Utilization of Bitter orange seeds as a novel source for recovery of pectin: Compositional and rheological characterization

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Utilization of Bitter orange seeds as a novel source for recovery of pectin: Compositional and rheological characterization

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Abstract

The seeds from bitter orange, a by-product from the juice making step, hold the potential to facilitate novel, easy yet high quality pectin extraction. To test this hypothesis, the pectin from orange seeds (OSP) were extracted by distilled water and its compositional parameters and rheological behavior then evaluated. Results showed that galacturonic acid was the major component of OSP (~ 425 mg/g) confirming the purity of extracted pectin, followed by glucose and some minor neutral sugars. M_w, R_n and, R_z for the OSP were 4511.8 (kDa), 61 (nm), and 61.1 (nm), respectively. Rheological measurements showed shear-thinning behavior for OSP that by increasing temperature from 5 to 45 ºC, the viscosity of the gum decreased. Power law fitted as the best rheological model describing the flow behavior of OSP. Strain sweep dynamic rheological measurements confirmed an entangled network structure for OSP and the addition of NaCl to the gum dispersion, decreased the consistency coefficient from 35.6 to 23.18 $Pa.s^n$, while the flow behavior index remained unchanged. These results demonstrate for the first time that the OSP can be used as a new source of pectin, with likely a wide range of applications in food industry.

Keywords: Pectin; Orange seeds; Sugar composition; Molecular weight; Rheological properties.

1. Introduction

Hydrocolloids are water-soluble biopolymers with a wide application in food industry. They are commonly used to improve textural properties of food as a gelation and viscosity improving agents (Nejatian et al., 2020). Pectin one of the major hydrocolloids in food processing industry is a polysaccharide with 1, 4-galacturonic acid units and some of its carboxyl groups can be substituted with methyl esters or amide groups (Löfgren and Hermansson, 2007). It is widely used as an ingredient for providing specific textural and rheological properties to processed food as a gelling/thickening agent or as an emulsion stabilizer for acidified dairy drinks (Christiaens et al., 2016).

The source of pectin, the extraction procedures, the particle size distribution, the patterns of acylation, the degree of esterification (DE) and the nature and position of the neutral sugars have a great impact on the specifications of pectin from the various origins (Maxwell et al., 2012). Pectin is considered as an invaluable by-product of the citrus processing industry. About 85% of the globally commercial pectin is sourced from citrus waste which can be produced from fresh or processed peels of lemon, grapefruit, and oranges (Berk, 2016).

Disadvantages associated with pectin extraction from citrus peels include the use of corrosion mineral acid, high temperature and extended extraction time. Together these may affect pectin quality and moreover damage equipment. Furthermore, the steps of filtration, discoloration and concentration of pectin solution before alcohol precipitation are associated with both high cost and use of time (May, 1990). Therefore, new sources of pectin with both lower extraction time and production costs is both economically and technologically appealing (Shan, 2016). Previous studies examining pectin extraction have been varied in their approach. The extraction and physiochemical properties of pectin from the heads of sunflowers was studied by Peng et al. (Peng

et al., 2020). Asgari and co-workers (Asgari et al., 2020) studied the walnut processing waste as a novel source of pectin. Gharibzahedi et al. (Gharibzahedi et al., 2019) evaluated the pectin extracted from Fig (*Ficus carica* L.) skin. Chaliha and co-authors (Chaliha et al., 2018) extracted pectin from *Terminalia ferdinandiana*- a native Australian fruit. Finally, the possibility of using Palmyra Palm (*Borassus aethiopum* Mart.) fruit was evaluated by Assoi et al. (Assoi et al., 2017).

The bitter orange (*Citrus aurantium*) contains many seeds and differs from the orange by several characters including the acidic pulp and bitterer albedo (Moufida and Marzouk, 2003). It is commonly used for essential oils, in the perfume industry and for the production the marmalade. In the Middle East, the juice of the ripen fruit can be used as a salad dressing or as a flavoring (Zibaee et al., 2020).

It is necessary to understand the rheological properties and determination of sugar composition of pectin from new sources to evaluate their thickener potential. The flow behavior of hydrocolloids solutions is important to assess processing parameters, textural properties of formulated foods, design of unit operations and development of product engineering (Balaghi et al., 2011; Rincón et al., 2014). To date there is no report on the extraction of pectin from orange seeds and hence an evaluation of its compositional and functional properties. Therefore, the main objective of this research was to extract and characterize the sugar composition, molecular weight, and rheological properties of pectin from bitter orange seeds as a novel source of pectin.

