Physicochemical, Rheological, and Functional Properties of Quince Peel Pectin Extracted via Conventional and Microwave-Assisted Citric Acid Methods

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Abstract

This study compared conventional acid extraction and microwave-assisted acid extraction in terms of their effects on the physicochemical, rheological, and functional properties of pectin recovered from quince peels. Quince peel powder was extracted using 0.1 M citric acid at pH 2. Conventional extraction was conducted at 85°C for 60 min, whereas microwave-assisted extraction was performed at 600 W for 4 min. The recovered pectin was precipitated with ethanol, freeze-dried, and compared with standard high- and low-methoxyl pectins. Moisture content, solubility, free carboxyl groups, pH, viscosity, gel strength, cohesiveness, elasticity, and purity were evaluated. The moisture contents of acid- and microwave-extracted pectins were 10.34 and 10.36%, respectively, with no significant difference between them. Microwave-extracted pectin showed the highest solubility at 91.00%, which was comparable to that of standard high-methoxyl pectin at 90.73% and higher than that of acid-extracted pectin at 85.01%. The viscosity values ranged from 4.36 to 10.65 mPa·s, with microwave-extracted pectin recording 10.64 mPa·s, close to the value of standard high-methoxyl pectin. Microwave-extracted pectin also exhibited greater gel strength than acid-extracted pectin, reaching 283.60 and 257.00 g, respectively. No significant differences were observed in cohesiveness among the samples. Pectin purity was 88.25% for microwave-assisted extraction and 88.08% for conventional acid extraction. HPLC analysis confirmed the presence of galacturonic acid, with microwave-extracted pectin showing the same retention time as the standard compound. These findings indicate that microwave-assisted acid extraction can substantially reduce extraction time while producing quince peel pectin with favorable solubility, viscosity, gel strength, and purity.

Keywords: Quince peel, Citric acid extraction, microwave-assisted extraction, Food hydrocolloids, Galacturonic acid, Gel properties.

Introduction

The increase in fruit processing has increased the amount of organic waste generated from peeling, juicing and processing which has led to research into practical ways of utilising it instead of discarding it as waste. Fruit peels are by-products of plants that can be used to isolate value-added compounds such as dietary fibre, phenolic compounds and polysaccharides. Quince fruit (Cydonia oblonga Mill.) contains relatively high levels of pectin and its processing into jams, jellies and juices produces quantities of peel and solid waste that could be used as an alternative source of pectin for the food industry. Recent studies have shown that the use of quince waste can contribute to reduce the environmental burden and increase the economic value of this waste in the framework of the circular economy [20].

Pectin is a complex polysaccharide that is found mainly in the primary cell walls and

ISSN 2072-3857

middle lamella of plant tissues. The structure consists mainly of galacturonic acid units linked by glycosidic bonds and varying amounts of neutral sugars. This is important from a practical point of view because it can increase viscosity, form gels, stabilise emulsions, and improve the texture of food products. Thus, pectin is utilised in the manufacturing of jams, jellies, confectioneries, dairy products and beverages, and in its contemporary applications in food coatings and carrier systems for active ingredients. The efficacy of the application of pectin is based on its chemical structure, molecular weight, degree of esterification, and distribution of carboxyl groups on the molecular chain [4].

The properties of pectin are dependent not only on the plant source but also on the type of tissue, stage of maturity, pre-extraction treatments and conditions of separation and purification. A recent study on quince pectin showed a high degree of methylation of the pectin and the solutions showed a quasi-plastic behaviour, indicating its potential application as a thickening agent in some food products. However, the physicochemical and rheological properties of quince pectin may vary depending on the plant part used and the extraction conditions. Variations in extraction pH, temperature, and duration can influence the molecular structure, degree of esterification, and galacturonic acid content of pectin, thereby affecting its solubility, viscosity, and gel-forming ability. Therefore, post-extraction characterisation is essential to assess its quality and determine its suitability for specific food applications [8].

