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International Journal of Biological Macromolecules

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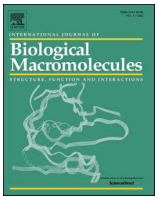
https://doi.org/10.1016/j.ijbiomac.2023.127957

International Journal of Biological Macromolecules 256 (2024) 127957

Contents lists available at ScienceDirect

International Journal of Biological Macromolecules

journal homepage: www.elsevier.com/locate/ijbiomac



Physicochemical, structural, and functional characterization of pectin extracted from quince and pomegranate peel: A comparative study

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ARTICLE INFO

Keywords:
Pomegranate
Quince
Pectin
Extraction
Characterization

ABSTRACT

Pectin's physicochemical, structural, and functional characteristics vary widely depending on the source of extraction. In this study, pectins were extracted from seedless quince and pomegranate peel, and their physicochemical, structural, and functional properties were investigated. A Box-Behnken Design with three factors and three levels was applied to optimize the pectin extraction yield from each matrix. As a result, the best extraction yields for quince pectin (QP) and pomegranate peel pectin (PPP) were 11.44 and 12.08 % (w/w), respectively. Both extracted pectins exhibit a linear structure, with the homogalacturonan domain dominating the rhamnogalacturonan I. Both pectins are highly methyl-esterified (DM > 69 %) with a higher degree of acetylation for PPP than QP, with 12 and 8 %, respectively. Unlike QP, PPP has a narrow, homogenous distribution and greater molecular weight (120 kDa). Regarding functionality, 1 g of QP could retain 4.92 g of water, and both pectin emulsions were more stable at room temperature than at 4 °C. When the concentration of QP is increased, rheological measurements demonstrate that it exhibits pseudoplastic behavior. Finally, QP can be used as a thickener, whereas PPP can be utilized as starting material for chemical changes to create multifunctional pectins.

1. Introduction

Pectin is a heteropolysaccharide originating from plants, located mainly in the plant cell wall's primary barrier and middle lamella. Pectin fraction comprises almost 35 % of fruit and vegetable cell walls [1]. Generally, pectin molecular structure is composed of three main regions: homogalacturonan (HG), rhamnogalacturonan I (RG-I), and rhamnogalacturonan II (RG-II). HG is the predominant region in pectin, presenting the backbone, and has a linear structure comprising α-1,4-linked galacturonic acid (GalA) units. Whereas RG-I region consists of the repeating disaccharide [→4)-α-D-GalA-(1 → 2)-α-L-rhamnose-(1→] branched at C-4 position of rhamnose by various side chains composed of neutral sugars, mainly D-galactose and/or L-arabinose [2]. RG-II is a highly complex region with an HG backbone of about nine α-1,4-linked GalA units and four lateral chains structurally conserved and containing 12 different glycosyl residues [3].

Galacturonic acid is the principal sugar of pectin molecules, making up at least 65 % and can be found in an acidic form, or methyl-esterified at the carboxyl group at C6, and/or acetyl-esterified at O2 and/or O3 of the hydroxyl groups [2]. According to its degree of methyl-esterification, two kinds of pectin can be distinguished: high methoxy pectin (DM > 50 %) and low methoxy pectin (DM < 50 %). The former can form a gel network in an acidic medium in the presence of sucrose, while the latter can make a gel through ionic cross-links (egg-box conformation) with divalent cations, such as calcium [4,5]. However, a high degree of acetylation (over 4 %) may block the interaction between pectin molecules in solution, but it could improve their emulsifying properties [6,7]. Furthermore, protein content significantly affects the emulsifying properties of pectin as well due to its hydrophobic nature [8], while molecular weight and its distribution also impact pectin's

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https://doi.org/10.1016/j.ijbiomac.2023.127957
Received 29 April 2023; Received in revised form 16 September 2023; Accepted 6 November 2023
Available online 10 November 2023

rheological properties [9].

The primary sources of pectin extraction are citrus peel, apple pomace, and, to a lesser extent, sugar beet due to their richness and particular functionalities [4]. However, the acidic extraction using mineral acids (such as hydrochloric acid and sulfuric acid) at high temperatures (between 80 and 100 °C), followed by filtration, precipitation, washing, and drying, is still the primary extraction technique used to extract commercial pectin due to its cheap cost and high efficiency, recovering pectin with high amount of HG blocks [10,11].

According to Industry Experts [12], around 60,000 tons of pectin were consumed as an additive in 2018, and it is expected that the global market of pectin will know growth of 5.8 % CAGR from 2018 to 2024. Thanks to their unique functional properties, pectins have a wide range of applications across various sectors: mainly in the food industry (production of jams, jellies, concentrated juices, and confectionery products), in the pharmaceutical industry (encapsulating agent for drug delivery), and the medical sector (immunity stimulator, wound healing, and anticarcinogenic effect) [11,13].

Pectin's physicochemical and functional properties change over a wide range according to the source of extraction [4]. According to the literature, a few studies were conducted on quince (*Cydonia oblonga*) pectin, including a preliminary exploration of some physicochemical properties, such as the GalA content, degree of methyl-esterification, and FTIR analysis, without addressing the functional proprieties [14,15]. Further characterization of other essential and determining physicochemical properties such as monosaccharide composition, degree of acetylation, and molecular weight and distribution is required in this context. In addition, exploring quince pectin's functional properties will help predict the possible uses and implications of this molecule to valorize it. On the other hand, several studies were conducted to valorize the Moroccan pomegranate peel by exploring its phenolic composition, antioxidant, antibacterial, and antifungal properties, but without investigating its pectic fraction [16,17]. Therefore, this is the first study on Moroccan pomegranate peel pectin. Furthermore, several studies demonstrated that the environment and pedoclimatic conditions could affect the content and characteristics of fruit cultivars substances such as pectin [7,18].

In Morocco, quince and pomegranate fruit production has grown tremendously in the last decade. The quince fruit production has increased from 36,240 tons in 2010 to 57,700 tons in 2020 [19], while pomegranate production reached 133,000 tons in 2018 compared to 53,511 tons in 2007 [16]. A large part of this production is processed by the food industries to produce juices, jams, jellies, and compotes, generating each year a massive amount of byproducts (peels and pomaces) that are not valued except for negligible traditional use of pomegranate peels [16], despite their richness in high-added value substances such as pectin [14,20–24]. As a result, valuing these massive amounts of byproducts will have a substantial socioeconomic and environmental impact on the country.

First, this study aims to optimize the pectin extraction yield from seedless quince and pomegranate peel through a Response Surface Methodology (RSM) using Box-Behnken Design (BBD), with three factors and three levels to fix the conditions that give the highest yield of pectin from each matrix. Second, to characterize the physicochemical, structural, and functional properties of quince and pomegranate peel pectins extracted under optimal conditions by investigating their protein content, monosaccharide composition, degree of methyl-esterification (DM), and acetylation (DA), molecular weight (Mw) distribution, water holding capacity, oil holding capacity, emulsifying activity, emulsion stability, thermal stability (TGA), infrared spectroscopy (FTIR), Nuclear Magnetic Resonance (NMR), and rheological behavior. The potential applications of the extracted pectins should be predicted after this characterization.

Table 1
Box-Behnken design with experimental and predicted yields of pectin extracted from quince fruit and pomegranate peel.

Factors		Unit	Actual levels		
			- 1	0	+ 1
Temperature	X1	°C	70	80	90
pH	X2	–	1.5	2	2.5
Time	X3	min	90	120	150

Run	X1	X2	X3	Quince fruit		Pomegranate peel	
				[a]EY (%)	[a]PY (%)	[a]EY (%)	[a]PY (%)
1	70	1.5	120	7.97	7.69	7.26	7.27
2	90	1.5	120	11.44	11.38	12.08	12.11
3	70	2.5	120	3.02	3.08	2.30	2.27
4	90	2.5	120	4.87	5.15	4.19	4.18
5	70	2	90	5.25	5.51	4.67	4.68
6	90	2	90	8.19	8.23	6.86	6.85
7	70	2	150	6.09	6.06	4.92	4.93
8	90	2	150	9.36	9.10	9.52	9.51
9	80	1.5	90	8.66	8.68	8.38	8.36
10	80	2.5	90	3.37	3.06	2.70	2.72
11	80	1.5	150	8.88	9.19	10.66	10.64
12	80	2.5	150	3.99	3.97	3.35	3.36
13	80	2	120	5.71	5.68	5.44	5.41
14	80	2	120	5.58	5.68	5.36	5.41
15	80	2	120	5.74	5.68	5.43	5.41

[a] EY and PY are experimental yield and predicted yield, respectively.

2. Material and methods

2.1. Material and reagents

Ripe quince (*Cydonia oblonga*) and pomegranate (*Punica granatum*) fruits were collected at their ripening period in early October from a farm in the Meknes region in Morocco. After washing, the quince seeds were removed, and the pomegranate fruits were peeled. Both matrices were treated separately. First, the seedless quince and pomegranate peel were cut into small pieces (about 10 mm in diameter) and blanched at 90 °C for 5 min to inactivate any pectinolytic activity [25]. Next, the materials were dried at 45 °C in an air-oven drier (DHG-9140A, manufactured in China) until they attained a constant weight. Finally, the dried matrices were ground, sifted through a 40 mesh sieve, and stored in air-tight plastic bags in a dry environment until use.

Ethanol, methanol, hydrochloride acid, sulfuric acid, trifluoroacetic acid, sodium hydroxide, m-hydroxy-diphenyl, phenolphthalein, and standard monosaccharides including galacturonic acid, arabinose, galactose, rhamnose, xylose, mannose, fucose, and glucose, were purchased from Sigma Chemicals.

2.2. Experimental design for pectin extraction

First, the one-factor-at-a-time method was used to determine the best liquid-to-solid ratio (LSR). The LSR was varied from 10 to 60 ml/g while the other parameters were constant: the temperature at 80 °C, pH at 2, and extraction duration at 120 min [26]. The corresponding LSR to the highest extraction yield recorded was used to optimize the other parameters.

A Box-Behnken Design (BBD) was used in Minitab Software to optimize the conditions of pectin extraction from seedless quince and pomegranate peel. In this context, three independent variables, such as temperature, pH, and extraction time, with three levels were employed. The variables and their corresponding actual and coded values are presented in Table 1. After entering these parameters and levels in the

BBD, 15 experimental runs with different conditions were created, as shown in Table 1. Different factors with different level combinations organized by BBD were used for pectin extraction using the following protocol.