2. Material and methods

2.1 Materials and extraction method

81 The bitter orange fruit (*Citrus aurantium* L.) was used in this study, collected from trees growing
82 in Mazandaran province of Iran during October and November 2020 and according to the
83 permission and the national guideline of Agricultural Research Education and Extension
84 Organization of Iran. Phenotypic identification of the specimen was done by Dr. Saeid Hazrati,
85 academic member of Shahid Madani University of Tabriz, Iran.

86 The seeds were collected from bitter oranges during the juice extraction. Seeds were washed with
87 tap water to remove the fruit pulp and drained completely to remove the excess water. Afterward,
88 the seeds soaked in distilled water (pH= 7) in a seed ratio of 6:1 at 70 ± 1 ºC and stirred for 30 min
89 (solid/liquid ratio selected based on preliminary test). Thereafter, pectin coats were removed from
90 the seeds by passing through a 600 µm sieve and collected in a flask. The extracted solution mixed
91 with three volumes of 96% v/v ethanol and placed in the fridge (4 ºC). After 24 h, the flocculated
92 pectin collected and dried in an oven with circulating air at 30 ºC. The dehydrated pectin (OSP)
93 was pulverized with a miller, packed, and kept in cool and dry condition prior test.

94 Standards of monosaccharides (galacturonic acid, glucose, arabinose, galactose, rhamnose, and
95 fucose) and trifluoracetic acid (TFA) with purity of ≥ 99% were purchased from Merck
96 (Darmstadt, Germany).

97 *2.2 Compositional analysis*

98 AOAC method (AOAC, 2016) was used for determination of moisture content and total ash
99 content. Kjeldal method was used for the determination of total protein content with the nitrogen
100 value of 6.25 (Razavi et al., 2014).

101 After hydrolysis 4 g/l of OSP by TFA (2 M) for 2h at 121 ºC, the monosaccharide composition
102 analyzed by high-performance anion-exchange chromatography with a pulsed amperometric

103 detector (DECADE Elite). Separations carried out in a CarboPac PA1(4×250mm) column (Dionex
104 Corp., Sunnyvale, CA). Samples were passed through 0.22 μm filter prior injections into the
105 column. The monosaccharides separation through the column was carried out according to the
106 method of (Gavlighi et al., 2013).

107 *2.3 Degree of esterification (DE)*

108 The titration method proposed by Chaharbaghi et al. (Chaharbaghi et al., 2017) was used for
109 determination of DE of pectin sample. To do this, 100 mg of dried powder of sample was added
110 to 2 ml of ethanol and dissolved in 20 ml deionized water at 40 ºC. Afterward, 5 ml of
111 phenolphthalein reagent was added to the solution and titrated with NaOH (0.1 M). The amount
112 of NaOH used for titration recorded as V_i. Afterward, 10 ml of NaOH was added to the solution
113 and mixed for 30 min for complete hydrolysis. 10 ml of HCl (0.1 M) was introduced to the solution
114 and mixed vigorously to completely disappear the pink color. After the addition of drops of
115 phenolphthalein, the excessive amount of HCl was titrated with NaOH to obtain a pale pink color
116 and the volume of NaOH was recorded as the V_f. The DE of pectin was calculated according the
117 following equation (Hosseini et al., 2016):

118 $$DE = (V_f / V_i + V_f) \times 100$$

119 *2.4 Determination of molecular parameters*

120 Molecular weight averages (M_n, M_w, M_z) of the OSP were determined by using a size exclusion
121 chromatography system (flow rate of 0.4 mL/min with 0.15 M $NaNO_3$ and 0.02% NaN_3) and
122 equipped with TSK G5000 PW column (7.5 × 600 mm; Toso Biosep, Montgomeryville, PA, USA)
123 joint to a UV detector (Waters, 2487), multi-angle laser light scattering (HELEOS; Wyatt
124 Technology Corp, Santa Barbara, CA, USA) and a refractive index detector (Waters, 2414)

125 (HPSEC-UV-MALLS-RI). Bovine serum albumin (BSA) was used as a standard for determination
126 of the volume delays among the US, MALLS, and RI detectors. ASTRA 5.3 software (Wyatt
127 Technology Corp.) applied for data acquisition and to calculate the M_w average, M_n, and M_z.

