Original Research

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Changes in bioactive compounds, quality attributes and rheological behaviour of black grape juice caused by microwave and conventional heating

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Abstract

In this study, black grape juice containing different concentrations of soluble solids (15, 20, 30 and 40°Brix) obtained by microwave or conventional heating at different operational pressures (12, 38.5 and 100 kPa) was investigated for bioactive compounds, quality attributes and rheological behaviour. The more concentrated the juice, the higher the anthocyanin and phenolic compound degradation, which in turn resulted in decreased antioxidant potential. All Hunter colour parameters (L*, a* and b*) decreased with juice concentration. Microwave heating showed better performance compared to conventional thermal heating in terms of colour retention, anthocyanin and total phenolic contents, and the antioxidant activity of the juice concentrate. Samples processed at lower operational pressures showed a slighter decrease in quality attributes regardless of the heating method used. The results indicate that the use of a higher microwave power (600 W instead of 450 W) at an evaporation pressure of 38.5 kPa resulted in better preservation of quality characteristics against thermal destruction. The juice concentrates obeyed Newton's law and the heating method did not influence rheological behaviour.

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Introduction

Black grape (*Vitis vinifera* L.) juice is becoming more attractive to consumers due to its high levels of antioxidants, especially phenolic compounds [1]. Phenolic compounds are very important for grape quality owing to their contribution to colour, sensorial properties, oxidation reactions, interactions with proteins, and the ageing behaviour of grape juices and wines. The phenolic compounds in grapes mainly consist of flavonoids (anthocyanins, flavan-3-ols, flavonols and dihydroflavonols) and non-flavonoids (hydroxybenzoic and hydroxycinnamic acids and derivatives, stilbenes and volatile phenols) [2].

Anthocyanins, a group of water-soluble pigments, are major flavonoid compounds in grape juice [2] and are responsible for the blue, red and purple colours of leaves, flowers and fruit. Anthocyanins are found almost exclusively in the skin, with only a few varieties present in the pulp [3]. Anthocyanins also demonstrate a wide range of antioxidant and therapeutic activity including genomic DNA integrity, and potent cardioprotective, neuroprotective, anti-inflammatory and anticarcinogenic properties [4].

Interest has focussed on increasing the availabil-

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ity of phenolic compounds, especially anthocyanins, in food products during processing. Various thermal and non-thermal procedures have been exploited for concentrating fruit juice, including membrane concentration, ultrafiltration, nanofiltration and reverse osmosis. However, the removal of water through non-thermal processes has some significant drawbacks, for example higher costs and other operational limitations in obtaining a high solid content [5].

Several studies have noted that microwave-assisted extraction of bioactive compounds is energy efficient and provides higher recoveries quickly and with reduced solvent consumption while maintaining quality attributes such as nutritional value, colour and original flavour, making this procedure an attractive alternative to conventional extraction procedures [3, 6, 7]. It also provides homogeneous heating for the solvent and plant matrix and can be used instead of conventional heating to uniformly concentrate fruit juice [8].

The process of fruit juice concentration consists of partial removal of free water while the solid constituents of the fruit remain unchanged. The favourable quality attributes of the final product such as flavour, colour, aroma, appearance and mouth feel, depend on careful concentration of the fruit juice [9].

There are no reports in the literature on the effect of different heating methods on the quality and nutritional value of black grape juice. Therefore, the purpose of the present work was to study the effects of conventional heating and microwave heating at different operational pressures on the bioactive compounds, quality attributes and rheological behaviour of black grape juice during concentration.

Materials and methods

Chemicals and reagents

Methanol, potassium chloride, sodium acetate and sodium carbonate were supplied by Merck (Darmstadt, Germany). Gallic acid and Folin–Ciocalteu's reagent were provided by Sigma-Aldrich (Oakville, ON, Canada). Malvidin-3-*O*-glucoside and 2,2-diphenyl-1-picrylhydrazyl (DPPH) were purchased from Fluka (Buchs, Switzerland).

Sample preparation and grape juice concentration

Black grapes were obtained during the 2013–2014 growing season from a commercial farm in Paveh (Kermanshah, Iran). Ripe grapes were inspected carefully and intact, cleaned and uniform fruit was selected for further tests. The grapes were pressed gently using a commercial press (model AZS-SJ50/150; Zhengzhou Azeus Machinery Co., Zhengzhou, China) to obtain juice which was clarified using a cotton cloth with a 60–250 μm mesh. The clarified juice containing 20% of total soluble solids was rapidly cooled and frozen at –25°C until use.