2.3. Pectin extraction protocol

Pectin was extracted from quince and pomegranate peel powders using a hot-acidic extraction described by Kratchanova, Pavlova, and Panchev [27], with some modifications. First, quince or pomegranate peel powder (2 g) was dispersed in distilled water then the pH was adjusted to the corresponding values (shown in Table 1) using 0.5 M HCl. Afterward, the slurry was transferred to a water bath with a fixed temperature and time (Table 1). Next, the extracted pectin was centrifuged at 3260 x*g* for 20 min at room temperature, and the filtrated supernatant was added to an equal volume of ethanol 96 % and kept under slow agitation for 10 min for the coagulation. The coagulum was left overnight at 4 °C to precipitate. After precipitation, pectin was centrifuged at 3260 x*g* (for 20 min, RT) and washed twice with ethanol at 80 % and 96 % (*v*/v) to desalt and remove the attached mono and disaccharides, then dried at 45 °C until the weight became constant. Finally, the dried pectin was crushed in a cold-water circulator blender and stored in air-tight plastic bags in a dry environment for further characterization. The yield of extraction was calculated following Eq. (1):

$$Yield\ (\%, dry\ weight) = \frac{Weight\ of\ dried\ extracted\ pectin\ (g)}{Weight\ of\ dried\ powder\ (g)} \times 100 \qquad (1)$$

2.4. Chemical characterization

2.4.1. Protein content and Monosaccharide composition of pectin

The amount of proteins retained in the pectin samples (0.2 g) after their extraction was measured using the Kjeldahl method (N × 6.25) [7]. The experiment was repeated three times.

The monosaccharide composition of pectin extracted under the optimal conditions from quince fruits and pomegranate peel was determined following the method described by Broxterman, van Erven, and Schols [28] with slight modification. An ICS3000 HPAEC system (Dionex Corporation, Sunnyvale, CA, USA) was coupled to an ED pulsed amperometric detector (ICS5000 ED). First, pectin powders were methanolized with 3 M HCl in absolute methanol at 80 °C for 16 h, followed by hydrolysis in 2 M Trifluoroacetic acid (TFA) for 1 h at 121 °C. Two PA-1 columns (2 × 50 mm; 2 × 250 mm) were used, in addition to a post-column (Thermo Scientific). The hydrolyzed pectins (10 μl) were eluted at 0.4 ml/min using three mobile phases: Milli-Q water for 27 min, 0.17 M NaOAc in 0.1 M NaOH for 11 min, and 1 M NaOAc in 0.1 M NaOH for 5 min, respectively. In the post column, the separated monosaccharides were mixed with 0.5 M NaOH and performed 0.1 ml/min between 0 and 35 min and 65 min. Chromeleon software 7.2.6 (Thermo Scientific) was used to analyze the data.

The uronic acid content of the extracted pectins was determined by an autoanalyzer (Skalar, Breda, the Netherlands) as described by Thibault [29] using the *m*-hydroxydiphenyl colorimetric method of Ahmed & Labavitch [30]. For this purpose, the samples were hydrolyzed with sulfuric acid at 72 % (w/w) for 1 h at 30 °C, then with 1 M sulfuric acid for 3 h at 100 °C (Seaman hydrolysis).

2.4.2. Degree of methyl and acetyl esterification

The degree of methyl esterification (DM) and the degree of acetylation (DA) of quince and pomegranate peel pectins were determined by the quantification of methanol and acetic acid released after a saponification reaction [28]. First, the samples (5 mg) were saponified overnight with 0.1 M NaOH. Afterward, the methanol released was analyzed by GC-head space using TRACE™1300 Gas Chromatograph (Thermo Scientific™) equipped with J&W DB-WAXetr (30 m × 0.25 mm × 0.25 μm) ion trap column (Agilent Technologies, USA). Xcalibur software (Thermo Scientific) was used to analyze the data.

The degree of acetylation was evaluated using the HPLC technique. For this purpose, the remaining saponified solutions were first centrifuged at 18,000 x*g* for 10 min and then analyzed by an Ultimate 3000 system (Thermo Scientific) equipped with an Aminex HPX 87H column (300 mm × 7.8 mm) and a guard column (30 mm × 4.6 mm) (Bio-Rad, Hercules, USA). The elution occurred at 40 °C with 50 mM H_2SO_4 at a flow rate of 0.6 ml/min, and the process was monitored using a Shodex RI-101 detector (Showa Denko K.K.). Chromeleon 7.2.6 (Thermo Scientific) was used for data analysis. At the same time, acetic acid standard concentrations from 5.3 to 525 μg/ml were used to calibrate the system. The degree of acetylation and methyl esterification were calculated as moles of acetic acids and methanol per 100 mol of galacturonic acid.

2.4.3. Molecular weight distribution

Pectins extracted under the optimal conditions from quince and pomegranate peel were analyzed for their molecular weight distribution using high-performance size exclusion chromatography (HPSEC). First, pectin solutions at 2 mg/ml were centrifuged for 10 min at 18,000 x g and then analyzed using an Ultimate 3000 HPLC system (Dionex, Sunnyvale, CA, USA) coupled to a Shodex RI-101 RI detector (Showa Denko, Tokyo, Japan). A set of three TSK-Gel columns used in series, 4000, 3000, 2500 SuperAW (150 mm × 6 mm) preceded by a TSK super AW-L guard column (35 mm × 4.6 mm) (Tosoh Bioscience, Tokyo, Japan). The samples (10 μl) were injected and eluted at 0.6 ml/min with 0.2 M $NaNO_3$ at 55 °C. Pectin standards (0.6, 1.8, 5, 16, 50, and 150 kDa) from the Laboratory of Food Chemistry of Wageningen University (The Netherlands) were used to estimate the molar mass [31]. The Mw of pectins was estimated based on the top of the peak.

2.5. Structural characterization

2.5.1. Fourier-transform infrared (FTIR) spectroscopy

Infrared spectroscopy analysis of pectin extracted from quince fruits and pomegranate peel was performed using a Thermo Nicolet iS50 FTIR (Thermo Fisher Scientific Co., Waltham, MA, USA). The Attenuated Total Reflection (ATR) method recorded the results. First, the pectin powders were placed on the diamond crystal and pressed with the system tip-flap. The spectra were recorded at a resolution of 4 cm^{-1} with 32 scans. Before each analysis, the system background was set by registering the empty ATR plate spectrum.

2.5.2. Nuclear magnetic resonance (NMR) analysis

^{1}H, ^{13}C, and HSQC NMR analysis of QP and PPP pectin extracted at optimal conditions was performed using a JOEL NMR spectrometer (JNM-ECZ600R/M1). Pectin samples were dissolved in D_2O and spectra were obtained at an internal temperature of 35 °C and a frequency of 600 and 150 MHz for ^{1}H and ^{13}C, respectively.

2.6. Functional properties characterization

2.6.1. Water holding capacity (WHC) and oil holding capacity (OHC.)

The WHC and OHC of quince and pomegranate peel pectins were measured [32]. First, in centrifuge tubes, 0.2 *g* of each pectin was mixed with distilled water or sunflower oil (10 ml). After that, the mixture was homogenized for 1 min by a vortex and then centrifuged for 30 min (3000 ×*g*, RT). Finally, the pellet was weighed after removing the supernatants. The results were expressed as grams of water or oil retained in 1 g of pectin (considering the oil density). The tests were repeated three times.

2.6.2. Emulsifying activity and emulsion stability

The emulsifying properties of quince and pomegranate peel pectin extracted under the optimal conditions were determined in triplicate according to the method of Dalev and Simeonova [33] with some

modifications. Briefly, aqueous pectin solution at 0.5 % (*w/v*) was added to an equal volume of sunflower oil (1:1; *v*/v). Afterward, the mixture was homogenized at 10,000 × g for 4 min by an ultra-rapid homogenizer. After that, 10 ml of pectin emulsion was poured into transparent and graduated centrifuge tubes and then centrifuged at 4,000 ×*g* for 5 min. In the end, three distinct layers were observed: a small oil layer, an emulsified layer, and an aqueous layer containing dispersed pectin molecules from the bottom to the top. The emulsifying activity (EA) of each pectin emulsion was calculated as follows (Eq. (2)):

$$EA\ (\%) = \frac{Volume\ of\ emulsified\ layer\ (ml)}{Total\ Volume\ (ml)} \times 100 \quad (2)$$

Regarding the emulsion stability (ES), pectin-oil emulsions of each pectin were prepared as above, then stored for one day at 4 °C and room temperature. After storage, three different layers were observed as above, and the ES was determined using the following equation (Eq. (3)):

$$ES\ (\%) = \frac{Volume\ of\ the\ remaining\ emulsified\ layer\ (ml)}{Total\ volume\ (ml)} \times 100 \quad (3)$$

2.7. Rheological measurement

The steady flow behavior of quince and pomegranate peel pectins at different concentrations (1, 2, and 3 % (*w/v*)) was measured using a rotational cylinder rheometer (Physica MCR 501; Anton Paar Co.) equipped with a concentric cylinder geometry as previously described [34]. First, pectin was solubilized in distilled water at 50 °C for 7 h and stored overnight at 4 °C before being analyzed. Before measurements, a transient viscosity at different sharing points was performed to determine the measuring point duration from which the viscosity gets to its steady state. Then, the tests were realized at controlled shear rates (between 1 and 100 s^{-1}, at 25 °C), and the viscosity curves were recorded.

2.8. Thermogravimetric analysis (TGA/DTG)

The thermal stability of pectin samples was measured using a thermogravimetric analyzer (TGA Q500, TA Instrument). Each pectin (~10 mg) was heated at a 10 °C/min heating rate from room temperature up to 700 °C. The experiments were conducted at a 60 ml/min flow rate under a nitrogen atmosphere.

2.9. Statistical analyses

Minitab Lab. software was used to do regression analysis on the experimental data. The F-test with a *p*-value (0.05) was used to determine the significance of the regression coefficients. The adequacy of the regression model was assessed using the coefficient of determination R^2 and adjusted R^2. ANOVA was used to examine the significant independent variables in the model, with a confidence level of 95 %. The contour and surface plots were generated using regression coefficients. The graphs and Tukey's test ($p < 0.05$) were created with the Origin program.

3. Results and discussion

3.1. Pectin extraction yield and model fitting of experimental data

One factor at a time was used to determine the best liquid-to-solid ratio (LSR) for optimizing pectin extraction from quince and pomegranate peel. As a result, the maximal extraction yield values were obtained at an LSR of 30 and 50 ml/g of quince and pomegranate peel powders, respectively. The increase in extraction yield while increasing LSR could be explained by the increase of the surface area between powder particles and the solvent and the decrease of the viscosity and pectin concentration in the solvent, which pushes pectin to leave cell tissue due to the difference in osmotic pressure [35]. The best LSR for each matrix was kept constant during the optimization of pectin extraction through BBD, while the experimental results and the predicted values obtained are presented in Table 1. As can be seen, quince pectin (QP) and pomegranate peel pectin (PPP) extraction yields were in the range of 3.02–11.44 and 2.30–12.08 % (w/w, dry basis), respectively, using different combinations of levels of the three factors (Table 1).