128 *2.5 Rheological Measurements*

129 In order to determine the rheological characteristics of gum, OSP dispersions at concentrations
130 0.1, 0.3, 0.5, 0.7 and 1 % (w/v) were first prepared by dissolving of the required amount of dry
131 powdered gum in distilled water and gently stirred at room temperature for 2 h. The gum
132 dispersions were stored overnight at 5 ºC to assure that the hydration of the polysaccharide was
133 complete.

134 Both steady shear viscosity and oscillatory shear tests were performed by Physica MCR 301
135 rheometer (Anton Paar GmbH, Graz, Austria) so that a concentric cylinder geometry (radius ratio
136 of 1.035) and a parallel plate geometry (25-mm diameter; 0.5-mm gap) were used for dilute
137 samples and concentrated dispersions, respectively. The temperature was adjusted to 25 ºC with a
138 Viscotherm VT2 circulating bath and a controlled Peltier system (Anton Paar, GmbH) with an
139 accuracy of ±0.01 ºC.

140 Shear sweeps were conducted at 25 ºC between 0.001 to 1000 s^{-1} so as to obtain flow curves. Flow
141 behavior of the dispersions were assessed by fitting the shear rate versus shear stress values to five
142 usual models, i.e., Newtonian ($\tau = m\dot{\gamma}$), Power-law ($\tau = m\dot{\gamma}^n$), Herschel-Bulkley ($\tau = m\dot{\gamma}^n + \tau_0$),
143 Bingham ($\tau = m\dot{\gamma} + \tau_0$), and Casson ($\sqrt{\tau} = m\sqrt{\dot{\gamma}} + \sqrt{\tau_0}$), Where τ is the shear stress (Pa), m is the
144 consistency coefficient (Pa.sn), $\dot{\gamma}$ is the shear rate (1 s^{-1}), the exponent n is flow behavior index
145 (dimensionless) and τ_0 is the yield stress (Pa) (Nejatian and Abbasi, 2019; Nejatian et al., 2018).

146 The effect of temperature on the flow properties was measured by performing shear sweeps for 1
147 % (w/v) OSP dispersion at 5 °C, 25 °C and 45 °C. Also, the temperature dependency of apparent
148 viscosity was evaluated by fitting the Arrhenius model ($\eta = \eta_0 exp^{\frac{E_a}{RT}}$) in which η_0 is the pre-
149 exponential factor (Pa.), E_a the activation energy for viscous flow (J/mol), R the universal gas
150 constant (8.314 J/mol K), and T the absolute temperature (K).

151 The 1 % (w/v) OSP gum dispersion was prepared for oscillatory shear measurements. Strain sweep
152 test was performed over the range 0.05–100 % at a fixed frequency (1 Hz) to determine the linear
153 viscoelastic region (LVR). Frequency sweep tests were also carried out at a wide range of
154 frequencies (0.01–20 Hz) and a constant strain (<LVR, ~ 0.5 %) to evaluate the dynamic
155 rheological properties (G′ and G'').

156 In addition, the rheological behavior in response to the salt concentration (0.2 M) and type (NaCl
157 and $CaCl_2$) were determined just at a certain gum concentration, 1 % (w/v) and temperature, 25
158 °C.

159 *2.6 Statistical analysis*

160 All the measurements were made in triplicate and data were presented as mean ± standard
161 deviation. Microsoft Excel Software (Microsoft Office, Package 2012) used for plotting the
162 rheological curves.

163 **3. Results and discussions**

164 *3.1. Compositional analysis*

165 The physicochemical and molecular parameters of the OSP are presented in Table 1. The results
166 showed the OSP contained 9.17% moisture, 1.88% ash, 2.14% of protein, 86.8% of total

carbohydrates, and DE of 79.68%. Plant's variety and growing conditions, extraction and purification process are important factors affecting the chemical composition of hydrocolloids (Razavi et al., 2014). Similar chemical composition for pectin extracted from sour oranges and the total ash, moisture content, and protein content were 1.89, 8.81, and 1.45, respectively (Hosseini et al., 2019).