Heating acid extraction is one of the most commonly used traditional methods for obtaining pectin from plant sources. The method consists in treating the raw material in an acidic medium and at high temperatures for a sufficient time to convert the insoluble protopectin into soluble pectin, separating the extract and precipitating the pectin with alcohol. Although this method is easy to apply and widely used, prolonged exposure to acidity and heat may lead to the decomposition of some molecular chains or change the degree of esterification, and the long extraction period and increased energy and solvent consumption are among its most prominent limitations. Therefore, modern research has turned to developing faster and more efficient methods, while preserving as much as possible the functional properties of the extracted pectin [22].

Microwave-assisted extraction is one technique used to reduce pectin extraction time and improve the transfer of the material between plant tissue and solvent. Polar molecules absorb electromagnetic energy and convert it into heat within the medium, leading to rapid heating, increased pressure inside plant cells, and weakening of cell walls, thus facilitating the release of pectin into solution. Some studies have shown that microwave extraction can provide suitable yields with short extraction times, producing pectin with physicochemical and rheological properties suitable for use in food systems [25].

However, using a microwave does not necessarily guarantee better pectin in all cases, as the efficiency of the process depends on the equipment power, exposure time, temperature, pH, solid-to-solvent ratio, and the insulating properties of the raw material. Excessive power or treatment time can lead to pectin chain degradation and a decrease in molecular weight, which may negatively affect viscosity and gel formation. Therefore, microwave-assisted extraction should be evaluated based on the quality and functional properties of the

ISSN 2072-3857

resulting pectin, rather than solely on extraction speed or yield [17].

Despite numerous studies on pectin extraction from citrus and apple peels and some plant residues, information on directly comparing conventional acid extraction with microwave-assisted acid extraction of quince peel pectin remains limited. Furthermore, most studies have focused on extraction yield and certain chemical properties, with less attention paid to integrated evaluations that combine solubility, acidity, pH, viscosity, gelling properties, and purity, as well as comparisons of the extracted pectin to standard types. These aspects represent a significant knowledge gap, as the suitability of pectin for food use is more closely related to its quality and functional behavior than solely to the quantity produced.

Therefore, the current study aimed to compare the effect of conventional acid extraction and microwave-assisted acid extraction using a microwave oven (Midea Group, China) operated at 600 W for 4 min on the physicochemical, rheological, and functional properties of quince peel pectin. The evaluation included moisture content, solubility, free carboxyl groups, pH, viscosity, gel strength, cohesion, and elasticity, along with estimating the pectin purity using high-performance liquid chromatography (HPLC) and comparing the results with standard high- and low-methoxyl pectin.

Material and Methods

Sample Collection and Preparation:

Forty-five kilograms of quince fruit were collected from local markets in Mosul. The undamaged fruit was selected, washed, and peeled by hand. The peels were then scalded with hot water at 90–95°C for 2–5 minutes, followed by immediate cooling with ice water to minimize heat retention.

The peels were then dried at 50°C until a uniform weight was achieved. The resulting powder was ground and sieved through a 30-mesh sieve to obtain relatively homogeneous particles. The powder was stored in airtight containers in a cool, dry, and dark place until needed for extraction.

Pectin Extraction:

Pectin was extracted from quince peel powder using two methods: traditional acid extraction and microwave-assisted acid extraction, with the aim of comparing the effect of the extraction method on the properties of the resulting pectin.

Conventional Acid Extraction:

Traditional acid extraction was performed using the method described by [18], with some modifications to suit the study conditions. Ten grams of quince peel powder were mixed with 250 mL of 0.1 M citric acid solution, and the pH of the solution was adjusted to 2. The mixture was heated at 85°C for 60 minutes, with continuous stirring throughout the extraction process to ensure homogeneity of the sample and maximize contact between the peel powder and the acidic solution.

Microwave-Assisted Acid Extraction:

Microwave-assisted acid extraction was performed according to the method described by [11], with some modifications. The same ratio of peel powder to acid solution used in conventional extraction was employed; 10 g of peel powder was mixed with 250 mL of 0.1 M citric acid solution at pH 2. The

mixture was microwaved at 600 W for 4 minutes. After treatment, the extract was allowed to cool before proceeding to filtration and pectin recovery.

