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Study of the Physical and Functional Properties of Pectin Extracted from Some Plant Residues

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Abstract:

The study was conducted at the College of Agriculture / University of Tikrit. The study aims to extract pectin from the peels of some plant sources using different extraction solutions, study its physical and functional properties. The results of the extracted pectin yield using citric acid (0.5 N), ammonium oxalate (at a concentration of 2%), oxalic acid (2%) and hydrochloric acid (0.5 N), showed that the highest percentage of pectin was for citric acid, as it reached (64.16, 24.70, 33.33, 62.5, 45.83 and 58.33) % for the peels of eggplant, watermelon, pumpkin, orange, pomelo and pomegranate, respectively. The lowest percentages of hydrochloric acid were (16.6, 10, 7.5, 24.1, 21.6 and 6.6%) for the same samples, respectively.

The study of the physical properties of pectin extracted with citric acid and ammonium oxalate showed that the viscosity values, expressed by the time of the solution run-off, ranged between (1.5208 - 1.2161), (1.7250 - 1.6083), (1.6958 - 1.0875), (1.7416 - 1.5941), (2.1208 - 1.7875), (1.3916-1.3041) two years respectively, while the time required for holding for pectin extracted with citricacid was (8, 7, 8, 7, 8 and 7) minutes and for ammonium oxalate (10, 6, 7, 8, 7 and 6 minutes, respectively).

The study of the functional properties of pectin extracted with citric acid and ammonium oxalate showed the ability of the activity of emulsion that is ranging between (55 and 53), (52 and 47), (54 and 46), (59 and 54), (57 and 48), (50 and 44). % of pectin from the peels of eggplant, watermelon, pumpkin, orange, pomelo and pomegranate, respectively.

The estimation of the solubility of pectin in water and cold alkali models was also recorded, soluble and insoluble, and total solubility in hot water and hot alkalis. The results of estimating the holding capacity of water or oil for pectin extracted with citric acid showed higher degrees than pectin extracted with ammonium oxalate.

Key words: peels, fruits, vegetables, pectin, viscosity,solubility.

Introduction

Fruit and vegetable peels are a good source of dietary fiber and active substances as well as antioxidants (Arun et al., 2015). Secondary products of vegetables and fruits are an important source of phenolic compounds, minerals, sugars, phenols, tannins, and other important compounds.Pectin is one of these compounds of high biological and industrial value in the peels of fruits and vegetables.

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It is a food that is characterized by a high nutritional value. It is used as a fixative and gelling agent in many food industries. It is usually found in plant cell walls, and was first discovered by the French chemist Henri Braconnot in 1925, in Indian date.

It has been used as a gelling and texture-holding agent, especially in the manufacture of jams, jelly, marmalade and sweet products (Devi et al., 2014). The pectin extraction process is a physicochemical 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 (Pinherioc et al., 2008).

The aim of this study.

Given the aforementioned importance of the peels of multiple fruits and vegetables, therefore, attention was directed towards studying the nutritional importance of them and trying to benefit from them by demonstrating the efficiency of the studied waste in pectin production (productivity), studying somefunctional properties andPhysical .

Materials and working methods

1. Collection and Preparation of Samples

The samples were obtained during the month of October of the year 2020 from the local market of Anbar Governorate. They included a group of fruits (pomelo, orange, and pomegranate) and fruits of a group of vegetables (watermelon, eggplant, and pumpkin). The peels were isolated from the pulp of all fruits and spread on aluminum foil. For a certain period with continuous stirring and using an air source to speed up the drying process, then complete the drying using an electric oven at a temperature of 50°C until the peels are completely dry. Then the dry peels are crushed by a home grinder and then passed through a sieve the size of slots (60 mesh) to get a powder smoothly homogeneous.

2- Determining the optimal conditions for extracting pectin from the peels of the samples 2-1: Extraction with citric acid

The method mentioned by Kliemann et al (2009) in extracting pectin from the samples of the husks, under study, add 1 g of dry peel powder to 30 ml of 5.0 N citric acid, and heat the mixture at a temperature of 90 °C for 90 minutes.