Monosaccharides analysis using liquid chromatography (HPLC) showed that galacturonic acid was the major component in OSP (~ 425 mg/g, about 85% of sugar composition). This was followed by glucose (54 mg/g, about 10% of sugar composition). The wide diversity of composition by acid and glucose indicates that OSP has rich pectin content but also some cellulose or starch-like glucans. Such sugar composition was previously observed in the commercial low methoxyl pectin (Peng et al., 2020) and some pectin from different food waste streams (Müller-Maatsch et al., 2016). Also, minor quantities of fucose, galactose, arabinose, and rhamnose (about 4% of sugar composition) were also identified which can explain the complex polysaccharide composition of OSP (Razavi et al., 2014). It has been reported that galactose, rhamnose, arabinose, xylose and fucose are the principal neutral sugars found in pectin side chains (Hosseini et al., 2019). Hydrocolloids with a higher amount of fucose, xylose, galacturonic acid, methoxyl groups, and lower amounts of arabinose and nitrogenous fractions are reported to exhibit high viscosity (Anderson and Grant, 1988). Similar observations were also reported by Balaghi et al. (2011) and showed that tragacanth gums with a greater quantity of galacturonic acid and fucose exhibited higher consistency coefficients (Balaghi et al., 2011). Similarly, Hosseini et al. (2019) observed that galacturonic acid (65.3%) was the main monosaccharide of pectin extracted from sour orange peels (Hosseini et al., 2019).

3.2. Molecular weight parameters

The results of molecular weights parameters are presented in Table 1. M_w (weight-average molar mass), R_n (number average molar mass) and, R_z (z-average molar mass) for the OSP were 4511.8 (kDa), 61 (nm), and 61.1 (nm), respectively. The M_w measurement indicates a large M_w for OSP confirming that such polysaccharides have a tendency to exhibit a higher viscous and pseudoplastic properties when dissolved in water (Hosseini-Parvar et al., 2010). The high molecular weight of OSP is similar to other hydrocolloids such as xanthan (4200 kDa), locust bean gum (50-3000 kDa), Basil seed gum (1045-5980 kDa), guar (50-8000 kDa), psyhyllium (1500 kDa), and Karaya (10,000 kDa) (Faria et al., 2011; Harding et al., 2017; Imeson, 2011; Milani and Maleki, 2012; Naji-Tabasi et al., 2016). Different studies have shown that the pH of extraction may have an effect on the molecular weight of pectin and pectin extracted in higher pH shows the higher M_w due to the higher DE value of the pectin (Cho et al., 2019; Yapo et al., 2007). Using distilled water (pH=7) to extract pectin from OSP, may explain the high M_w observed for the extracted pectin in this research. Gavlighi and co-workers (2018) reported that the M_w of pectin extracted from pomegranate peels depended on the extraction condition and the highest M_w and R_g observed for the buffer extracted pectin (18,631.85 × 10^3 g/mol and 102.80 nm) (Gavlighi et al., 2018).

3.3. Rheological properties

3.3.1. Flow behavior

Figure 1a compares the flow behavior of OSP dispersions within a concentration range of 0.1-1 wt%. On the basis of the highest determination coefficient (R^2) and the lowest root mean standard error (*RMSE*), the flow behavior of all dispersions of OSP with different concentrations were particularly nonlinear and best fitted to Power-law model ($\tau = m\dot{\gamma}^n$). The coefficients of evaluated rheological models are shown in Table 2. The power law model has a wide application in food

212 industry and many studies have proved this model as the most appropriate model for flow behavior
213 study of the majority of food hydrocolloids (Khodaei et al., 2014).

214 The dependency of the apparent viscosity on the shear rate for the OSP dispersions in various
215 concentrations is shown in Figure 1b. As evident, the OSP dispersions indicated the typical
216 viscosity *vs* shear rate relationship of a colloidal food system including the polysaccharides
217 solutions. At lower shear rate (~ 0.1 1/s) and especially at higher gum concentrations (0.5-1 wt%),
218 the apparent viscosity was nearly independent of the shear rate. Indeed, when the shear rate is low,
219 the Brownian motion dominates the structural forces and favors the alignment of the elongated
220 coil along the flow direction. However, the viscosity reduced with increasing shear rate (shear
221 thinning behavior) arose from the equilibrium between the hydrodynamic forces and the structural
222 forces (Barnes, 2000; Windhab, 1995). In fact, above a critical shear rate, the deformation rate of
223 the gum chain entanglement due to the application of external forces becomes greater than the
224 formation rate of the new entanglement (Lapasin and Pricl, 1995).

