, were 978.35 g. Bagade *et al.* (2017) estimated the values of the equivalent weight of orange peel pectin as 86.78 g. The equivalent weight values of the pectin of pomelo peels was 1098.8 g, as indicated by Daud *et al.* (2019), and for pomegranate peels, Sari and Birlik (2020) mentioned the equivalent weight of the extracted pectin was 488.01 g.

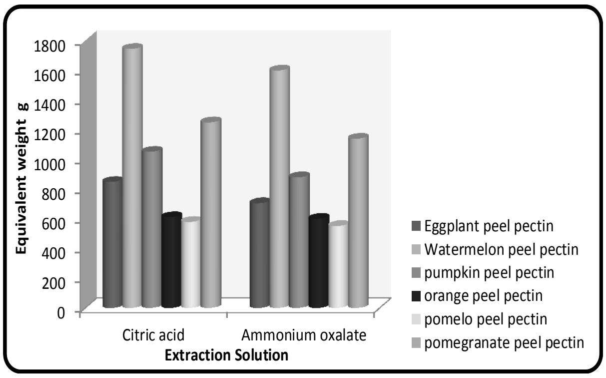

Fig. 4. Values of the equivalent weight of pectin extracted from the peels of the samples under study using citric acid and ammonium oxalate.

The methoxyl content of pectin extracted using citric acid was 6.22% in eggplant peels; 9.72% in watermelon, 6.15% in pumpkin, 7.3% in orange, 7.44% in pomelo and 5.39% in pomegranate peels, whereas the percentage of methoxyl content of pectin extracted using ammonium oxalate for eggplant peels was 5.2%, watermelon 10.03%, pumpkin 6.12%, orange 6.02%, pomelo 7.53% and pomegranate 5.63% (Fig. 5). Pomegranate peels gave the lowest percentage using citric acid, while the eggplant peels gave the lowest percentage when extracting with ammonium oxalate. These results were lower than what Perez *et al.* (2021) found in eggplant peel, which was 10.45%. However, the results converged with what was observed by Don (2019) when estimating the methoxyl content of the pumpkin peel as 6.55 and 7.72%. Similarly, Fakayode and Abodi (2018) found that the methoxyl content was 6.23% in orange peel and 5.3% in pomelo peels. For the pomegranate peels, the results converged with Sari and Birlik (2020), who estimated 5.74% methoxyl content.

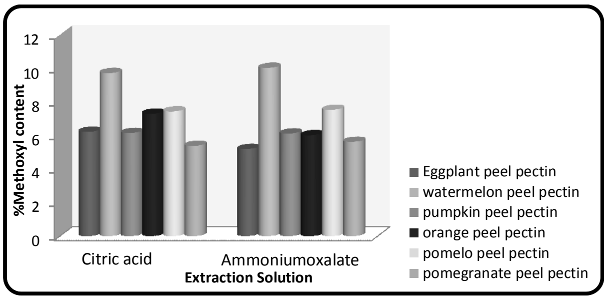

Fig. 5. Percentages of the methoxyl content of pectin extracted from the peels of the samples under study using citric acid and ammonium oxalate.

The high percentage of ahydronic acid was found in the pectin of the peels of both pomelo and watermelon in both methods of extraction, followed by the peels of orange and pumpkin, and lowest in peels of eggplant and then pomegranate (Fig. 6). In general for all samples, it reached 55.89% in the pectin of eggplant peels, 74.5% in watermelon, 59.14% in pumpkin, 66.41% in orange, 77.3% in pomelo and 50.52% in pomegranate for pectin extracted using citric acid, while the content of anhydronic acid for the extracted pectin using ammonium oxalate was eggplant 59.01%, watermelon 70.51%, pumpkin 59.46%, orange 60.19%, pomelo 75.55% and

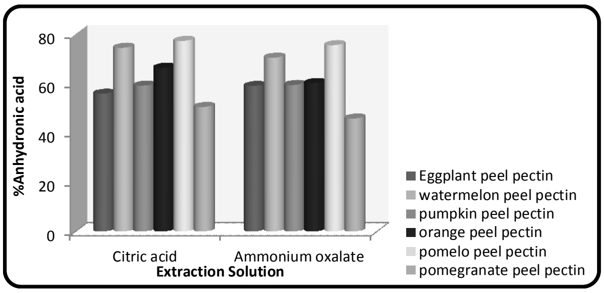

Fig. 6. Percentages of anhydronic acid content in pectin extracted using citric acid and ammonium oxalate.

pomegranate 45.84%. Karamzadeh and Ansari (2019) found that the acid content calactronic for eggplant peel pectin ranged between 74.7-51.3%, and the study for watermelon peel pectin was lower than what Perez *et al.* (2021) mentioned 81.33%, while Abidin *et al.* (2020) found 55.24%. The percentage of the pumpkin peel pectin was lower than 66.67%. Similarly, Twinomuhwezi *et al.* (2020) found 100.03% anhydronic acid content in orange peel pectin and Roy *et al.* (2018) found 74.9 and 84.29% in pomel peels which was lower than 99.35% as reported by Daud *et al.* (2019). Sari and Birlik (2020) estimated 68.64% acid in the pectin of pomegranate peels. Thus, there is nutritional importance of each of the peels of different fruits and vegetables.

CONCLUSION

Extraction with citric acid gave the highest percentage of pectin production from the peels of the studied samples in comparison with ammonium oxalate extraction. The pectin extracted from the peels of the plants was distinguished by many chemical properties which qualifies it for use in many food industries, including the manufacture of jam and marmalade.

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