2-2 Extraction pectin by Ammonium Oxalate

The method mentioned by Sabir et al. (1976), was followed to extract pectin from the husk samples of the samples under study, by adding 5 g of dry husk powder to 120 ml of 2% ammonium oxalate in 250 ml separately, then heating the mixture at a temperature of 90 °C. for 90 minutes.

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2-3 Extraction pectin by Oxalic acid

The method mentioned by Sabir et al. (1976), was followed to extract pectin from the samples of the peels under study, adding 5 g of dry peel powder to 100 ml of 2% oxalic acid in 250 ml flasks separately, then heating the mixture at a temperature of 90 °C for 90 minutes.

2-4 Extraction pectin by hydrochloric acid

The method mentioned by Kulkarni and Vijayanand (2010), was followed to extract pectin from the peels of the samples under study, by adding 1 gm of dry peels to 30 ml of 0.5 N hydrochloric acid and then heating the mixture at a temperature of 90 °C for 90 minutes in a water bath. After completing the mixing of all samples of peels under study as mentioned in the previous items with extraction solutions, then heating the mixture on a magnetic stirrer at a temperature of 90 ° C for 90 minutes, then filtering through a perforated cloth and obtaining the filtrate containing pectin, then precipitating the pectin for all samples by adding a similar volume of ethanol, a concentration of 98% for the first three methods, and at a ratio of 1:2 (volume: volume) for the fourth method, and the samples were left in the refrigerator for 60 minutes, thenthe samples were filtered through filter paper (Whatman) No. (1), then The resulting precipitate (pectin) was separated and dried at a temperature of 50 °C until drying, then crushed and stored in labeled and sealed plastic containers until use. The percentage of extracted pectin was calculated by adopting the following equation:

$$\text{Pectin percentage } \% = \frac{\text{dried pectin weigh}}{\text{dry peels powder weight}} \times 100$$

3- Determination of some physical properties of pectin

3-1: Determination of the viscosity of pectic solutions

The viscosity of the pectin samples extracted from all the samples under study was estimated according to the method (Yoo et al., 2006), by dissolving 1 g of pectin in 100 ml of distilled (pure) water at a temperature of 25 ° C, and it was passed through an Ostwald tube with a capacity of 15 ml and was calculated The flow time of the pectin solution and distilled water, and the viscosity of the prepared pectin solutions were extracted from the following equation:

$$N = d_1\, t_1 / d_2\, t_2$$

As N represents the relative viscosity d1 - density of distilled water d2_ is the density of the solution t1 - Distilled water run-off time

t2 - time of perfusion of the solution

3-2 Estimation of gel formation time

The holding time was estimated according to the Mc Cready method(1970), as the gel was prepared from the pectin samples under study, by mixing 0.5 g of each of pectin and citric acid, then adding 32.5 g of sucrose and 10 ml of distilled water and stirring the mixture on a hot magnetic stirrer at a temperature 106 ° C, then according to the formation time, from the moment the materials are poured into the packing container until the time of the formation by using a stopwatch.

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4-Functionalpropertiesofpectin4-1 Emulsifying activity

The method described by Hosseini et al. (2016) was followed by mixing 0.5 g of pectin extracted from the samples under study with 100 ml of distilled water containing sodium azide (prepared by dissolving 0.02 g of sodium azide in 100 ml of distilled water). 5 ml of the solution was placed in a 15 ml centrifugal tube, then 5 ml of sunflower oil was added to it, then the mixture was mixed well and then centrifuged at 4000 rpm for 5 minutes. The activity of the emulsion was calculated through the following equation:

$$\text{Emulsifier activity } \% = \frac{\text{emulsion layer volume}}{\text{total volume liquids}} \times 100$$

4-2 Estimation of Pectin solubility in hot and cold water

The method of Bhavya et al. (2015) was followed by dissolving 0.3 g of the pectin samples under study in 10 ml of ethanol at a concentration of 95%, then adding 50 ml of distilled water while stirring the mixture on a magnetic stirrer with a cold surface at room temperature and following its dissolution, after which the mixture was heated On a hot surface magnetic stirrer at 85-95°C for 15 minutes, the solubility was recorded by follow-up with increasing temperatures.