The best pectin extraction yield for both matrices was obtained at a temperature of 90 °C, a pH of 1.5, and an extraction time of 120 min, corresponding to run n°2 (Table 1). Under these conditions, PPP shows a slightly higher extraction yield than QP, with 12.08 ± 0.22 and 11.44 ± 0.31 % (w/w, dry basis), respectively. According to previous studies, several factors could influence the pectin extraction yield, mainly the source, extraction technique, and extraction conditions [11]. In this context, Japanese quince (*Chaenomeles japonica*) pectin extracted using a sequential extraction method showed a similar extraction yield of 11 % compared to QP [20]. Chinese quince (*Chaenomeles sinensis*) pectin showed a lower extraction yield of 9.7 % based on its alcohol-insoluble solid fraction after a sequential extraction [24]. On the other hand, the PPP extraction yield was higher than UK pomegranate peel pectin (11.2 %) extracted using citric acid as a solvent [10]. A similar trend was observed when using nitric acid to recover pectin from Tunisian (11 %) and Chinese (8.5 %) pomegranate peels [22,23], showing lower extraction yields compared to PPP pectin extracted with hydrochloric acid. This result could be due to the difference in the acids' strength [36], proving that HCl is more effective in extracting pectin from pomegranate peel. Concerning the extraction technique, the hot-acidic extraction used in this study allows for recovering more pectin from pomegranate peel, compared to the ohmic-heating extraction, which gave 8.16 % [35]. Similarly, hot-acidic extraction is more efficient than enzymatic extraction, which recovers less pectin from pomegranate peel with 6.8 % [37]. In contrast, using ultrasonic force to extract pectin from pomegranate peel resulted in a higher yield (23.87 %) compared to the current study [21]. This can be attributed to the combination of cavitation energy with a very low pH (pH 1.27) and a high temperature [21]. Despite its effectiveness in extracting pectin, several studies have found that ultrasonic power causes depolymerization of pectin molecules and reduces their molecular weight, limiting the functional qualities of the extracted pectin [38–40].

The second-order polynomial equations related to the extraction yield of quince pectin (Eq. (4)) and pomegranate peel pectin (Eq. (5)) were obtained using Minitab Lab. Software and used to calculate the predicted yield mentioned in Table 1.

$$\begin{aligned} Quince\ Yield\ (\%) = {} & 5.677 + 1.441X_1 - 2.712X_2 + 0.356X_3 + 1.073X_1^2 \\ & + 0.075X_2^2 + 0.473X_3^2 - .0.405X_1X_2 + 0.082X_1X_3 \\ & + 0.100X_2X_3 \end{aligned} \quad (4)$$

$$\begin{aligned} Pomegranate\ Yield\ (\%) = {} & 5.410 + 1.687X_1 - 3.230X_2 + 0.730X_3 + 0.634X_1^2 \\ & + 0.414X_2^2 + 0.449X_3^2 - 0.732X_1X_2 + 0.602X_1X_3 \\ & - 0.407X_2X_3 \end{aligned} \quad (5)$$

X1, X2, and X3 are the coded temperature, pH, and extraction time values.

The model's statistical meaning was investigated using analysis of variance (ANOVA), as demonstrated in Table 2. The p-value of both models was significant, alongside the higher p-value of the lack of fit, which indicates that the model fits the response well. Furthermore, the determination coefficient (R^2) and the adjusted determination coefficient (Adj-R^2) were calculated to test the adequacy of the model (Table 2). As can be seen, the R^2 and Adj-R^2 were high, with 99.38 % and 98.27 % for quince fruits and 99.99 % and 99.98 % for pomegranate

Table 2
Multiple regression analysis for the pectin extraction yield from quince fruit and pomegranate peel.

Matrix	Source	DF	Some of the squares	Mean square	F-value	p-Value
Quince fruit	Regression	9	82.038	9.115	89.53	0.000
	Residual error	5	0.509	0.102		
	Lack-of-fit	3	0.495	0.165	22.79	0.042
	Pure error	2	0.014	0.007		
	Total	14	82.548			
	R^2			99.38		
	Adj R^2			98.27		
	Pred R^2			90.37		
Pomegranate peel	Regression	9	117.270	13.030	9176.05	0.000
	Residual error	5	0.007	0.001		
	Lack-of-fit	3	0.003	0.001	0.58	0.683
	Pure error	2	0.004	0.002		
	Total	14	117.277			
	R^2			99.99		
	Adj R^2			99.98		
	Pred R^2			99.95		

peel, respectively. This indicates that each matrix model is accurate and that most variations in the response can be predicted and explained.

3.2. Effect of extraction factors on pectin yield

Pareto charts (Fig. 1) and the ANOVA of the Plackett-Burman design (Table A.1, supplementary material) show the importance and the statistical significance of the linear, quadratic, and interaction effects of all variables in the model on the extraction yield. The horizontal bars reflect the positive and negative effects of the components in the response variables, while the vertical line assesses the significance of the effects at the 95 % confidence level. Regarding QP extraction yield (Fig. 1a), five significant effects are detected: the linear effect of pH and the linear and quadratic effects of temperature and time. In contrast, PPP extraction yield is significantly influenced by all the factors, including their linear, quadratic, and interaction effects (Fig. 1b). This difference in the response could be attributed to the structure and the composition of the matrix's cell wall.

Furthermore, contour and surface plots were generated using Minitab Lab. software to elucidate the effect of each factor on the extraction yield of QP (Fig. 2) and PPP (Fig. 3). As indicated in Figs. 2 and 3 (a, b, e, and f), among the three factors tested, the pH had the major impact on QP and PPP pectin extraction yields, and its negative effect (slope coefficients of −2.71 and −3.23, respectively, Table A.1) indicates that the yield increases when the pH of the solvent is decreased. These results could be explained by the ability of acids to hydrolyze water-insoluble pectin (protopectin) into a soluble form, which increases its recovery [41]. Similar trends were found during optimizing pectin extraction from sugar beet [7,42]. After pH, the temperature is the second most influential factor on QP and PPP pectin extraction yields (Figs. 2 and 3 (c, d, e, and f)). As shown in Table A.1 (supplementary material), the positive slope coefficients of temperature (+1.44 and +1.69 for QP and PPP, respectively) reflect its positive effect on yield. The fact that pectin extraction yield increases with increasing temperature has been linked to the ability of heat to improve solvent penetration into the plant matrix by reducing its viscosity and increasing its ability to solubilize cell wall compounds [22]. Similarly, the extraction time had positively affected the pectin extraction yield from both matrices (Figs. 2 and 3 (a, b, c, and d)) but at a lower extent compared to pH and temperature (slope coefficient of +0.36 and +0.73 for QP and PPP, respectively). The increased yield due to the longer extraction time is most likely owing to more mass transfer from the raw material to the solvent [43].

3.3. Physicochemical characteristics

3.3.1. Monosaccharide composition

The monosaccharide composition of pectin extracted from quince fruit and pomegranate peel under optimal conditions is indicated in Table 3. As can be seen, quince pectin (QP) showed a higher galacturonic acid (GalA) content compared to pomegranate peel pectin (PPP), with 46 % and 38 %, respectively. The same tendency was observed concerning the amount of neutral sugars, which is higher in the case of QP (40 %) than in PPP (14 %). This difference is mainly due to the nature of the raw material. The presence of low sugar content in PPP pectin (about 52 %) indicates the presence of other compounds, such as proteins, salts, and moisture. In this context, the moisture and ash content were analyzed by thermogravimetry (Fig. 9) and found higher in PPP than QP pectin: PPP has 22 % moisture and 14 % ashes, while QP shows 16 % moisture and 7 % ashes. These justify the higher sugar content in QP than in PPP pectin. Different studies have reported that pectin composition changes according to the location and climatic conditions of the cultivated plant [7,18]. In this context, PPP pectin shows a high GalA content compared to pectin of pomegranate peels grown in Iran and extracted using chemical and enzymatic methods with 30.8 % and 19.9 %, respectively [37]. In contrast, Tunisian pomegranate peel pectin had a higher GalA and neutral sugars contents, with 48.9 % and 25.6 %, respectively [22]. On the other hand, QP shows a lower GalA content and higher neutral sugars amount in comparison to the Japanese quince (*Chaenomeles japonica*) pectin obtained through a sequential extraction

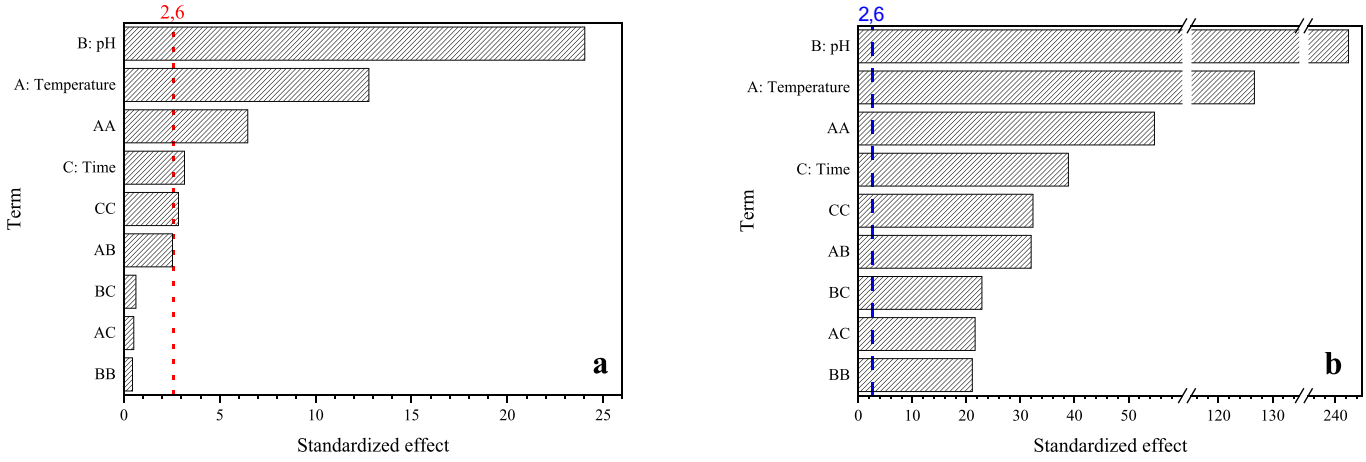

Fig. 1. Pareto diagrams for the effect of extraction factors on pectin yield from (a) quince fruit and (b) pomegranate peel.