225 As seen (Fig. 1b), the apparent viscosity decreased with reducing the gum concentration to 0.1%,
226 throughout the shear rate study. In addition, as the concentration of dispersion increases, the
227 intermolecular interactions increase and improve the viscosity. The positive effect of gum
228 concentration on the apparent viscosity can be also followed in Power law model parameters
229 (Table 1), so that with increasing in OSP concentration, the consistency coefficient (k) and flow
230 behavior index (n) increased and decreased, respectively. Generally, it seems that the apparent
231 viscosity of OSP is similar or even better than that of some other polysaccharides such as some
232 species of gum tragacanth (Balaghi et al., 2010), pectin (Marcotte et al., 2001), Persian gum
233 (Fadavi et al., 2014), guar gum (Kayacier and Dogan, 2006), carboxymethyl cellulose (Yasar et
234 al., 2007), etc.

Strain sweep dynamic rheological measurements showed that for OSP dispersion (1wt%) storage modulus (G′) was somewhat higher than loss modulus (G″) (Fig. 2a). Nevertheless, the superiority of each of these modules over the other in the frequency sweep test depended on the frequency. As is evident in Fig. 2b, there was the transition from a predominantly viscous response at longer time scales (G″ > G′) to a predominantly elastic response at shorter time scales (G′ > G″), indicating the dispersion has an entangled network structure. Such rheological behavior can be compared to some gum tragacanth species (*Astragalus parrowianus* and *fluccosus*) (Balaghi et al., 2011) and deacetylated *Sterculia striata* polysaccharide (De Brito et al., 2005).

3.3.2. Effect of salt and temperature on rheological properties

The Addition of salt (NaCl) to the OSP dispersion (1 wt%) decreased the apparent viscosity with no changes in the reduction pattern or flow behavior (Fig. 3a). Moreover, the consistency coefficient of 1 wt% OSP aqueous dispersion in the presence of 0.2 M NaCl decreased from 35.60 to 23.18 $Pa.s^n$ while the flow behavior index remained almost unchanged (0.22 against 0.25). This effect can be assigned to charge screening effect of the salt on long-range electrostatic repulsion among the uronic acid residues of OSP (Balaghi et al., 2010). Based on strain sweep test, the salt addition reduced amount of both G′ and G″, but the G′ values in LVR were still greater than G″ (Fig. 2a). On the contrary to the NaCl-free OSP dispersion, the dispersion containing NaCl (0.2 M) indicated a crossover point at a high frequency (Fig. 2b). Furthermore, in the presence of NaCl, the G′ and G″ gap was larger. This suggests that NaCl caused a more drastic decrease in elastic component than the viscous component. Interestingly, as is evident (Fig. 3a), incorporation of $CaCl_2$ to OSP dispersion increased the viscosity which can be related to the bridging effect of Ca^{2+} forming a strong gel network of polysaccharide chains.

Figure 3b shows the temperature influence on the apparent viscosity of 1 wt% OSP dispersion as a function of shear rate. As seen, the viscosity significantly declined by increasing in temperature from 5 to 45 °C. This effect can be also observed in Power law model parameters so that by increasing the temperature, the consistency coefficient decreased and the flow behavior index increased (Table 3). However, the flow behavior index of the dispersion is still far from the typical value of a Newtonian fluid (i.e., n = 1). In addition, the shear rate had a considerable effect on the temperature dependence of OSP dispersion viscosity. Figure 3c shows the value of activation energy obtained at two shear rates, 0.1 and 10 1/s for 1 wt% OSP aqueous dispersions. High R^2 values suggested that the apparent viscosity of dispersion in relation to temperature follows the Arrhenius model. A higher flow E_a value at low rates of shear implicates more sensitivity of OSP viscosity to temperature changes. This trend was also reported for other hydrocolloids such as pectin (da Silva et al., 1994).

4. Conclusion

The compositional and rheological behavior of pectin extracted from bitter orange seeds (OSP) as a novel source of high-quality pectin was evaluated in this study. Monosaccharides compositions revealed that galacturonic acid was the major structure of pectin followed by glucose. Arabinose, galactose, rhamnose, and fucose were also observed in smaller amounts and confirm the side chain structure of the pectin. Molecular weight analysis of the pectin showed a high M_w of 4512 kDa. OSP dispersions exhibited a non-Newtonian shear thinning behavior. Strain sweep dynamic rheological measurements exhibited a higher storage modulus (G′) than loss modulus (G″) and it confirms the entangled structure of OSP. In conclusion, the mucilage extracted from orange seeds

279 is rich in pectin and demonstrates potential use as gelling or emulsion stabilizer in food
280 applications. Further studies to evaluate the functional properties and characterization of this novel
281 source of pectin is needed but if fully elucidated could potentially revolutionize the use of pectin
282 in the food industry.