4-3 The Ability of Pectin solubility in hot and cold alkali

Bhavya et al. (2015) method was followed, by dissolving 0.1 g of the pectin samples under study in 10 ml of 0.1 N sodium hydroxide solution while stirring the mixture on a magnetic stirrer at room temperature and continuing its dissolution, then heated using a magnetic stirrer with a hot surface at a temperature of 85-95 °C for 15 minutes and the solubility was recorded by follow-up.

4-4: Water holding capacity (WHC) of the extracted pectin

The method of Bayar et al. (2018) was followed with some modification by dissolving 1 g of the pectin samples under study in 10 ml of distilled water in a centrifuge tube and then centrifuged for 20 minutes at 300 x g, then the centrifuge was tilted at an angle of 45 degrees to get rid of the the excess water was not bound for 30 minutes on filter paper and then the increase in weight of pectin was recorded as g of absorbed water.

4-5: Oil holding capacity (OHC) of the extracted pectin

The method of Bayar et al. (2018) was followed, by dissolving 1 g of the pectin samples under study in 10 ml of sunflower oil (oil density 0.9 g / ml) in a centrifuge tube and then centrifuged at 300 x g for 20 minutes, then the tube wastilted centrifuge at a 45° angle to get rid of the unbound oil for 30 minutes on filter paper and then the weight gain of pectin was recorded as g of absorbed oil.

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Results and Discussion

1-Efficient extraction of pectin

The results in Figure (1) show the percentages of the pectic material yield from the peels of the samples under study, using different solutions that included ammonium oxalate, citric acid, oxalic acid and hydrochloric acid at a temperature of 90 °C for 90 minutes. As it is noted, the citric acid is distinguished by extracting the highest percentage of pectin. It reached 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.

When extracting 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%. Extraction with oxalic acid gave a pectin percentage of 29.80% for eggplant peels and 28.69% for orange peels, Pomegranate 18.03%, pomelo 34.31%, pumpkin 21.37%, and watermelon 17.5%. The lowest percentage was when extracted with hydrochloric acid, as it reached 16.6% for eggplant peels, 24.1% for oranges, 6.6% for pomegranate, pomelo 21.6%, pumpkin 7.5%, and for watermelon peels 10%.

The results show that the best extraction solution was using citric acid, and this was confirmed by Kazemi[b] et al. (2019). The percentage obtained was lower than what was found in this study, reaching 29.17%, followed by extraction with ammonium oxalate. The lowest percentage obtained when extracting with hydrochloric acid.

The percentages extracted from pumpkin peels were also higher than what was obtained by Hamed et al (2017) and Don, (2019) when extracted with citric acid and nitric acid, which amounted to (6.80 and 7.72%), and when extracted with citric acid and hydrochloric acid were (7.30 and 11.04%) respectively.

Lee and choo (2020) indicated that the percentage of pectin in watermelon peels when extracted with citric acid and hydrochloric acid was (8.38 and 6.52%), respectively. The percentage of pectin extracted from orange peels in this study was higher than what was obtained by Tiwari et al (2017) and Alamineh et al (2018), which amounted to 52.90% and 14.3%, respectively, while the percentage of pectin extracted from pomelo was higher than that obtained by Quoc et al (2015) and Roy et al (2017), which amounted to 23.83 and 16.07%, respectively. The percentage of pectin that was extracted from pomegranate peels in this study is higher than that obtained by Cheikhrouhou et al (2017), Sari and Birlik (2020), which were 10.1-6.8 and 25.96%, respectively, when they studied pectin extracted from pomegranate peels.