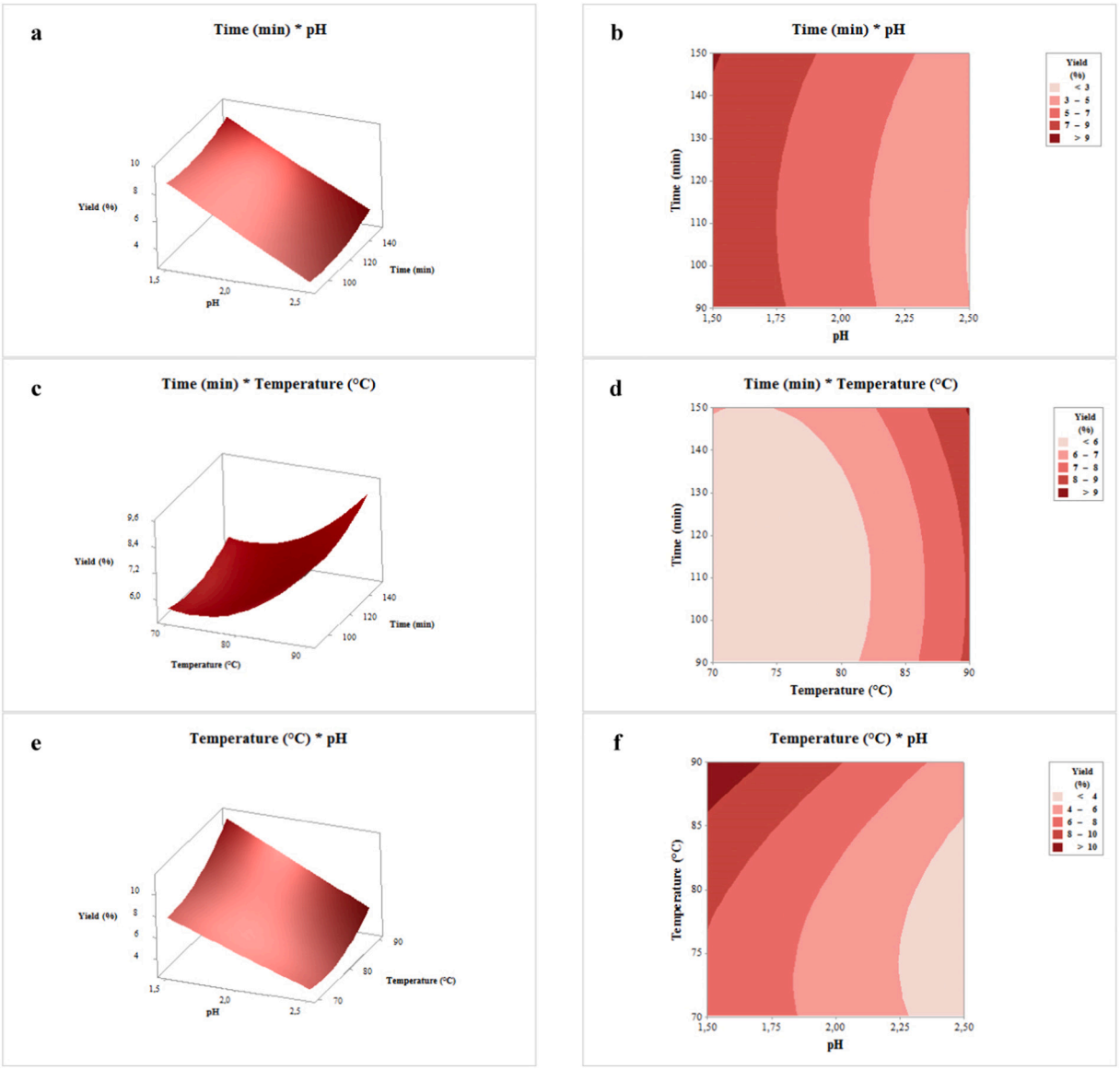

Fig. 2. Surface (a, c, and e) and contour (b, d, and f) plots for the effect of the independent factors on pectin yield from quince fruits.

method using different solvents at different temperatures and pH values (GalA = 57.3 and 57.5 %; Neutral sugars = 18.6 and 23.0 %) [20].

Table 3 shows the molar percentage of each monosaccharide that constitutes QP and PPP pectin molecules. The GalA is the principal sugar in both QP and PPP pectin, with 52 and 70 mol%, respectively. In the meantime, the neutral sugar composition is dominated by galactose with 4 and 7 mol% in QP and PPP pectin, respectively. Rhamnose and arabinose show 5 mol% each in PPP pectin, while QP pectin has only 1 mol% each. These values indicate the presence of arabinan, galactan, or arabinogalactan as molecule side chains, which are extensive in PPP compared to QP pectin. According to previous studies, the fruit variety and the extraction technique and conditions significantly affect the monosaccharide composition of pectin molecules [44]. Chinese quince (*Pseudocydonia sinensis*) pectin obtained after a sequential extraction had shown a higher GalA content (86.7–96.2 mol%) than QP pectin, but the rhamnose (0.3–1.8 mol%) and arabinose (1.5–3.2 mol%) were inferior [24]. Regarding pomegranate pectin, the PPP pectin molecule has a higher GalA content than Chinese pomegranate peel pectin extracted using the same technique, with 59.4 to 61.1 mol% [45]. In contrast to PPP, the neutral sugars of Chinese pomegranate pectin molecule were dominated by arabinose (6.6–10.8 mol%) over galactose (4.1 to 8.0 mol %) and rhamnose (1.2–1.9 mol%) [45]. These results could be due to the different environmental conditions in which the fruits had grown.

From a structural point of view, both QP and PPP pectins have a linear structure with the dominance of the HG domain over RG-I. According to Table 3, the HG domain makes 50 and 60 % of the QP and PPP pectin structure, while RG-I presents 7 and 22 %, respectively. This indicates the dominance of the smooth region over the hairy region for both samples. The amount of glucose found in QP pectin (34 %) was significantly higher than in PPP pectin (6 %), which could be due to the nature of the raw material. The glucose residues found in a pectin extract might come from the partial hydrolysis of cellulose, hemicellulose, and/or residual starch during the process [22]. Quince pulp has a higher starch level than pomegranate peel, which leads to the extraction of more starch besides pectin molecules. A high glucose level in the pectin samples could impair their functional properties, which require further purification. Compared to PPP pectin, Tunisian and Iranian pomegranate peel pectins have shown much higher glucose levels of 18.7 and 34.9 %, respectively [22,37], which indicates that Moroccan pomegranate peel pectin is purer than the Tunisian and Iranian one. Considering that glucose is not part of the monosaccharides that make up the pectin molecule, the purity of the QP and PPP samples is 52 % and

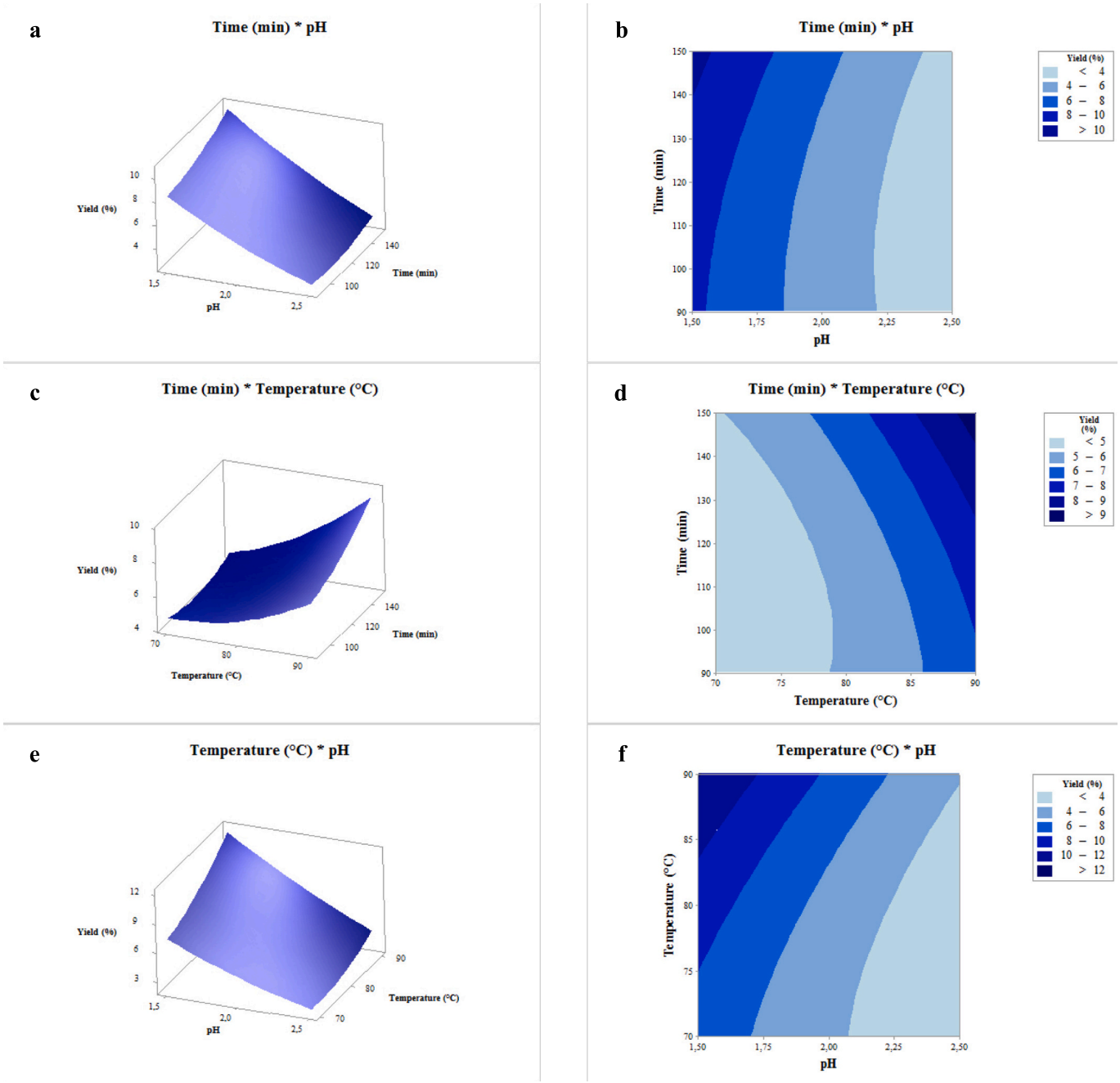

Fig. 3. Surface (a, c, and e) and contour (b, d, and f) plots for the effect of the independent factors on pectin yield from pomegranate peel.

46 %, respectively.

3.3.2. *Degree of methyl and acetyl esterification*

The degree of methyl esterification (DM) and degree of acetylation (DA) are defined as the number of galacturonic acids (GalA) esterified by a methyl or acetyl group on the total number of GalA. The DM and DA of quince and pomegranate peel pectins are presented in Table 3. As can be seen, the degree of methyl esterification of QP pectin is higher than PPP pectin, with 80 % and 69 %, respectively. The DA was higher in PPP pectin than QP, with 12 % and 8 %, respectively. These results classify QP and PPP pectin as acetylated high methyl-esterified pectins (AHMP). Based on the literature, pectin with a DM > 50 % could make gel at low pH in the presence of sucrose [4]. However, a high degree of acetylation could hinder pectin's molecular interactions and improve the emulsifying properties [6]. The DM of QP pectin extracted by hydrochloride acid is higher than that obtained previously from the same matrix using nitric acid as an extraction solvent, with 60.97 % [14]. This result could be explained by the strength of the acid used [36]. Meanwhile, QP pectin shows a higher DM and DA than Japanese quince (*Chaenomeles japonica*), with 61.3–63.7 % and 5 %, respectively [20]. On the other hand, PPP pectin shows a higher DM than Tunisian pomegranate pectin (30 to 55 %) [22]. The same statement was detected in Chinese pomegranate pectin, which had a lower DM ranging from 52.27 to 58.74 % [45]. In contrast, Russian pomegranate pectin showed a higher DM of 75 % compared to PPP pectin [46]. This difference could be explained by the mild conditions used during the extraction (aqueous extraction at a low temperature of 70 °C for 2 h) [46]. Regarding the degree of acetylation, PPP pectin exhibits a similar DA to Chinese pomegranate pectin (12.24–14.58 %) [45]. At the same time, Russian pomegranate pectin had more acetyl groups (DA of 15 %) [46]. According to the

Table 3
Chemical composition and functional properties of pectin extracted at the optimum point from pomegranate peel (PPP) and Quince (QP)[a].