Pectin Recovery and Drying:

The extracts obtained from both methods were cooled and then filtered to separate the remaining solids from the pectin-containing filtrate. The pectin was precipitated by adding 95% ethanol to the filtrate at a ratio of 1:2 (filtrate to ethanol), and the mixture was incubated at 4°C for 12 hours to allow sufficient time for precipitation. The pectin precipitate was centrifuged at 4000 rpm for 40 minutes. The precipitate was washed three times with 95% ethanol to reduce associated materials and soluble impurities. The pectin was then frozen at −18°C for 24 hours and freeze-dried to constant weight. The dried pectin was ground and stored in airtight glass containers until testing.

Moisture Content Determination:

The moisture content of the pectin samples was estimated according to the method adopted by the [2]. The estimation was performed with three replicates for each sample, and the moisture content was expressed as a percentage of the sample weight, according to the following equation:

$$Moisture\ content\ \% = \frac{W_1 - W_2}{W_1} \times 100$$

W_1= weight of the sample before drying (g).

W_2= weight of the sample after drying (g).

Solubility Determination:

The solubility of the pectin samples was estimated using the method described by [26], with some modifications. Distilled water was used as the solvent, and freeze-drying alone was used instead of freeze-drying followed by oven drying. The estimation involved centrifuging the insoluble fraction of the pectin, followed by drying and weighing. The soluble fraction was calculated as the difference between the initial weight of the dry sample and the weight of the insoluble fraction after drying. Measurements were performed in triplicate for each sample, and the solubility was expressed as a percentage using the following equation:

$$Pectin\ solubility\ \% = \frac{W_i - W_u}{W_i} \times 100$$

W_i = initial dry pectin weight before dissolution (g).
W_u = weight of insoluble pectin remaining after centrifugation and drying (g).

Determination of Titratable Acidity and Free Carboxyl Groups:

The titration acidity, expressed as the free carboxyl groups in pectin, was estimated using a basic titration method as described by [21], with some modifications to suit the study conditions. The results are expressed as a percentage equivalent to anhydrogalacturonic acid units, based on their equivalent weight, according to the following equation:

$$Titratable\ acidity\ \% = [\frac{V \times N \times 176}{W}] \times 100$$

V = volume of alkali used.
N = normality of the alkali.
W = sample weight (g).
176 = equivalent weight of an anhydrogalacturonic acid unit (g/mol).

pH Measurement:

The pH value of the pectin samples was measured using the method described by [1]. The measurement was performed with three replicates for each sample, and the average of the readings was used to express the results.

ISSN 2072-3857

Viscosity Measurement:

Pectin solutions were prepared according to the method described by [6], with the solution concentration adjusted to 2% w/v. The viscosity of the solutions was measured at 25°C using a HAAKE Viscotester 3 rotary viscometer, manufactured by Thermo Fisher Scientific, with spindle R2 at a constant speed of 20 rpm, according to the manufacturer's instructions. Measurements were performed in triplicate for each treatment, and readings were initially recorded in deciPa·s (dPa·s) and then converted to milliPa·s (mPa·s).

Determination of Gel Strength, Cohesiveness, and Elasticity:

The gel strength and texture characteristics of extracted pectin samples, as well as high-methoxyl and low-methoxyl standard pectin, were evaluated in the graduate laboratory of the College of Food Sciences at Al-Qasim Green University in Babylon Governorate. A Brookfield CT3 texture analyzer from AMETEK was used for the measurements.

The gel samples were prepared according to the standard method described by [14], with the ingredient quantities adjusted to achieve a final weight of 100 g per sample. Distilled water was used instead of deionized water, and citric acid was added to all samples to standardize the conditions before measurement. After gel formation, each sample was placed in the center of the analyzer platform and subjected to two consecutive compression cycles using the TA10 probe at a speed of 1 mm/s and a deformation of 30% of the sample height. Measurements were performed in triplicate for each treatment, and gel strength, cohesion, and elasticity values were recorded directly from the apparatus. Gel strength was considered an indicator of the strength of the formed gel network, while cohesion reflected the extent to which the gel maintained its internal bonds after compression. Elasticity represented the gel's ability to return to part of its original shape after the removal of the applied force.