The conditions of extraction, the concentration of the acid or the extraction solution, and the time are among the important factors affecting the extraction process. The high percentage of the product using citric acid is due to it being a natural acid present in plants, as the acidic function is among the natural conditions of 1.5 plants. Of course it does not affect the pectin. This was confirmed by Canteri - Schemin et al (2005) in the lack of effect of citric acid on pectin upon extraction.

When using oxalic acid, which gave medium values as an acid, it plays a role in the formation of insoluble compounds such as calcium and magnesium oxalates, in addition to extracting free pectic substances. as well as it is important in disengaging metal ions from pectin materials and their ease

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of dissolution (Saget, 1988). It also leads to an increase in the ionization of acidic groups in pectic chains, and thus easy loss of metal ions (Kliemann et al, 2009).

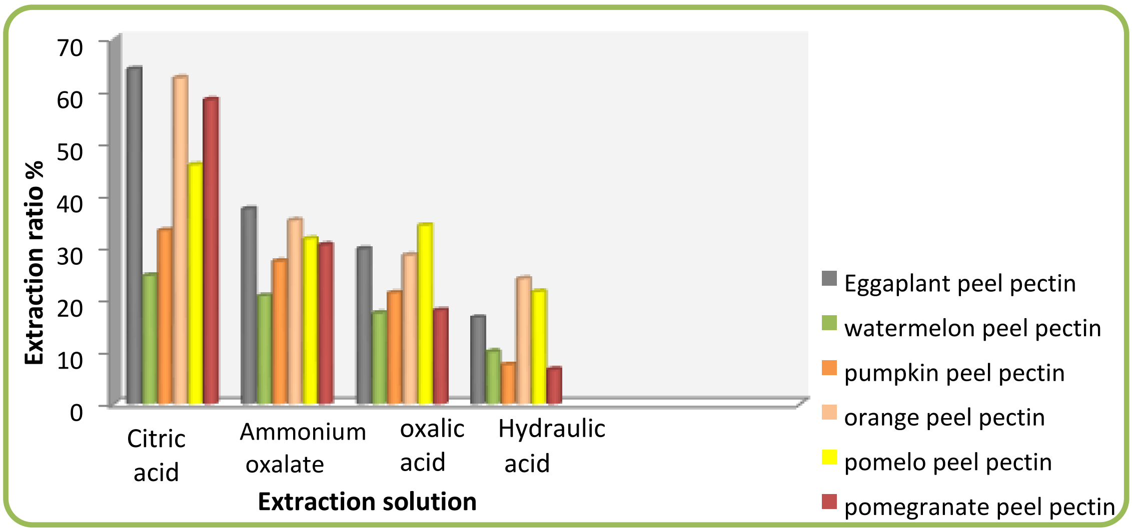

Figure (1) The effect of using different solutions on the percentages of pectin yield from the peels of the samples under study

2- Properties physical of pectin

2-1 RelativeViscosity of pectin

The results in figure (2) show the viscosity values of pectin samples extracted from the peels of the plants under study, estimated by the time of flow of the solution in Astoled tube. As the figure shows, the viscosity values for all pectin samples extracted using citric acid and for all samples are higher than their values for the same samples extracted using ammonium oxalate. The value of the viscosity of pectin extracted using citric acid from eggplant peels was 1.5208 cp and watermelon 1.7250 cp and pumpkin 1.6958 cp, orange 1.7416 cp and pomelo 2.1208 cp and pomegranate 1.3916 cp, compared to pectin extracted with ammonium oxalate that is amounted in eggplant peels. 1.2161 cp, watermelon peels 1.6083 cp, pumpkin 1.0872 cp, orange 1.5941 cp and pomelo 1.7875 cp and pomegranate 1.3041 cp. Balsslsse and Fahloul (2018) and Al-Ali et al., 2012) indicated that the viscosity of pectin extracted from peels of pumpkin was
5.65, and the viscosity of pectin extracted with citric acid from pomegranate peels was 1.1965 cp, and the pectin of watermelon peels was 1.2002 cp.