283 **Conflict of interest**

284 All the authors declare no conflict of interest.

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Table 1. Composition and molecular parameters of Bitter orange seed pectin (OSP)

Composition	
Moisture (%)	9.17 ± 0.37
Total ash (%)	1.88 ± 0.19
Total proteins (%)	2.14 ± 0.2
Total carbohydrates (%)	86.82 ± 0.33
DE (%)	79.68 ± 0.65
Monosaccharides (mg/g):	
Galacturonic Acid	424.99
Glucose	54.10
Rhamnose	8.37
Arabinose	5.54
Galactose	4.83
Fucose	1.78
M_w (kDa)	4511.75 ± 135.65
R_n (nm)	61± 0.8
R_z (nm)	61.1 ± 0.9

DE (Degree of esterification) M_n (number average molar mass), M_w (weight average molar mass) and M_z (z-average molar mass)

Table 2. Flow behavior of OSP dispersions (0.1-1% w/v) fitted to rheological models.

	Concentration (% w/v)				
Rheological models and variables	**0.1**	**0.3**	**0.5**	**0.7**	**1**
Newtonian					
K (Pa s^n)	0.008	0.019	0.043	0.070	
R^2	0.98	0.85	0.60	0.41	0.00
RMSE	0.252	1.607	5.937	11.740	
Power law					
K (Pa s^n)	0.005	0.490	3.068	7.206	35.6
n	1.05	0.504	0.348	0.290	0.219
R^2	0.98	1 (0.998)	1(0.992)	0.98	0.97
RMSE	0.259	0.151	0.798	1.869	8.314
Herschel–Bulkely					
K (Pa s^n)	0.003				
n	1.11				
τ_0 (Pa)	0.113				
R^2	0.98	-*	-*	-*	-*
RMSE	0.250				
Bingham					
K (Pa s^n)	0.008	0.018	0.037	0.057	0.156
τ_0 (Pa)	0.051	0.900	3.762	7.961	36.300
R^2	0.98	0.89	0.74	0.64	0.49
RMSE	0.259	1.39	4.846	9.192	33.720
Casson					
K (Pa s^n)	0.441	3.93	26.470	78.750	925.600
τ_0 (Pa)	2.22×10^{-14}	2.22×10^{-14}	2.22×10^{-14}	3.62×10^{-14}	3.37×10^{-13}
R^2	0.84	0.84	0.94	0.97	0.96
RMSE	1.667	1.667	2.161	2.423	9.478

***** Data fitting to Herschel–Bulkely and Bingham models for these samples indicated negative values of yield stress, so the obtained results for these two models in such samples were not reliable.

Table 3. Power law parameters and flow activation energy for OSP (1 % w/v) dispersion at different temperatures.

Temperature (°C)	**Power law parameters**			
	***K* (Pa.s^n)**	***n***	***R^2***	***RMES***
5	43.51	0.188	0.94	11.03
25	35.6	0.219	0.97	8.31
45	19.39	0.252	0.97	5.40

Figure 1. Comparison of the flow curves (a) and influence of shear rate on apparent viscosity of the OSP dispersions in different concentrations [0.1 wt% (◆), 0.3 wt% (■), 0.5 wt% (▲), 0.7 wt% (●), 1 wt%(✱)]

Figure 2. Comparison of the dependency of G′ (filled) and G″ (hollow) on (a) strain and (b) frequency for 1 wt% OSP dispersions in the absence (□, ■) and presence (○,●) of added NaCl (0.2 M).

Figure 3. Effect of salts (a; 0 wt% (●), 0.2 M NaCl (▲) and 0.2 M $CaCl_2$ (■) and temperature (b: 5 ºC (□), 25 ºC (○) and 45 ºC (△) on the apparent viscosity of 1 wt% OSP dispersion as a function of shear rate. Arrhenius plots (c) at shear rate of 0.1 1/s (■) and 10 1/s (□) for 1 wt% OSP dispersions.

Figure 1.