Juice concentration methods

Microwave heating method

A programmable domestic microwave oven (Butane MR-1; Butan, Tehran, Iran) with a maximum output power of 900 W at 2,450 MHz was used for microwave evaporation. Pressure applied by a pump was kept constant using a vacuum controller device (CVC2111; Vacuubrand, Littleborough, UK). Operational parameters, such as microwave power and pressure, were monitored using a PC, and the temperature was recorded intermittently. Juice samples were taken periodically for analysis. Juice was concentrated from an initial concentration of 10°Brix to a final concentration of 40°Brix, under 100 (atmospheric pressure), 38.5 and 12 kPa.

Conventional heating method

A rotary vacuum evaporator (Heizbad HB Control; Heidolph, Schwabach, Germany) was employed for convectional heating at 12, 38.5 and 100 kPa.

Soybean oil was used as the operative liquid because of its high boiling temperature of 120°C. Sampling was performed using the same method as for microwave heating. Data were recorded without interrupting the process.

Total soluble solid content measurement

The total soluble solid content of concentrated juice was determined after evaporation using an Abbe refractometer (Atago Rx-7000a; Tokyo, Japan) at 20°C and expressed in °Brix.

Colour analysis and browning index
The colour parameters (L^*, a^* and b^*) of fruit juices were measured using a HunterLab Labscan XE colorimeter (HunterLab, Reston, VA). The colorimeter was calibrated with a standard reference having L^*, a^* and b^* values of 97.55, 1.32 and 1.41, respectively. The colour parameters were used to calculate the browning index (BI) using the following formula:

$$BI = \frac{100(x-0.31)}{0.17}$$

$$x = \frac{a^*+1.75L^*}{5.645L^*+a^*-3.012b^*}$$

Evaluation
Total phenolic compounds
The total phenolic content in the juice was measured at 760 nm on a UNICO spectrophotometer (UNICO, Dayton, NJ, USA) according to the modified Folin–Ciocalteau method [10, 11]. Gallic acid was used for plotting the standard curve and total phenol content was represented as milligrams gallic acid equivalent per gram dry weight (mg GAE/g sample).

Determination of total anthocyanin content
The total anthocyanin content of the grape juice was calculated by the pH differential method according to Giusti and Wrolstad [12] using a spectrophotometric assay whereby absorbance of extracts is measured at pH 1.0 and 4.5. The absorbance of the samples was determined at 5100 and 700 nm using a spectrophotometer and total anthocyanin content (mg/l) was stated as malvidin-3-*O*-glucoside according to the following equation:

$$\text{Total anthocyanin content}\left(\frac{mg}{L}\right) = \frac{A\times MW\times DF\times 1000}{\varepsilon\times C}$$

where A is the absorbance of the juice and calculated as follows:

$$A = (A_{510} - A_{700})_{pH\ 1.0} - (A_{510} - A_{700})_{pH\ 4.5}$$

and where MW is the molecular weight of malvidin-3-*O*-glucoside (493.4 g/mol), DF is dilution factor, ε is the molar absorptivity of malvidin-3-*O*-glucoside (28,000) and C is the concentration of the buffer in mg/ml. The results were represented as mg malvidin-3-*O*-glucoside equivalents/g dry weight (mg m-3-gE/g dry weight).

Antioxidant activity
The DPPH scavenging capacity of the juices was evaluated according to Garavand *et al.* [13] with some modifications. In brief, after dilution in distilled water (1:20), 0.1 ml of fruit juice was mixed with 3.9 ml methanolic DPPH solution (25 mg/l), shaken thoroughly and left to stand for 30 min at room temperature. A DPPH solution with no added extract was used as control. The percentage inhibition of DPPH was calculated at 515 nm according to the following equation:

$$\text{Inhibition of DPPH}(\%) = \frac{A_{control}-A_{sample}}{A_{control}}\times 100$$

where A_{sample} and $A_{control}$ are the absorbance of the sample with DPPH and the absorbance of the control DPPH solution, respectively.

Viscosity measurement
The viscosity of fruit juice was measured with a rotational programmable viscometer (LVDV-II Pro; Brookfield Engineering, Middleboro, MA, USA) using an LV spindle at 25°C. About 20 ml of each sample was poured into the device cylinder and the shear rate was programmed to increase from 1.6 to 76.6 s^{-1} at 5 s intervals.

Statistical analysis
The data are reported as means±standard deviation. The results of different treatments were compared using analysis of variance (ANOVA) with SPSS Statistics (Version 18.5). When significant differences were identified, individual comparisons were conducted with Tukey's test. Values of $p<0.05$ were considered statistically significant for comparison of means.