The reason for the difference in the viscosity values of the samples under study compared with previous studies may be due to the difference in the percentage of impurities, which is the increase in the percentage of ash, which obscures the viscous behavior of calactronic acid, as well as the decomposition that occurs on pectinia materials due to the use of different acids when extracting (Kareem, 2018).

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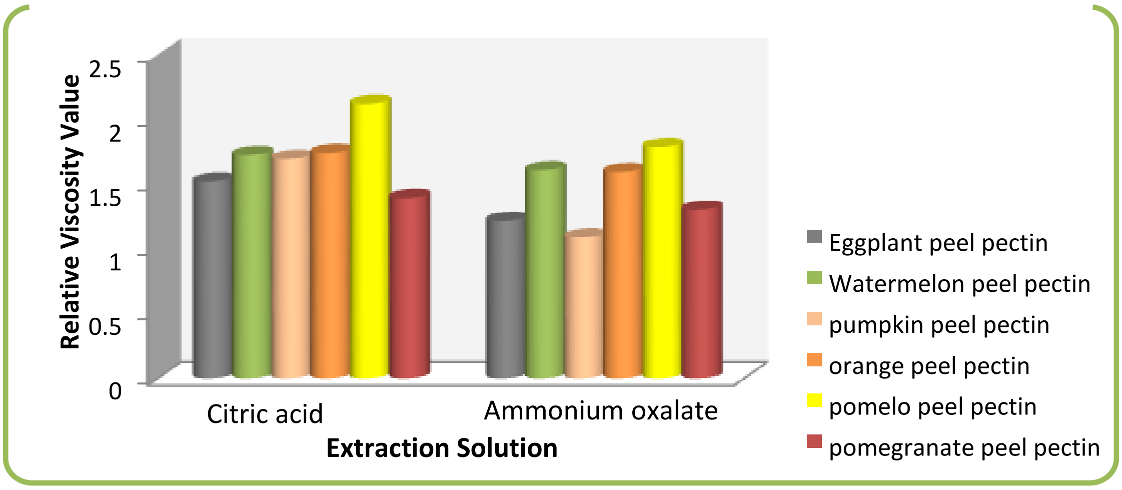

Figure (2) Viscosity values of pectins extracted from the peels of the samples under study using citric acid and ammonium oxalate.

2-2 Time of holding

Figure (3) shows the holding time of pectin extracted from the peels of the plants under study using citric acid and ammonium oxalate.). The values, when extracting with citric acid, of the pectin of the peels of eggplant, watermelon, pumpkin, orange, pomelo and pomegranate were (8, 7, 8, 7, 8, and 7) minutes, respectively, while when extracting with ammonium oxalate the pectin of the peels of eggplant, watermelon, pumpkin, orange, pomelo and pomegranate it was (10, 6, 7, 8, 7, 6) minutes, respectively.

These times were similar to what was obtained by Kareem (2018) and Al-Ali et al. (2012), when they studied the time of holding the gel for green, yellow and black banana peels, which amounted to (8, 10, 9) minutes, and the time for the holding time of the pectin of pomegranate peel to 12 minutes, respectively.

