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# Rheological properties of pomegranate peel suspensions: The effect of fibrous material and low-methoxyl pectin at acidic pH

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ABSTRACT

The gel formation behaviour of four tunisian pomegranate peel powders was investigated at pH 3 in the presence of 30% sucrose and 1 g/L $CaCl_2$. All gels show a rapid formation with G'>G″, which reveals a typically gel-like structure. Since the mechanical properties of pomegranate peel gels are governed mainly by soluble pectins and/or insoluble fibrous material, the rheological properties of pectin and fibrous material gels were individually investigated. Results show that all systems displayed a typical solid-like behaviour, however the profiles obtained for the fibrous material gels (35 g/L fibrous material) were quantitatively higher than those obtained for pectins (7 g/L pectin). Thus, the properties of the peel gels seem then to be mostly governed by fibrous material. However, the G′ and G″ moduli obtained for fibrous material and pectin gels were significantly lower than those of peel suspensions. The rheological properties of peel gels would thus result from a strong synergism between fibrous material and pectins. The mechanical treatment was found to have a significant effect on fibrous material gel strength improvement which could be related to a lower sedimentation of suspensions. The particle size distributions revealed a decrease of fibrous material's particle size, and so an increase in surface area and a decrease of suspension's sedimentation. By heating samples at 82 °C for 1 h, trimodal distributions were obtained for peel samples.



## 1. Introduction

Pomegranate (*Punica granatum* L.) is a fruit that belonging to the Punicacea family and it is considered to be a functional product of great benefit in the human diet (Mditshwa, Fawole, Al-Said, Al-Yahyai, & Opara, 2013). The pomegranate fruits are normally consumed fresh or as fresh pomegranate juice, however, the pomegranate processing industry deals with the large percentage of byproducts such as peels.

Pomegranate peel which constitues up to 40% of the whole fruit, could have industrial applications and be used as ingredient in functional foods particularly as gelling agent due to its richness in pectin (the soluble pectic fraction) and fibrous material (the whole fibre complex including insoluble material). Gelation of pectins is greatly affected by both extrinsic and intrinsic parameters including the degree of methylation (DM). Indeed, the mechanism of gel formation is different in both high-methoxyl (HM) and low-methoxyl (LM) pectins. HM pectins (DM>50%) form gels if the pH is below 3.6 and a cosolute is present, typically sucrose at a concentration greater than 55% by weight (Thakur, Singh, & Handa, 1997). For low-methoxyl pectins (DM<50%), gelation is due to the chelation of calcium ions in regular arrays of electronegative cavities formed by the galacturonic acid residues. It requires so the presence of a controlled amount of calcium or other divalent cations. The gel formation occurs over a wide range of pH values, and the efficient $Ca^{2+}$-binding is an important factor both at low and high pH values (Cardoso, Coimbra, & Lopes da Silva, 2003). Pereira et al. (2016) reported that the degree of methylation of the extracted pectins from pomegranate peel, using citric acid, varied from 47% to 71%, so mostly high methoxyl pectins were extracted. On the other hand, Srivastava and Malviya (2011) reported that the

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composition of pectins (including the DM) varies with the source and the conditions applied during isolation.

Concerning the fibrous material, this fraction can provide a multitude of functional properties when it is incorporated in food systems. Thus, fibrous material addition contributes to the modification and improvement of the texture, sensory characteristics and shelf-life of foods due to their waterbinding capacity, gel-forming ability, texturising and thickening effects (Dikeman, Murphy, & Fahey, 2006; Gelroth & Ranhotra, 2001; Kunzek, Muller, Vetter, & Godeck, 2002). As fibrous plant cell walls play in important role in determining the food's quality that they go into, and as pectins and/or fibrous material would be the principal components governing the mechanical properties of the cell walls, knowing informations related to the rheology of these three components is highly important.

This study focuses on better understanding the rheological properties of pomegranate peel powder dispersions in water (from four varieties) in order to understand more precisely the effect of using pomegranate peel as gelling agent. The rheological behaviour of peel dispersion prepared from each variety was compared to that of pectin and fibrous material extracted from the same variety at acidic pH (pH3).

## 2. Materials and methods

### 2.1. Plant material

Mature pomegranate fruits, ecotypes “Acide” (Ac), “Gabsi” (Ga), “Nebli” (Ne) and “Tounsi” (To), were collected from the same oasis at Gabes region (southeast of Tunisia). Fruits were manually peeled then the collected peels were cut into small pieces. Samples were dried at 50 °C, ground and milled through 0.5 and 1.25 mm sieves. Final powders with sizes between 0.5 and 1.25 mm and particles size <0.5 mm were retained for pectin extraction and for gelation, respectively.