	QP	PPP
Chemical composition (%)[b]		
Optimal yield	11.44 ± 0.31[a]	12.08 ± 0.22[b]
Protein	4.49 ± 0.11[a]	4.20 ± 0.11[b]
Neutral sugars	40.0	14.0
Uronic acid	46.0	38.0
DE	87.32 ± 0.59[a]	80.72 ± 1.55[b]
DM	80.0	69.0
DA	8.0	12.0
Mw (kDa)[c]	80.0–17.0	108.0
Monosaccharide composition (% mol)[d]		
GalA	52.0	70.0
Gal	4.0	7.0
Rha	1.0	5.0
Ara	1.0	5.0
Xyl	1.0	1.0
Fuc	ND	ND
Man	ND	ND
Glu	41.0	12.0
HG (%)	50.0	60.0
RG-I (%)	7.0	22.0
Functional properties[e]		
WHC (g of water / g of pectin)	4.92 ± 0.14[a]	2.87 ± 0.32[b]
OHC (g of oil / g of pectin)	2.41 ± 0.08[a]	2.82 ± 0.26[a]
EA (%) At RT	50.47 ± 0.50[a]	50.33 ± 0.58[a]
ES (%) at 4 °C for 1 day	58.00 ± 1.73[a]	66.67 ± 1.15[b]
ES (%) at RT for 1 day	71.33 ± 0.58[a]	70.33 ± 0.58[a]

[a] There were no significant differences between values followed by the same letters in the same row ($p < 0.05$).
[b] DE, DM, DA, and Mw are the degree of esterification, degree of methyl esterification, acetylation, and molecular weight, respectively.
[c] The molecular weight of pectins was estimated based on the top of the peak.
[d] GalA, Gal, Rha, Ara, Xyl, Man, Fuc, and Glu are galacturonic acid, galactose, rhamnose, arabinose, xylose, mannose, fucose, and glucose, respectively, while ND is not detected. HG, RG-I are homogalacturonan and rhamnogalacturonan I, respectively. HG = GalA – 2 x Rha; RG-I = 2Rha + Ara + Gal.
[e] WHC and OHC are water-holding capacity and oil-holding capacity, respectively, while EA, ES, and RT are emulsifying activity, emulsion stability, and room temperature.

findings, QP pectin may have good gelling qualities due to its large DM (80 %), whereas PPP pectin may have better emulsifying capacity due to its higher DA (12 %).

3.3.3. Molecular weight distribution by HPSEC.

The molecular weight (Mw) and corresponding distribution could significantly affect pectin's functional properties. Therefore, an HPSEC was used to determine the Mw distribution of QP and PPP pectins. As shown in Fig. 4, the elusion profiles recorded for both pectins were different. QP pectin shows a broad and heterogeneous distribution with an elution time ranging from 7.6 to 12.6 min and a small separated peak at around 14.8 min. In contrast, PPP pectin displays a narrow and homogeneous distribution in the presence of one significant population of high Mw (High RI signal intensity) and three small populations of small Mw (low RI signal intensity). The prominent peak of PPP was obtained at 8.45 min elution time, while the three others were retained at 12.9, 13.8, and 14.8 min. The broad distribution recorded for QP pectin may point to starch that may be extracted beside pectin molecules: 34 % of glucose in QP versus only 6 % in PPP (Table 3). The water (WSP) and chelator (CSP) soluble pectin fractions extracted from Chinese quince have shown a different Mw distribution model compared to QP, with a narrow and homogeneous distribution [24]. In contrast, the sodium-carbonate soluble fraction (NSP) was large and heterogeneously distributed [24]. On the other hand, unlike PPP pectin, the Iranian pomegranate pectin extracted by both hot-acid and enzymatic extraction methods showed a broader and heterogeneous distribution [37]. These differences could be due to the source and technique of extraction and the pedoclimatic condition of the planted raw material.

The molecular weight of QP and PPP pectins was determined according to the calibration curves established using charged pectin as standard (Table 3). The Mw distribution of QP pectin shows two shoulders: the first has a lower RI signal intensity at 8.75 min corresponding to 80 kDa, and the second with a higher RI signal intensity recorded at 10.3 min, which has 17 kDa. These values indicate the richness of QP-extracted pectin with two populations of different Mw, 80 and 17 kDa. Otherwise, the molecular weight of PPP pectin was 108 kDa according to the prominent peak detected at 8.45 min (Fig. 4). The remaining three tiny peaks identified were small molecules such as mono and disaccharides or salts isolated with pectin molecules. Pectin molecular weight depends on several factors such as the source, extraction technique and conditions, analysis technique, and environmental conditions of the planted plant [18]. The molar mass of QP pectin was found to be similar to the different pectin fractions obtained from Chinese quince, in which the WSP and CSP fractions had shown two population with 90–80 kDa and 1.5 and 2 kDa, respectively, while NSP fraction was below 32 kDa [24]. Whereas, The Mw of PPP pectin was smaller than that obtained from Iranian pomegranate using acidic (6385 kDa) and enzymatic (422 kDa) extraction techniques [37]. In the meantime, the hydrothermal extraction of pomegranate pectin led to the extraction of higher Mw molecules (142 kDa) compared to PPP extracted using acid [47]. Also, commercialized orange pectin had a higher molecular weight (146.7 kDa) than PPP and QP [25]. In contrast, PPP pectin molecular weight is bigger than Moroccan citrus pectin (80.0 to 90.0 kDa) [34]. This variation could be attributed to varied extraction and analysis procedures, plant origin and variety, and pedoclimatic conditions they grew under. According to a previous study [31], the stability of drinkable yogurt increases as pectin's average molecular weight rises. On the other hand, a higher molecular weight might slow down the solubility of pectin, but this could be overcome by its higher degree of methyl esterification [5].

3.4. Structural properties

3.4.1. Fourier-transform infrared (FTIR) spectroscopy

The FTIR spectrum (Fig. 5) of quince pectin (QP) and pomegranate peel pectin (PPP) show almost the same shape due to their similar chemical composition. A broad and strong band observed at 3351 cm^{-1} corresponds to the hydrogen banding stretching vibration of the hydroxyl groups related to galacturonic acid. The absorption peak at 2938 cm^{-1} referred to C—H (-CH, -CH_2, and -CH_3) elongating ambiances [38]. The asymmetric tension of the methyl-esterified carboxyl group (O-CH_3) appears at 1725 cm^{-1} as a stronger absorption signal than that of stretching vibration of the free carboxyl group (COO^-) at 1603 cm^{-1}, which is a characteristic of high methyl-esterified pectins (DM > 50 %) [48]. The peaks between 1277 cm^{-2} and 1012 cm^{-2} are attributed to the C-O-C glycosidic bond and C—O vibration in COOH and C-OH [38]. The absorption intensity of all bands mentioned was higher in pectin extracted from pomegranate peel (PPP) than quince fruit pectin (QP). This result confirms the pectic nature of the extracted polysaccharides and the qualitative and quantitative differences between both characterized samples, as indicated in Table 3.

3.4.2. Nuclear Magnetic Resonance (NMR)

The ^1H and ^{13}C NMR spectra of quince pectin and pomegranate peel pectin are presented in Fig. 6. As can be seen in Fig. 6a, both samples show a tall and sharp peak at 3.78 ppm, which corresponds to the protons of methyl-ester of the carboxylic group of galacturonic acid residues [49]. Furthermore, the signal peaks acquired at 4.94 and 4.80 ppm corresponded to protons bound to the esterified and non-esterified GalA units, implying that the pectins produced are both highly methyl-esterified [49]. Both extracted pectin revealed chemical shifts at

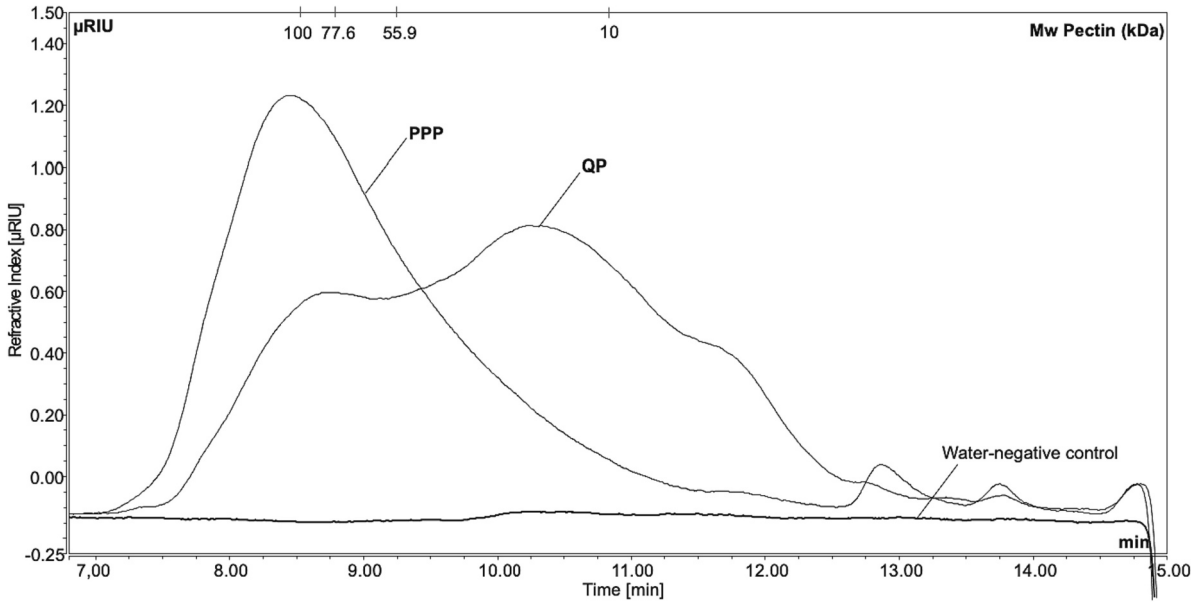

Fig. 4. High-Performance Size Exclusion Chromatography (HPSEC) patterns of the molar mass distribution of quince pectin (QP) and pomegranate peel pectin (PPP). Pectin calibration is indicated as the second X-axis.