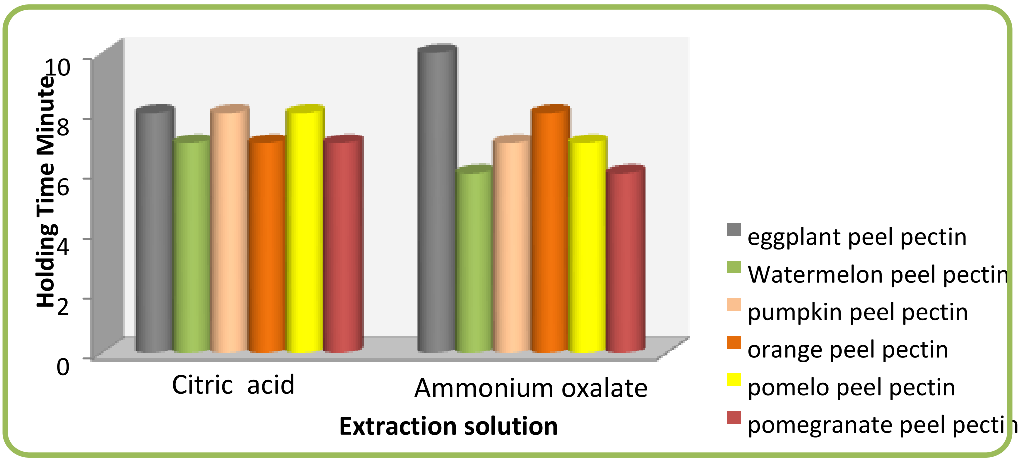

Figure (3) Holding time of pectin extracted from the peels of the samples under study using citric acid and ammonium oxalate.

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3- Functional properties of pectin

3-1 Emulsifying activity(EA)

Table (1) shows the emulsifying activity of pectin samples extracted from the peels of the samples under study. It is also noted that the emulsifying activity of all pectin samples extracted from the plants under study when extracted using citric acid compared with the activity of pectin extracted using ammonium oxalate.

Noting the superiority of the pectin of orange peels, then pomelo, and eggplant when extracted with citric acid, and the superiority of eggplant and orange peels when extracted with ammonium oxalate. The pectin of eggplant peels recorded values of 55% and 53% when extracted with citric acid and ammonium oxalate, respectively.

While the values were for watermelon peel pectin 52% and 47%, pumpkin peel pectin 54% and 46%, pomelo peel pectin 57% and 48%, orange peel pectin 59% and 54%, and pomegranate peel pectin 50% and 44% upon extraction with citric acid and ammonium oxalate, respectively.These results converge with what was mentioned by Kasmi[b] et al (2019) and Asgari et al. (2019) when they studied the emulsifying activity of eggplant peel pectin, which is 58.17%, and walnut peel pectin amounted to 54.26%, respectively.

These differences in the emulsifying ability of pectin samples may be attributed to the difference in their content of protein and calactronic acid, as well as the percentage of methoxyl, as they have a positive effect on this activity, and this is what was indicated by each (Elleuch et al., 2011 and Rubio et al., 2015).

Table (1) Emulsifying activity of pectin extracted from the peels of the samples under study using citric acid and ammonium oxalate

Sour type / Samples	Pectin extracted with citric acid	Pectin extracted with ammonium oxalate
	Emulsifier activity (EA) %	**Emulsifier activity (EA) %**
Eggplant peels	55	53
Watermelon peels	52	47
Pumpkin peels	54	46
Orange peels	59	54
Pomelo peels	57	48
Pomegranate peels	50	44

3-2: The solubility of pectin in hot and cold water

The results in Table (2) show the solubility of the pectin extracted from the peels of the samples under study, each of citric acid and ammonium oxalate. As the pectin extracted using citric acid and ammonium oxalate from the peels of watermelon, orange and pomegranate, recorded a slight Insolubility, while the rest of the samples recorded dissolution in cold water, but when water was heated, all pectin samples recorded total solubility. These results agreed with what wasmentioned by

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Udoune et al. (2016) and Bagade et al. (2017) when they studied lemon peel pectin and orange peel pectin, respectively.

3-3: The solubility of pectin in hot and cold alkali

The results in Table (2) show the solubility of pectin extracted from the peels of the samples under study for both citric acid and ammonium oxalate. The pectin extracted with citric acid and ammonium oxalate from the peels of eggplant, watermelon, pumpkin, orange, pomelo and pomegranate recorded a slight solubility in the cold alkali. When using heating, all pectin samples recorded total solubility, and these results agreed with what was mentioned by Udoun et al. (2016), Bagade et al. (2017) and Guzel and Kim (2019) when they studied the pectin of lemon peel, orange and pomegranate,respectively.