Results and discussion

Colour parameters
Fig. 1 shows the change in L^* value with juice concentration for the two evaporation methods at different operational pressures. The more concen-

trated the juice, the darker were the samples regardless of the heating method used. The changes in a* and L* values followed a similar trend to each other (Fig. 2). A decrease in Hunter colour parameters during the thermal processing of fruit juice has been observed by many authors [14, 15]. It is thought that non-enzymatic browning and polymerization reactions are largely responsible for L* loss, while the decrease in other colour parameters (a* and b*) is mostly attributed to pigment destruction [16]. The results indicated that colour parameters (L* and a*) declined less in juices concentrated using the microwave compared to those heated conventionally. This is in accordance with the results of Maskan [16]. As seen from Figs. 1 and 2, lower operational pressures showed better product colour retention (L* and a*) for both the microwave and conventional methods. Several researchers have shown that the time required to obtain a specific concentration for heated fruit juices is dramatically reduced with decreasing operational pressure [5, 9, 17, 18], which in turn may better protect product colour pigments against thermal destruction. Therefore, less colour loss in microwave-concentrated juices may be due to the shorter time required to reach the desired concentration. The results indicated that a higher microwave power of 600 W compared to 450 W is less destructive to juice colour during the concentration process. However, in contrast to the trend in L* value changes, the value of a* for all treatments increased until juices were concentrated to 20ºBrix and then declined. Increased redness could be closely related to pigment concentration in the initial stages of the concentration process [5].

As a result of changes in Hunter colour parameters, BI values also changed, so that BI values increased as the juices became more concentrated by both processing methods (Fig. 3). Browning reactions, such as the Maillard or caramelization reaction, seem to be enhanced at higher juice concentrations, due probably to the lower water content and higher temperature. As shown in Fig. 3, the trend for increased BI with increased concentration decreased in an operational pressure-dependent manner. This phenomenon was more evident for juices subject to conventional than microwave heating, but BI values were lower for those concentrated using microwave energy at the

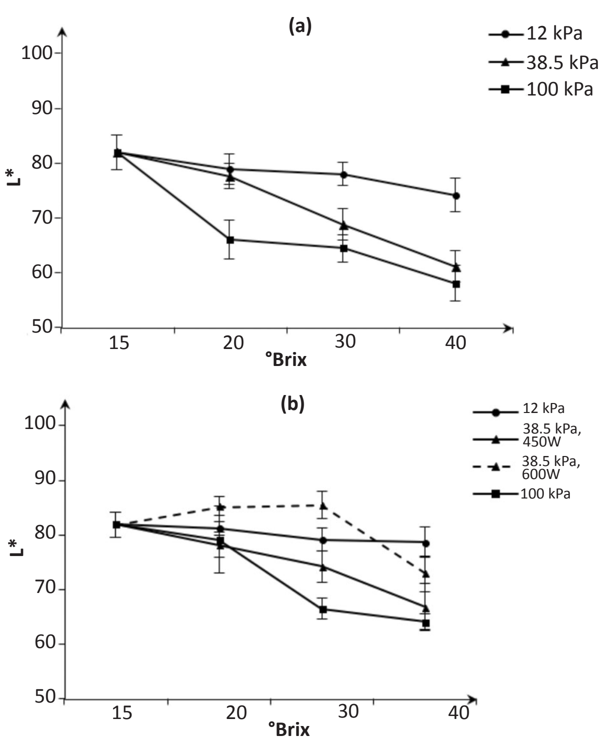

Figure 1 - L* values for grape juice with different soluble solid content obtained by (a) conventional and (b) microwave heating methods at different operational pressures

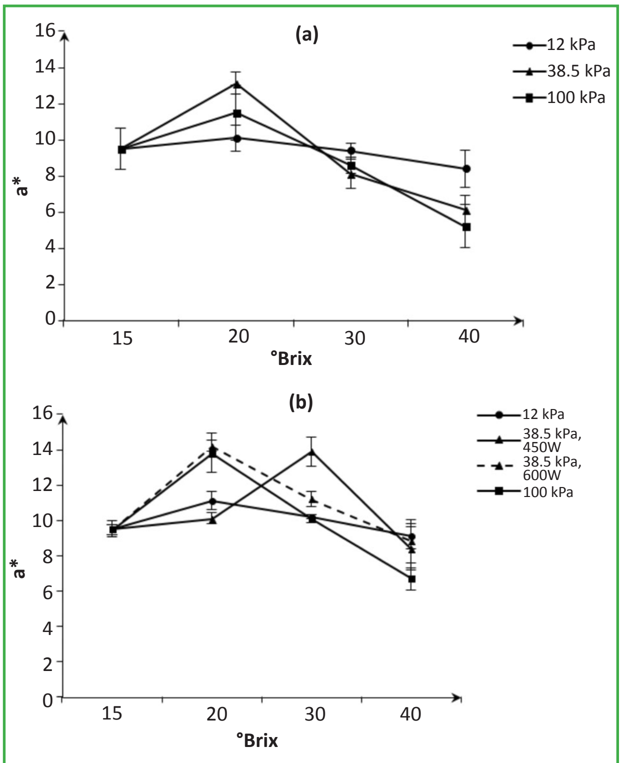

Figure 2 - a* Values for grape juice with different soluble solid content obtained by (a) conventional and (b) microwave heating methods at different operational pressures

same evaporation pressures. As mentioned above, the shorter time required to concentrate juices at lower operational pressures could explain our observation of lower BI values.