### 2.2. Pectin extraction

2 g of pomegranate peel was stirred at 400 rpm (Stirrer Heidolph RZR 20051 electronic, Germany) in 100 ml of the $HNO_3$ solution (solid–liquid ratio; 1:50; g/mL). The extraction conditions were 80 min, 86 °C and 20 mmol/L nitric acid. The resulting slurries were allowed to cool to room temperature (25 °C) and filtered through cheesecloth. Then, two volumes of 96% w/w ethanol were added to the filtrate for pectin precipitation and the obtained mixture was kept for 1 h at 4 °C. After centrifugation at 8000*g* for 20 min at 10 °C, the pectin precipitate was washed with 50%, 75% and two times with 100% ethanol and centrifuged at 5000 g for 10 min at 10 °C. Finally, the obtained pectin was dried at 45 °C to a constant weight, ground in a mortar and stored at room temperature for further gels' preparation.

### 2.3. Fibrous material extraction

The fibrous material was extracted from fresh pomegranate peel previously maintained in hot water (85 °C) at the ratio of 4:1 (v/w) for 5 min. The mixture was then filtrated with a thin cloth in order to separate the insoluble residue. The concentration of the fibrous material was realised by a succession of two rinsings (water at 40°C-10min) until the residue was exempted from simple sugars. The fibrous material extracts were dried at 50 °C to a constant weight then milled. Final powders with sizes <0.5 mm were retained for gelation.

### 2.4. Peel gel preparation

637.5 g demineralized water, 7.5 ml 54.3% w/v citric acid solution and sodium citrate solution (6% w/v) were mixed in a steel pot and 100 g peel powder, corresponding to 6.5–7.9 g dry pectin; 28–34 g total fibre (Table 1), was added. The suspension was heated at 82 °C while stirring for 1 h. Then about 40 g saccharose was added while stirring. The suspension was heated quickly until boiling, 224 g saccharose was added in 3 portions and the solution was boiled again. Afterwards, calcium chloride solution was added, giving calcium concentration of 0.16 g/g pectin, and while further boiling and stirring the total mass was reduced to 900 g (with final amounts: 0.72–0.88% pectin and 3.1–3.8% total fibre). The whole process should take no more than about 5 min.

### 2.5. Fibrous material gel preparation

In order to compare the rheological behaviour of peel and fibrous material gels, only the fibrous material suspension of To ecotype (To1) was prepared at the same conditions used previously for the gelation of peel dispersions (including heating at 82°C-1h) (section 2.4) with 3.5% of fibrous material (w/w) (since the final amount of fibre in peel gels corresponds to 3.1–3.8%). The dry solids content was reported to be as one of the parameters affecting gel strength (Imeson, 2010).

To get an indication of the effect of the dispersion mode, fibrous material suspensions of To ecotype were set for mechanical agitation with an Ultra Turrax at 20000 rpm for 3 and 6min. Then, the suspensions were heated quickly until 90 °C, saccharose was added and the solution was boiled again. Afterwards, calcium chloride solution was added. To3 and To6 are the fibrous material gels corresponding to 3 and 6min of mechanical agitation, respectively.

In order to compare the fibrous material dispersions' rheological behaviour of the different ecotypes, fibrous material extracted from Ac, Ga, Ne and To were subjected to gelation after mechanical agitation using an Ultra Turrax at 20000 rpm for 3min.

### 2.6. Pectin gel preparation

For gel preparation at pH3, distilled water, citric acid solution (54.3% w/v) and sodium citrate solution (6% w/v) (pH3) were mixed with dry pectin powder and stirred for 120 min at 20 °C at pH3, in order to get a complete hydration. The mixtures were then heated, saccharose was added in 3 portions to a final concentration of 30% and the solution was heated more until boiling. Afterwards, calcium chloride (1 g/L) was added and mixed under intense agitation. The solution was boiled again. The final pectin concentration was 7 g/L.

### 2.7. Rheological measurements

The applied rheometer was a Physica MCR 301 (Anton Paar, Germany). Oscillation measurements (temperature sweep) of storage modulus G′ and loss modulus G″, in the linear viscoelastic domain, were made using a coaxial cylinders wide gap CC18 for

**Table 1**
Sugar, fibre and pectin content in four selected pomegranate peels.

| (mg/g, DW) | Ac | Ga | Ne | To |
|---|---|---|---|---|
| Total sugar | 306.5 ± 7.0$^{a}$ | 335.8 ± 2.1$^{ab}$ | 330.0 ± 29.9$^{ab}$ | 348.3 ± 7.9$^{b}$ |
| Total fibre | 282.7 ± 9.0$^{a}$ | 281.0 ± 12.0$^{a}$ | 338.1 ± 4.2$^{b}$ | 339.3 ± 6.6$^{b}$ |
| Pectin | 66.3 ± 4.0$^{a}$ | 78.6 ± 8.1$^{b}$ | 75.9 ± 3.4$^{b}$ | 64.5 ± 2.3$^{a}$ |

Ac, Acide; Ga, Gabsi; Ne, Nebli; To, Tounsi; DW, dry weight basis. Each value in the table is represented as mean ± SE (n = 3). Significant differences between values in the same row are indicated by different letters ($P < 0.05$).