Determination of Pectin Purity Using High-Performance Liquid Chromatography:

Pectin purity was estimated using the method described by [12], with some modifications to suit the study conditions. The analysis was performed using an Agilent 1100 HPLC instrument manufactured by Agilent Technologies, Palo Alto, CA, USA, and equipped with ChemStation software for instrument operation and data processing.

Galactronic acid was identified in the samples by comparing the retention time of the peak observed in the sample chromatogram with that of the standard compound. Pectin purity was calculated as a percentage using the following relationship:

$$Pectin\ purity\ \% = \frac{W_g}{W_t} \times 100$$

Wg = weight of the separated fraction containing galacturonic acid (g).
Wt = total weight of the pectin sample before separation (g).

Statistical Analysis:

All measurements were performed in triplicate, and results were presented as mean ± standard deviation. Means were compared using Duncan's multiple range test, and a probability level of 0.05 was used to determine the significance of differences between treatments. IBM SPSS Statistics 26 software was used for statistical analysis. Means with different letter combinations within the same

ISSN 2072-3857

attribute were considered significantly different at the established probability level.

Results and Discussion

Effect of Pectin Type and Extraction Method on Moisture Content and Solubility:

Table (1) shows significant differences (P<0.05) in the moisture content of the pectin samples. High-methoxyl standard pectin recorded the highest average moisture content at 11.90%, followed by low-methoxyl standard pectin at 11.61%. Acid-extracted and microwave-extracted pectin recorded the lowest values at 10.34% and 10.36%, respectively, with no significant differences between them (P>0.05). The lower moisture content of the extracted pectin may be attributed to the efficiency of freeze-drying in removing water by sublimation under vacuum. These results are consistent with those of Ratti and [7, 27] regarding the effectiveness of this technique in reducing water content. The similarity of acid pectin and microwave pectin also indicates that the extraction method did not clearly affect moisture after uniform drying, and all samples were within the limits of the [13], as they did not exceed the maximum limit of 12%.

Regarding pectin solubility, the results showed significant differences (P<0.05) between the samples. Microwave-extracted pectin exhibited the highest solubility at 91.00%, followed by high-methoxyl standard pectin at 90.73%, with no significant difference between them (P>0.05). Acid-extracted pectin showed a solubility of 85.01%, while low-methoxyl standard pectin recorded the lowest value at 66.82%. The observed differences in solubility may be attributed to variations in pectin molecular organisation and degree of esterification, which influence polymer-chain hydration and aggregation in water. The higher solubility of microwave-extracted pectin may also be related to rapid internal heating, which promotes disruption of the quince peel matrix and facilitates pectin release while limiting prolonged exposure to acidic heating. In contrast, the lower solubility of low-methoxyl pectin may result from stronger intermolecular and ion-mediated associations that form compact aggregates and restrict water penetration and dispersion [11, 26].

Table 1. Effect of Pectin Type and Extraction Method on Moisture Content and Solubility of Pectin

Sample Type	Moisture %	Solubility %
Standard High-Methoxyl Pectin	11.90 ± 0.010 a	90.73 ± 0.015 a
Standard Low-Methoxyl Pectin	11.61 ± 0.028 b	66.82 ± 1.633 c
Acid-Extracted Pectin	10.34 ± 0.023 c	85.01 ± 0.916 b
Microwave-Extracted Pectin	10.36 ± 0.011 c	91.00 ± 0.714 a

 Values represent means ± standard deviation of three replicates.

** Different lowercase letters within the same column indicate significant differences at a probability level (P < 0.05), while similar letters indicate no significant differences according to Duncan's multiple range test.