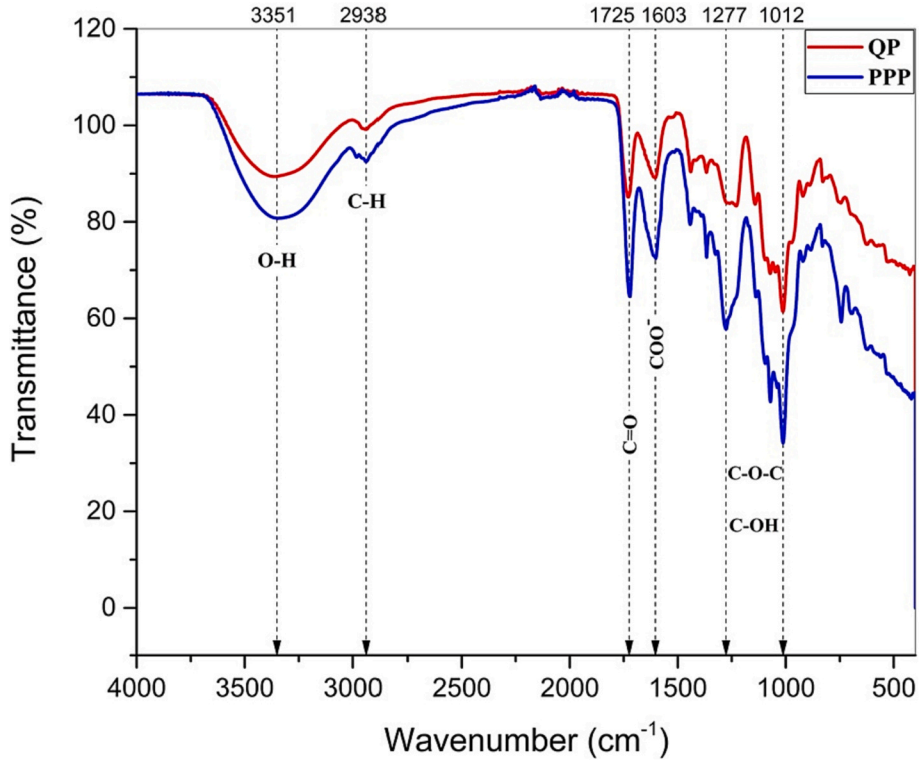

Fig. 5. Fourier Transform Infrared (FTIR) Spectra of quince pectin (QP) and pomegranate peel pectin (PPP).

around 5.05, 4.94, 4.44, 3.94, and 3.62 ppm, which correspond to GalA protons H1, H5, H4, H3, and H2, respectively [50]. Meanwhile, the anomeric proton of rhamnose units was shifted downfield at 5.36 ppm [51] and showed a longer peak in PPP compared to QP, which is consistent with the results of the monosaccharide composition (Table 3). The signal peaks around 1.1 ppm are ascribed to rhamnose methyl group linkages [49], implying that PPP contains more rhamnose than QP. The chemical shifts at 2.19, 2.06, 2.15, and 2.02 ppm in the PPP sample correspond to acetyl groups connected to C-2 of a 2-*O*-GalA, C-3 of a 3-*O*-GalA, and C-2 and C-3 in a 2,3-di-*O*-GalA units, respectively [52]. In comparison to PPP, QP pectin had just a single tall peak at 2.06 ppm, which matched the acetyl group banding at C-3 in a 3-*O*-acetyl residue. The signal intensity of these peaks was larger in the PPP sample than in the QP sample, with 1.352 and 1.162, respectively, indicating that the PPP pectin has more acetyl groups than the QP pectin. According to Wang et al. [53], the peak shifted at 3.32 ppm, which appears solely in QP pectin, is likely attributable to protons coupled to the hydroxyl groups C-2 and C-3 of glucose residue, indicating the presence of starch

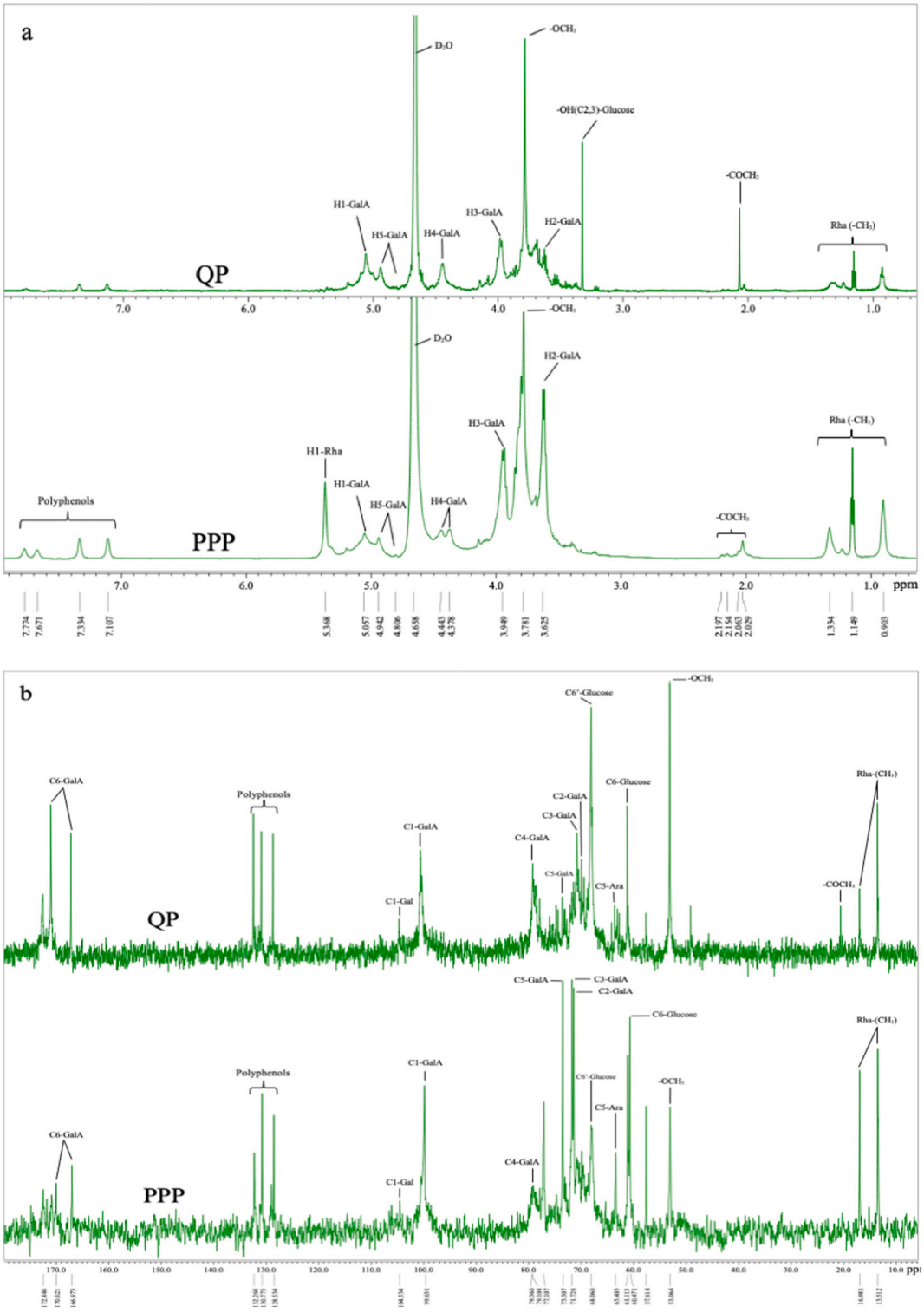

Fig. 6. 1D ^{1}H (a) and ^{13}C (b) NMR spectra of quince pectin (QP) and pomegranate peel pectin (PPP).

in QP samples. Differently, PPP pectin emits four signals between 7.10 and 7.77 ppm that correspond to polyphenol residues [52]. Finally, the NMR spectra indicate the extracted samples' pectic nature and a high degree of esterification, as well as differences in composition, such as the presence of phenolic substances in PPP and starch in QP pectin samples.

Regarding the ^{13}C NMR spectra (Fig. 6b), the signal obtained at 99.8 ppm is attributed to the anomeric carbon of GalA residues. The signals obtained around 170 ppm indicate the carbonyl group C6 of GalA residues methyl-esterified [54,55]. On the other hand, the signals of C2, C3, C4, and C5 of GalA were obtained at 71.4, 71.3, 77.1, and 73.5 ppm. The signal of the carbon of the methyl groups linked carboxyl groups to GalA was obtained at a chemical shift of 53.06 ppm with a higher intensity for QP than PPP confirming the methyl-esterification degree of both samples [56]. Two signals of high frequency were detected upfields at 13.51 and 16.98 ppm corresponding to the rhamnose C6 of the methyl groups. Whereas, the acetyl group signal has been detected at

20.45 ppm for the QP spectrum. The C5 signal of arabinose was detected at 63.40 ppm, while the signal obtained at 104.53 ppm corresponds to the anomeric carbon of galactose [55]. The C6 signals of glucose residues were detected at 60.47 and 68.06 ppm, which correspond to the linked and unlinked C6 and confirm the presence of starch [57,58]. However, three signals were obtained around 130 ppm could be attributed to the presence of phenolic compounds in both samples.

To elucidate the chemical structure of QP and PPP pectins, the correlations between the proton and carbon signals were evaluated. The 2D HSQC spectra of both samples are depicted in Fig. 7 and assigned according to literature values [51,59–61]. Not all the signals can be identified due to the complicated spectra found especially for QP. However, several signals have been detected in the anomeric region of QP. The $^{1}H/^{13}C$ chemical shift correlations of the anomeric carbon of GalA have been detected at 4.95/100.42 ppm and 5.06/100.04 ppm for methyl-esterified and non-methyl-esterified forms respectively. On the other hand, the signals obtained at 4.61/104.34 ppm and 5.37/99.62 ppm are attributed to galactose and rhamnose residues, respectively, while the signal at 4.62/95.83 ppm may belong to glucose residues. On the other hand, only one signal has been detected at the anomeric region for PPP at 5.37/99.82 ppm assigned to rhamnose residues. The chemical shift correlations of the methyl group of rhamnose showed at 1.24/16.57 ppm and 1.30/16.60 ppm. Whereas, the signal obtained at 2.07/20.45 ppm is assigned to the acetyl group in 3-*O*-GalA. Signals at 3.78/53.11 ppm and 3.78/52.83 ppm indicate the methyl esterification of the carbonyl groups of GalA. The chemical shift correlations obtained downfield at 7.13/130.59 ppm and 7.35/128.41 ppm and at 7.11/130.82 ppm and 7.33/128.46 ppm confirm the presence of phenolic compounds in QP and PPP samples, respectively.