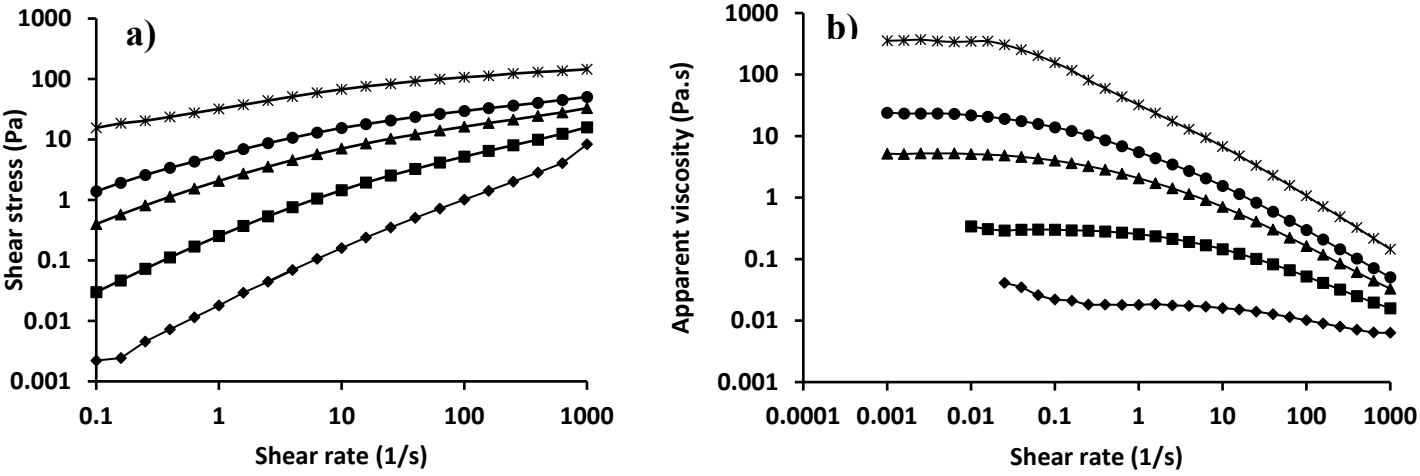

a)
Shear stress (Pa)
1000
100
10
1
0.1
0.01
0.001
0.1
1
10
100
1000
Shear rate (1/s)
b)
Apparent viscosity (Pa.s)
1000
100
10
1
0.1
0.01
0.001
0.0001
0.001
0.01
0.1
1
10
100
1000
Shear rate (1/s)

Figure 2.