ISSN 2072-3857

Effect of Pectin Type and Extraction Method on Titratable Acidity and pH:

Table 2 shows significant differences (P<0.05) in titrating acidity, expressed as free carboxyl groups (-COOH), among the pectin samples. The standard low-methoxyl pectin recorded the highest average acidity of 5.68%, significantly outperforming the other samples. This is attributed to its lower esterification level of 35.29%, which increased the proportion of titrizable free carboxyl groups. This finding is consistent with the findings of [3,24], who reported an inverse relationship between the degree of esterification and the free carboxyl group content. In contrast, no significant differences (P>0.05) were found between standard high-methoxyl pectin, acid-extracted pectin, and microwave-extracted pectin, with average esterification rates of 4.74%, 4.67%, and 4.69%, respectively. This is attributed to the similarity in esterification rates of 68.75%, 60.49%, and 68.88%, respectively. The similarity between microwave-extracted pectin and standard high-methoxyl pectin also indicates the ability of microwave extraction to preserve the pectin's functional groups. These results underscore the importance of free carboxyl groups as a chemical indicator related to the degree of esterification and the functional properties of pectin [3]

Table 2. Effect of pectin type and extraction method on titration acidity (free carboxyl groups) and pH

Sample Type	Free carboxyl groups (%) (-COOH)	pH
Standard High-Methoxyl Pectin	4.74 ± 0.190 b	3.26 ± 0.115 b
Standard Low-Methoxyl Pectin	5.68 ± 0.110 a	4.53 ± 0.057 a
Acid-Extracted Pectin	4.67 ± 0.036 b	3.30 ± 0.100 b
Microwave-Extracted Pectin	4.69 ± 0.052 b	3.26 ± 0.115 b

 Values represent means ± standard deviation of three replicates.

** Different lowercase letters within the same column indicate significant differences at a probability level (P < 0.05), while similar letters indicate no significant differences according to Duncan's multiple range test.

The results in the same table indicate significant differences (P<0.05) in pH values among the pectin samples. Standard low-methoxyl pectin recorded the highest average pH of 4.53, significantly exceeding the other samples. Microwave-extracted pectin recorded the lowest average pH of 3.26, with no significant difference from standard high-methoxyl pectin, while acid-extracted pectin had an average pH of 3.30. This variation is attributed to differences in chemical composition, degree of esterification, and the nature of the functional groups, as well as the effect of the extraction method on the physicochemical properties of the pectin. The pH values of standard high-methoxyl pectin and microwave-extracted pectin were similar, consistent with their similar esterification degrees of 68.75% and 68.88%, respectively. Acid-extracted

ISSN 2072-3857

pectin recorded an intermediate pH, which may be related to the extraction conditions.

The effect of pectin type and extraction method on rheological properties (viscosity, gel strength, cohesion and elasticity):

The results in Table (3) show a variation in the apparent viscosity of 2% pectin solutions, with values ranging from 4.36 to 10.65 mPa·s. High-methoxyl standard pectin recorded the highest value at 10.65 mPa·s, followed by microwave-extracted quince pectin at 10.64 mPa·s, and then acid-extracted pectin. Low-methoxyl standard pectin recorded the lowest value at 2.36 mPa·s.

Table 3. The effect of pectin type and extraction method on rheological properties (viscosity, gel strength, cohesion and elasticity)

Sample Type	Viscosity (mPa·s)	Gel strength (g)	Cohesion	Elasticity (mm)
Standard High-Methoxyl Pectin	10.65 ± 0.020 a	308.33 ± 8.504 a	0.51 ± 0.026 a	2.50 ± 0.529 b
Standard Low-Methoxyl Pectin	4.36 ± 0.040 c	199.60 ± 5.351 d	0.56 ± 0.041 a	3.40 ± 0.251 a
Acid-Extracted Pectin	10.56 ± 0.050 b	257.00 ± 12.288 c	0.50 ± 0.072 a	2.40 ± 0.702 b
Microwave-Extracted Pectin	10.64 ± 0.010 a	283.60 ± 10.479 b	0.52 ± 0.045 a	2.70 ± 0.264 b

 Values represent means ± standard deviation of three replicates.
** Different lowercase letters within the same column indicate significant differences at a probability level ($P < 0.05$), while similar letters indicate no significant differences according to Duncan's multiple range test.