3.5. Functional properties

3.5.1. Water holding capacity and oil holding capacity

Water holding capacity (WHC) is the amount of water held by 1 g of sample. WHC is an essential property influencing various food products' textural, physiological, and technological qualities [62]. As shown in Table 3, QP pectin had shown a significantly higher WHC than PPP pectin. Whereas 1 g of QP and PPP can hold 4.92 ± 0.14 and 2.87 ± 0.32

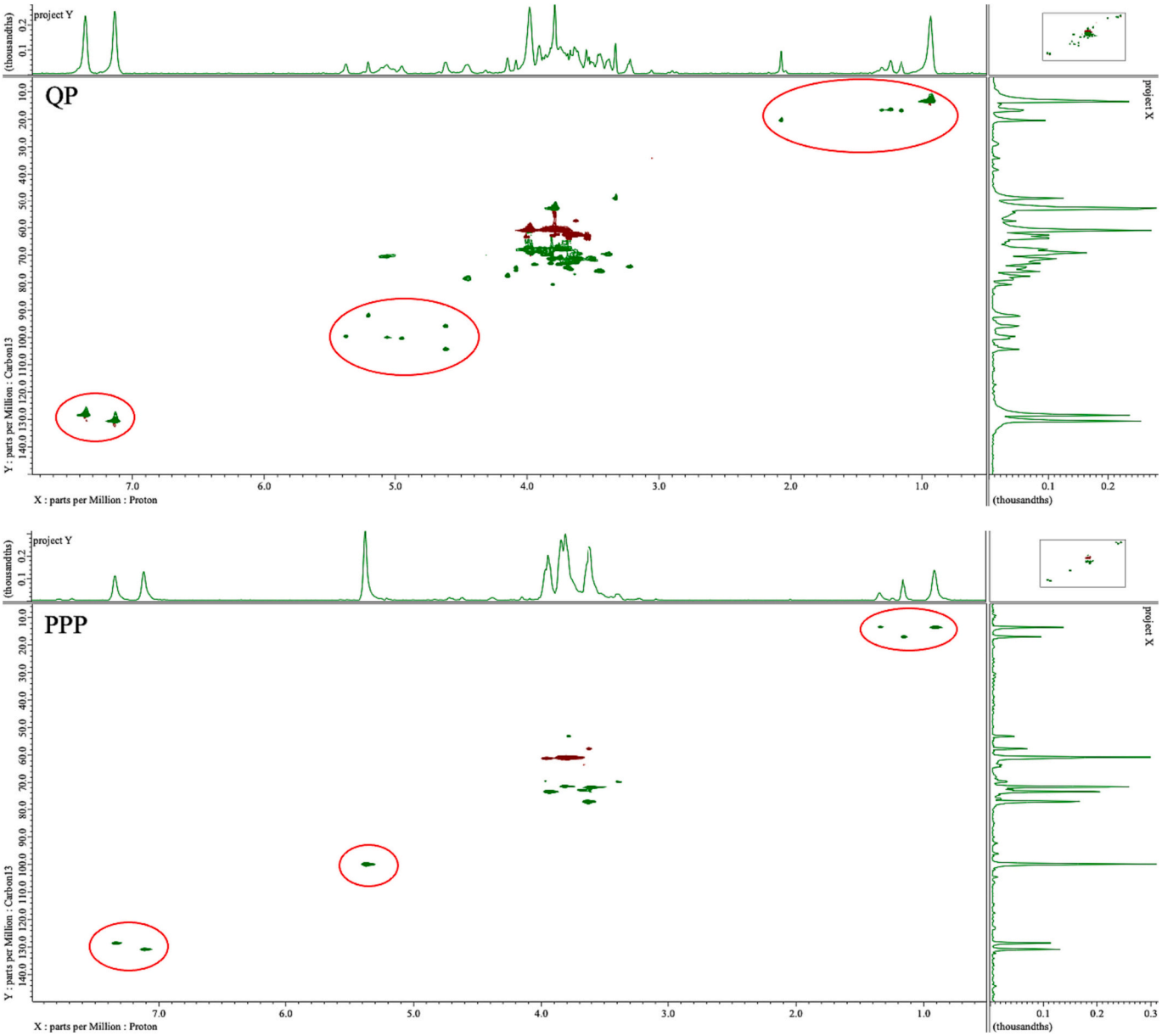

Fig. 7. 2D HSQC NMR spectra of quince pectin (QP) and pomegranate peel pectin (PPP).

water (w/w), respectively. This difference could be explained by the high number of acetyl groups in the PPP structure (DA = 12 %), which reduces the total number of free hydroxyl groups (OH). The WHC of QP pectin was higher than *Citrus limetta* peel and commercial citrus pectins, which can hold 3.07 and 3.19 g water per g of pectin, respectively [62]. On the other hand, PPP pectin holds more water than water-soluble polysaccharides extracted from pistachio (1.46 g/g) and almond (1.95 g/g) byproducts [63]. Contrariwise, PPP pectin had shown a lower WHC than the mixed banana–papaya peel pectin (8.23 g/g) and *Panax notoginseng* flower pectin (6.75–8.62 g/g) [38,64]. The result indicated that the water-holding characteristic of QP was greater than that of commercial pectin, implying that it has potential commercial uses as a thickening in the food sector.

Oil holding capacity (OHC) refers to the quantity of oil retained by 1 of the sample. The high OHC of the pectin molecule allows for stabilizing the high-fat food products and emulsions [65]. According to Table 3, a non-significant difference has been detected between PPP and QP pectins which can hold 2.82 ± 0.26 and 2.41 ± 0.08 g oil/g pectin, respectively. A similar result has been observed for commercial citrus (2.37 g/g) pectins [62]. However, QP and PPP pectin could hold more oil than Moroccan citrus pectin, which had a maximum OHC of 1.55 g oil/g pectin [34]. The OHC of *Panax notoginseng* flower and mixed banana–papaya peel pectins was higher with 5.24–5.98 and 3.44 g oil/g pectin, respectively [38,64].

3.5.2. Emulsifying activity and emulsion stability

The capacity of pectin to form an emulsion is a significant characteristic making it useful as an emulsifier and stabilizer in food products. Generally, pectin is predominantly hydrophilic, and with a high amount of proteins and acetyl groups in its structure, it acquires an amphiphilic feature [66]. The emulsifying activity (EA) and the emulsion stability (ES) of QP and PPP pectins are indicated in Table 3. As can be seen, both pectins had a higher emulsifying activity with 50.47 ± 0.50 and 50.33 ± 0.58 %, respectively, for QP and PPP. These results were higher than those obtained for sugar beet pectin, which had 47.1 % [7]. QP and PPP emulsifying capacity were more heightened than commercial pectin (46.67 %) [67]. Whereas PPP pectin exhibits a lower EA than that obtained using ohmic-assisted extraction (68.41 %) from the same matrix [35]. This difference could be due to the extraction technique and/or the analysis methodology using a lower centrifuged force than that used in the presented study (527 × g instead of 3000 × g) [35]. It was reported that pectin emulsifying ability could be affected by several parameters, such as protein content and quality, degree of methyl and acetyl-esterification, and molecular weight [8]. Acetyl groups and proteins could anchor on the oil particle surface, reducing its surface tension [8]. However, the amount of protein stuck with the extracted pectin was slightly higher for QP (4.49 ± 0.11 %) than PPP (4.20 ± 0.11 %), as indicated in Table 3. These values are closer, which may account for the non-significant difference in EA reported between QP and PPP. According to the Food and Agriculture Organization (FAO), the protein content of pectin must be lower than 15.6 % [68], which is in line with the obtained values.

On the other hand, the stability of emulsions made by QP and PPP pectin after one day of storage at 4 °C and room temperature (RT) is shown in Table 3. At 4 °C, PPP pectin showed higher ES than QP, with 66.67 ± 1.15 and 58.00 ± 1.73 %, respectively. This difference could be explained by the higher degree of acetylation of PPP (12 %) compared to QP (8 %) (Table 3). The number of GalA units and the percentage of the RG-I region in the pectin structure can also influence the pectin emulsion stability, thus forming a hydrated-thick layer around the oil droplets, avoiding coalescence [69]. At RT, a non-significant difference was observed between both samples: 70.33 ± 0.58 % for PPP and 71.33 ± 0.58 % for QP Regarding storage temperature, QP and PPP emulsions showed more stability at room temperature than at 4 °C. This result might be linked to a reduction in surface tensions between the produced oil droplets, which limits their accumulation. A similar tendency was observed for the emulsion made by pomegranate pectin (at 0.5 %) recovered using an ohmic-assisted extraction technique, in which the emulsion stability at 25 °C (79.95 %) was higher than 4 °C (35.30 %) [35]. Chen and Tao [70] have reported that a high temperature is favorable for emulsification because it decreases the viscosity and the interfacial tension. Compared to QP and PPP, the emulsion stability of the commercial citrus pectin (at 2 %) and mixed banana-papaya pectin were lower, with 64.28 and 29.33 %, respectively [64,67]. Due to their higher EA and ES than commercial pectin, QP and PPP might be employed as emulsifiers and stabilizers in the food industry.

3.6. Rheological measurement

The rheological properties of pectin depend on several factors, such as concentration, Mw and its corresponding distribution, GalA content, DM, and DA, which are mainly related to source and extraction technique [71]. The steady flow behavior of quince and pomegranate peel pectin solutions at different concentrations (1, 2, and 3 %) has been investigated (Fig. 8). As can be seen, the two pectins showed two distinct flow behaviors. Regarding QP pectin (Fig. 8a and b), the flow curves recorded were fitted to the power-law model (Eq. (6)),

$$\sigma = K.\gamma^{n} \quad (6)$$

where K is the consistency index (Pa.s^{n}), n is the flow behavior index (unitless), γ is the shear rate (s^{-1}), and σ is the shear stress (Pa). The corresponding values to the consistency and flow behavior indices (K and n) are given in Table 4. Generally, the K value is proportional to the viscosity of the solution, while n outlines the flow behavior. As shown in Fig. 8a and b, the flow behavior of QP pectin has been changed depending on the concentration. At 1 %, the QP pectin solution exhibits Newtonian flow behavior with almost no change in apparent viscosity over the entire shear rate range. This result was confirmed with the value of n obtained (n ≈ 0.9) close to 1, characteristic of Newtonian fluids. This behavior could be due to weak attractive forces between pectin molecules at low concentrations. Whereas the flow behavior of QP turns pseudoplastic when the concentration has been increased (Fig. 8a and b). At 2 and 3 %, It was observed that the viscosity of the solution increased with the decrease in the shear rate, which is due to the entanglement of the polymer network with the relative decrease in shear rate [72]. The shear thinning behavior of QP pectin solution became evident at 3 % concentration (Fig. 8a and b). On the other hand, PPP pectin has shown a Newtonian flow behavior, whatever the concentration used (Fig. 8c and d). As shown, the apparent viscosity of pectin solutions was constant and independent of shear rate despite increasing pectin concentration. This behavior is explained by the high degree of acetylation of pomegranate pectin (12.0 %) that hinders the interaction between pectin molecules preventing gel formation [6]. A linear relationship was recorded between shear stress and shear rate (Fig. 8d), which is well fitted with Newton's model (R^2 = 99.9 %) expressed by the following equation (Eq. (7));

$$\sigma = \eta.\gamma \quad (7)$$

where η is the viscosity (Pa.s). As shown in Table 4 and Fig. 8c and d, the viscosity increases from 0.004 to 0.026 Pa.s when the concentration increases. These results agreed with the WHC obtained, which was higher for QP pectin than PPP (Table 3). In conclusion, QP appears to be the appropriate pectin as a texturing agent highly sought by food industries.