Table (2) Solubility of pectin extracted with citric acid and ammonium oxalate from the husks of the samples under study in water and hot and cold alkali

Solvent type Cold water Hot water Cold Alkali Hot Alkali sample

Eggplant peel Insoluble Completely Little melted Completely **pectin** melted melted
Watermrlon Little melted Completely Little melted Completely **peel pectin** melted melted
Pumpkin peel Insoluble Completely Little melted Completely **pectin** melted melted
Orange peel Little melted Completely Little melted Completely **pectin** melted melted
Pomelo peel Insoluble Completely Little melted Completely **pectin** melted melted
Pomegranate Little melted Completely Little melted Completely **peel pectin** melted melted

3-4 Water-holding capacity(WHC) for Pectin

The results in Table (3) show the water-holding ability of pectin extracted from the peels of the samples under study using citric acid, as it was for eggplant peels 4.68 gm, watermelon 3.37 gm, pumpkin 2.65 gm, orange 2.53 gm, pomelo 4.57 gm, and pomegranate 2.43 gm, while the pectin extracted with the ammonium oxalates was 3.88 gm for eggplant peels, 1.23 gm for watermelon, 2.28 gm for pumpkin, 1.37 gm for oranges, 2.03 gm for pomelo, and 1.95 gm for pomegranate. As it is noted from these results, the high water holding capacity of pectin extracted using citric acid and for all samples compared with pectin extracted using ammonium oxalate for the same samples. This may be attributed to the lower humidity in pectin samples extracted using citric acid, which was confirmed by the results obtained compared with pectin extracted using ammonium oxalate.

These results agreed with what Don et al. (2019) mentioned when they studied the pectin of pumpkin peels, which amounted to 2.51 g, and was lower than what was indicated by Dias et al. (2020) and Kazemi[b] et al. (2019) when they studied the pectin of orange and eggplant peels to a ratio of 6.33 g and 6.02 g respectively.

3-5 Oil-holding capacity(OHC) for Pectin

The results in Table (3) show the ability to hold the oil for pectin extracted from the peels of the samples under study with citric acid and ammonium oxalate.

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The superiority of pectin from the peels of watermelon, pumpkin, orange and pomegranate extracted using ammonium oxalate compared with the same samples extracted using citric acid. Eggplant and pomelo had higher oil holding capacity in samples extracted using citric acid. Eggplant peels had 1.82 g, watermelon had 1.12 g, pumpkin 1.15 g, orange 1.37 g, pomelo 1.49 g, pomegranate 1.17 g, for pectin extracted with citric acid, while for pectin extracted with ammonium oxalate, eggplant peels had 1.46 g, watermelon 1.45 g, pumpkin 1.5 g and orange 1.64 g, pomelo 1.4, and pomegranate 1.67 g. These results converge with what Jayani et al. (2019) mentioned when they studied pumpkin pectin, when they reached a value of 1.76 g. Kazemi[b] et al. (2019) and Dias et al. (2020) and Gannasin et al. (2012) indicated when they studied pectin Peel of the eggplant, orange and apple, the oil holding capacity is 2.60g, 2.40g, and 2.11g, respectively.

Table (3) The ability to hold water or oil for pectin extracted from the peels of the samples under study using citric acid and ammonium oxalate

Samples	**Pectin extracted with citric acid**		**Pectin extraction with ammonium oxalate**	
	WHC	**OHC**	**WHC**	**OHC**
Eggplant peels	4.68	1.82	3.88	1.46
Watermelon peels	3.65	1.12	1.23	1.45
Pumpkin peels	2.65	1.15	2.28	1.5
Orang peels	2.53	1.26	1.37	1.64
Pomelo peels	4.57	1.49	2.03	1.4
Pomegranate peels	2.43	1.17	1.95	1.67

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