The texture analysis results in Table (3) showed significant differences ($P<0.05$) in gel strength between the samples. High-methoxyl standard pectin recorded the highest value at 308.33 g, followed by microwave-extracted pectin at 283.60 g, and then acid-extracted pectin at 257.00 g. Low-methoxyl standard pectin recorded the lowest value at 199.60 g. The superiority of high-methoxyl pectin is attributed to its higher degree of esterification and its role in forming a strong gel network in the presence of sucrose and acid, which is consistent with the findings of [23]. The increased gel strength of microwave-extracted pectin compared to acid-extracted pectin also indicates that it retains better functional properties, resulting from the effect of the extraction method on the pectin's properties and gelling behavior [5, 16].

As for cohesion, no significant differences were observed between the samples ($P>0.05$), with values ranging from 0.50 to 0.56, indicating similar internal bonding of the gel networks despite variations in gel strength. Standard low-methoxyl pectin exhibited the highest elasticity at 3.40 mm, compared to standard high-methoxyl pectin and acid- and microwave-extracted pectin, which reached 2.50, 2.40, and 2.70

ISSN 2072-3857

mm, respectively. This may be attributed to low-methoxyl pectin's reliance on ionic bonds with calcium, granting it greater resistance to deformation. These results are consistent with those reported by [9, 10].

Effect of Pectin Type and Extraction Method on Pectin Purity:

The results of the statistical analysis shown in Table (4) revealed significant differences ($P<0.05$) in pectin purity depending on its type and extraction method. Pectin extracted from quince waste using the microwave method recorded a purity of 88.25%, compared to 88.08% for pectin extracted using the acid method. This may be attributed to the shorter extraction time and reduced heat exposure, which could limit the transfer of non-pectic compounds into the extract. The purity of the extracted samples was lower than that of the standard high-methoxyl and low-methoxyl pectins, which recorded 90.73% and 89.08%, respectively.

Table 4. Effect of pectin type and extraction method on pectin purity

Sample Type	Purity%
Standard High-Methoxyl Pectin	90.73 a
Standard Low-Methoxyl Pectin	89.08 b
Acid-Extracted Pectin	88.08 d
Microwave-Extracted Pectin	88.25

* Different lowercase letters within the same column indicate significant differences at a probability level ($P < 0.05$) according to Duncan's multiple range test.

This may be related to variations in extraction and purification efficiency and the retention of limited amounts of associated materials. [19] indicated that purification methods affect pectin properties and composition and contribute to impurity removal. Nevertheless, the similarity of values indicates efficient extraction, precipitation, and washing processes, and all samples exceeded the minimum [13] specification, which requires a minimum galacturonic acid content of 65% on a dry, ash-free basis.

HPLC results supported the purity data, with standard galacturonic acid exhibiting a peak at 4.08 minutes, while acid-extracted pectin peaked at 4.02 minutes with an area of 4156.09 $mAU.s^{-1}$, and microwave-extracted pectin peaked at 4.08 minutes with an area of 5065.99 $mAU.s^{-1}$. High-methoxyl standard pectin peaked at 4.01 minutes with an area of 5899.70 $mAU.s^{-1}$, while low-methoxyl standard pectin peaked at 4.08 minutes with an area of 6321.04 $mAU.s^{-1}$. The variation in peak areas indicates a difference in galacturonic acid content, with increased area corresponding to higher concentration and pectin purity. [15] demonstrated the suitability of HPLC for the quantitative determination of galacturonic acid in pectin. The similarity of the retention times to the standard compound and the appearance of a clear main peak enhance the reliability of its diagnosis and the dependability of its results in estimating pectin purity.

Conclusion

Quince peels are a promising source of pectin with suitable physicochemical, rheological, and functional properties for food applications. Compared with conventional acid extraction, microwave-assisted acid extraction reduced the extraction time from 60 min to 3–5 min and produced pectin with higher solubility, viscosity, gel strength, and slightly greater purity. The similarity of microwave-extracted pectin to standard high-methoxyl pectin in solubility and viscosity indicates good retention of its functional properties.

ISSN 2072-3857

HPLC analysis also confirmed the presence of galacturonic acid. Therefore, microwave-assisted acid extraction can be considered an efficient method for recovering functional pectin from quince peels and supporting the valorization of fruit-processing by-products.

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