3.7. Thermogravimetric analysis (TGA/DTG)

The thermogravimetric curves of QP and PPP pectins extracted under optimal conditions are illustrated in Fig. 9. Three different regions were distinguished for both pectins depending on the heating temperature. Region I starts from room temperature (~ 25 °C) up to 200 °C (Fig. 9a),

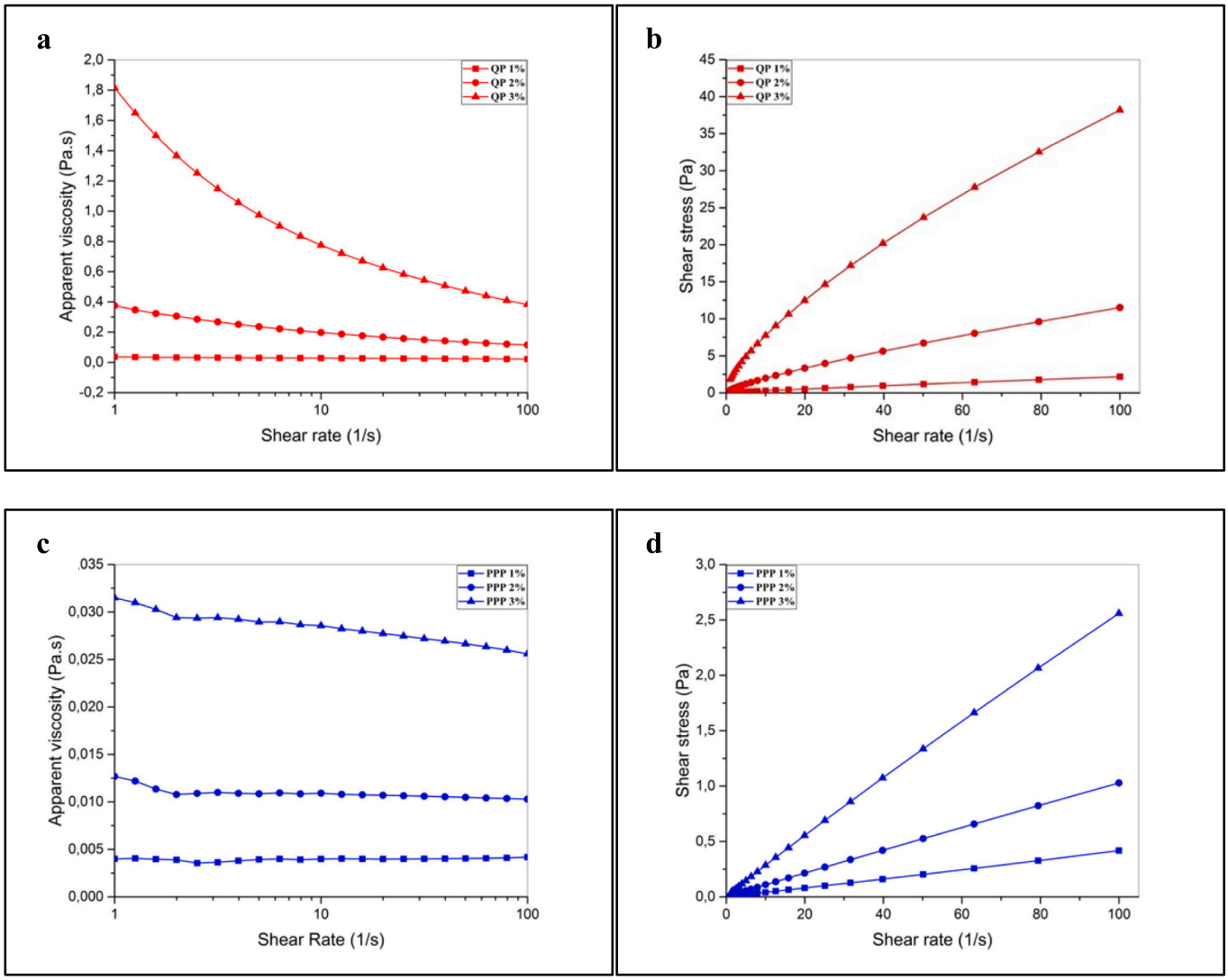

Fig. 8. Steady flow behavior of quince (a and b) and pomegranate peel (c and d) pectins at 1, 2, and 3 % (*w/v*).

Table 4
Flow behavior parameters of the apparent viscosity of quince (QP) and pomegranate peel (PPP) pectin.

Pectin	Concentration (%)	[a]K or [b]η (Pa.s^n)	[c]n	R^2
QP	1.0	0.035 ± 0.000	0.896 ± 0.001	0.999
	2.0	0.337 ± 0.003	0.766 ± 0.002	0.999
	3.0	1.600 ± 0.013	0.688 ± 0.002	0.999
PPP	1.0	0.004 ± 0.000	–	0.999
	2.0	0.010 ± 0.000	–	0.999
	3.0	0.026 ± 0.000	–	0.999

[a] K consistency index.
[b] η viscosity.
[c] n flow behavior index.

corresponding to a slight loss of pectin weight due to the evaporation of the impregned water. At this stage, PPP pectin lost more water (22 %) than QP (16 %), which indicated its higher moisture content. This loss difference is seen in the DTG curves (Fig. 9b), where the peak area and amplitude at Region I are much higher in PPP than in QP. Region II begins at 200 °C and ends at 350 °C, which is attributed to an increased weight loss due to extensive pyrolytic degradation of pectin molecule [48]. During this part, QP lost 52 % of weight, higher than PPP, which lost 40 %. This difference could be due to the high sugar content of QP compared to PPP (Table 3). At the same region, according to TGA curves (Fig. 9b), the maximum exothermic peaks were obtained at around 250 and 260 °C for PPP and QP, respectively. Furthermore, the half-life temperature (T_{50}) of QP pectin was slightly higher than PPP with 288 and 281 °C, respectively (Fig. 9a), which suggests that QP pectin has somewhat higher thermal stability. The last part of TG curves (Region III) ranged from 350 to 700 °C, corresponding to char decomposition [73]. This Region is characterized by a slight weight loss for both pectins (around 25 %), leaving a residual mass ratio of 14 % and 7 % for PPP and QP, respectively, which could be considered ashes. In conclusion, the degradation of QP and PPP pectins begins at high temperatures (around 200 °C), which gives them a high thermal stability behavior during the thermal processing of food.

4. Conclusion

Seedless quince and pomegranate peel showed a high potential to be used as a source to extract pectins with relevant properties. Large pectin extraction yields were obtained with 12.08 % and 11.44 % (w/w, dry basis) for pomegranate peel and quince, respectively. Quince pectin (QP) can be used as a thickener due to its high water-holding capacity; QP can hold approximately five times its weight in water. On the other hand, pomegranate peel pectin (PPP) presents a high potential molecule with outstanding physicochemical characteristics, such as a high degree

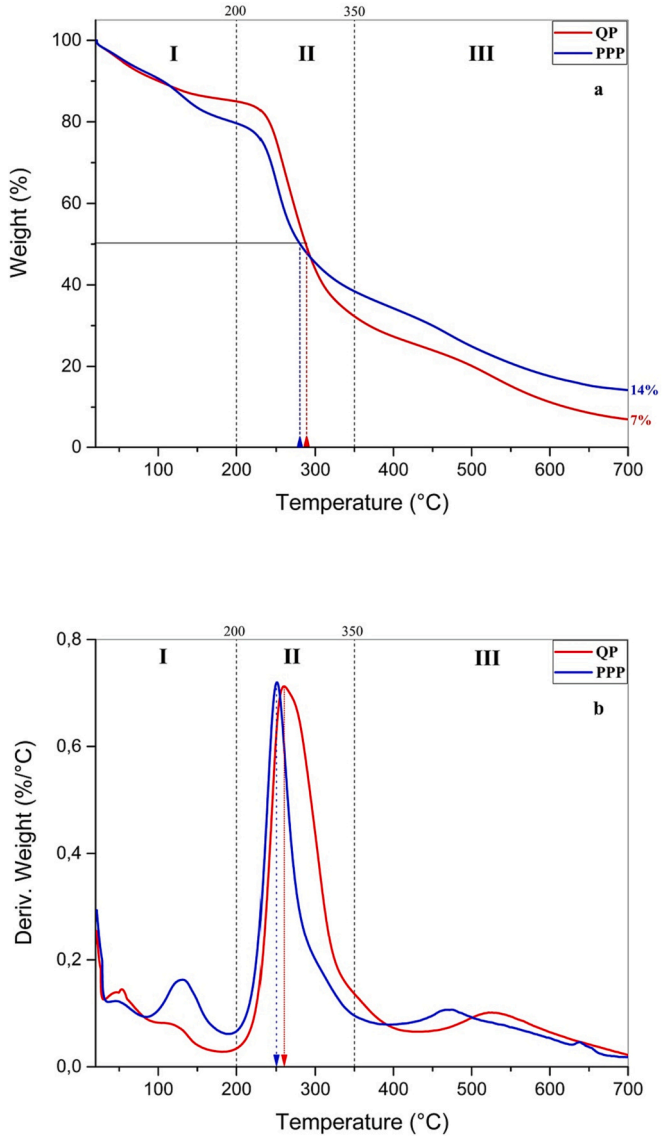

Fig. 9. TGA (a) and DTG (b) curves of quince pectin (QP) and pomegranate peel pectin (PPP).

of methyl-esterification (69 %) and degree of acetylation (12 %), big molar mass molecule (120 kDa) with narrow and homogeneous distribution, which make it a suitable starting material to produce different pectins with different functionalities. Both pectins have shown higher emulsion stability at room temperature than at cold, which could help increase the shelf-life of some foodstuffs. As perspectives, further purification of QP by eliminating the starch (using amylase, for example) could improve its functional properties. Regarding the PPP, works are in progress to modify its physicochemical properties. Better emulsifying properties could be attained by reducing PPP molar mass via physicochemical or enzymatic depolymerization. At the same time, the gelling ability could be restored by chemical deacetylation.

Supplementary data to this article can be found online at https://doi.org/10.1016/j.ijbiomac.2023.127957.

CRediT authorship contribution statement

Noussaire El Fihry: Conceptualization, Methodology, Investigation, Software, Data curation, Writing – original draft, Writing – review & editing, Visualization, Formal analysis. **Khalil El Mabrouk:** Resources, Methodology, Supervision, Writing – review & editing. **Mia Eeckhout:** Resources, Supervision, Writing – review & editing. **Henk A. Schols:** Software, Resources, Data curation, Formal analysis, Methodology, Writing – review & editing. **Hassan Hajjaj:** Conceptualization, Funding acquisition, Investigation, Resources, Validation, Supervision, Project administration, Writing – review & editing.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Noussaire El Fihry reports financial support, administrative support, equipment, drugs, or supplies, and travel were provided by VLIR-UOS.

Data availability

Data will be made available on request.

Acknowledgments

The authors would like to express their gratitude for the guidance and financial support from the Flemish Interuniversity Council throughout the VLIR-UOS-IUC project, which provides the best conditions to perform this work.

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