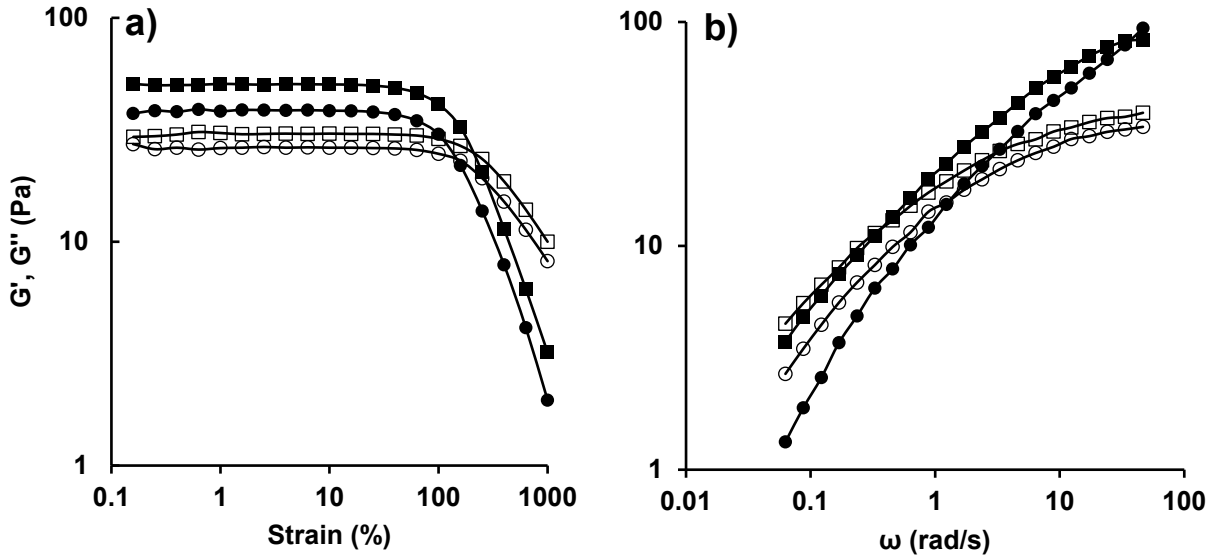
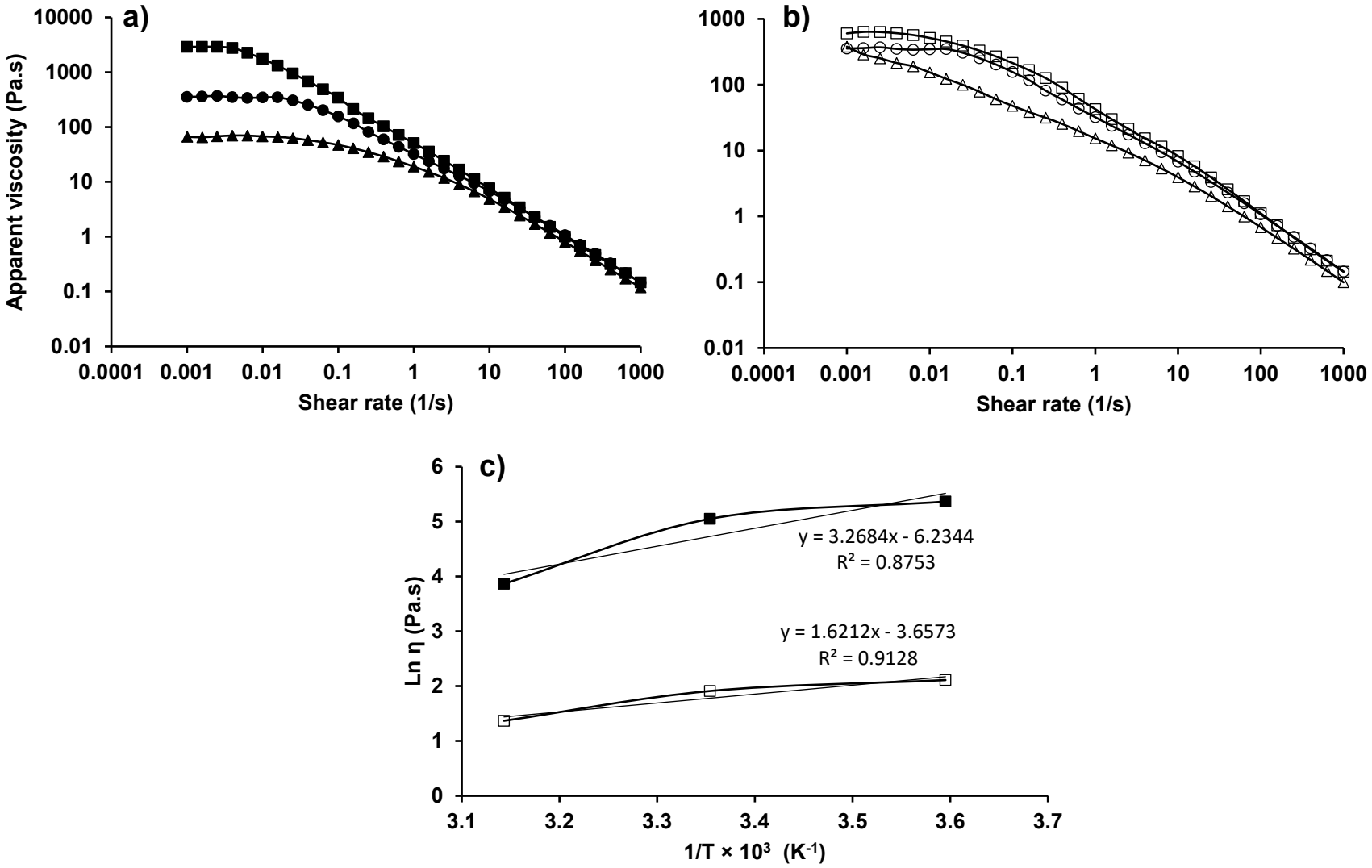

a)
100
10
1
G', G'' (Pa)
0.1
1
10
100
1000
Strain (%)
b)
100
10
1
0.01
0.1
1
10
100
ω (rad/s)

Figure 3.


a)
Apparent viscosity (Pa.s)
10000
1000
100
10
1
0.1
0.01
0.0001
0.001
0.01
0.1
1
10
100
1000
Shear rate (1/s)
b)
1000
100
10
1
0.1
0.01
0.0001
0.001
0.01
0.1
1
10
100
1000
Shear rate (1/s)
c)
Ln η (Pa.s)
6
5
4
3
2
1
0
y = 3.2684x - 6.2344
R² = 0.8753
y = 1.6212x - 3.6573
R² = 0.9128
3.1
3.2
3.3
3.4
3.5
3.6
3.7
1/T × 10³ (K⁻¹)