Bioactive compounds

Anthocyanins (phenolic derivatives) are responsible for flower, fruit and vegetable colour [19]. Consequently, loss of food colour during processing can reflect anthocyanin degradation [17]. Changes in total phenolic and anthocyanin levels in grape juice with different soluble solid contents at different operational pressures are shown in Figs. 4 and 5. Changes in the anthocyanin and phenolic contents of juices during processing showed the same trend as changes in colour parameters. The results demonstrate that the more concentrated the juice, the lower the phenolic and anthocyanin contents for both conventional and microwave heating methods (Figs. 4 and 5). Polyphenols and anthocyanins are heat sensitive and can be easily destroyed during thermal processing of fruit juices [20]. Many researchers have shown that the degradation of phenolic and anthocyanin compounds is dependent on temperature [21–24].

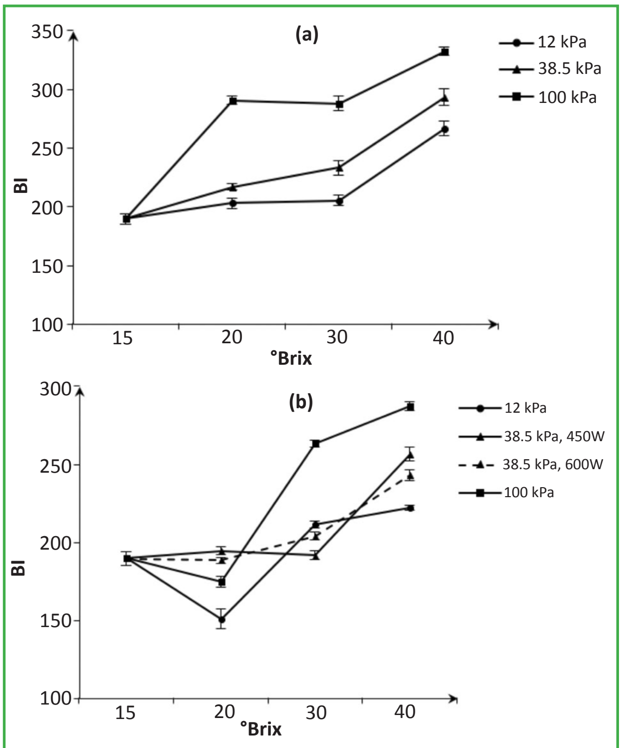

Figure 3 - Browning index (BI) values for grape juice with different soluble solid content obtained by (a) conventional and (b) microwave heating methods at different operational pressures

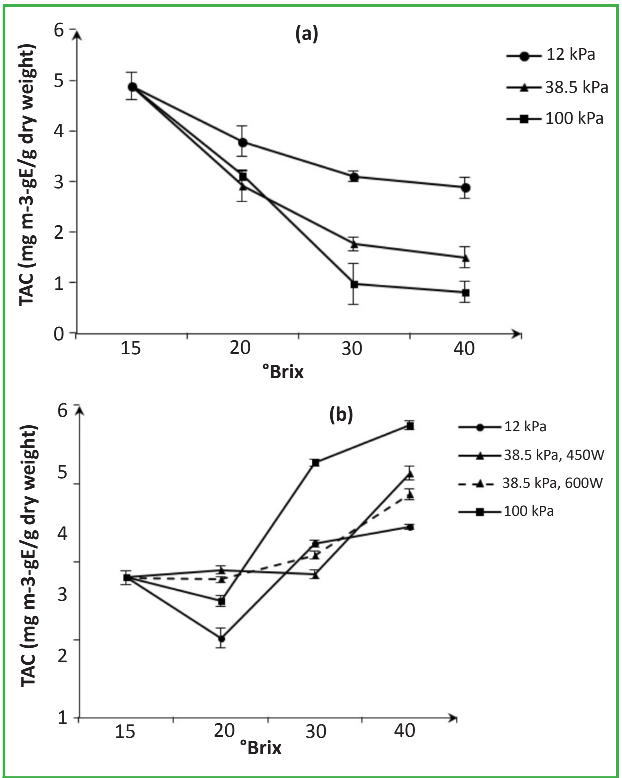

Figure 4 - Total anthocyanin content (TAC) values for grape juice with different soluble solid content obtained by (a) conventional and (b) microwave heating methods at different operational pressures

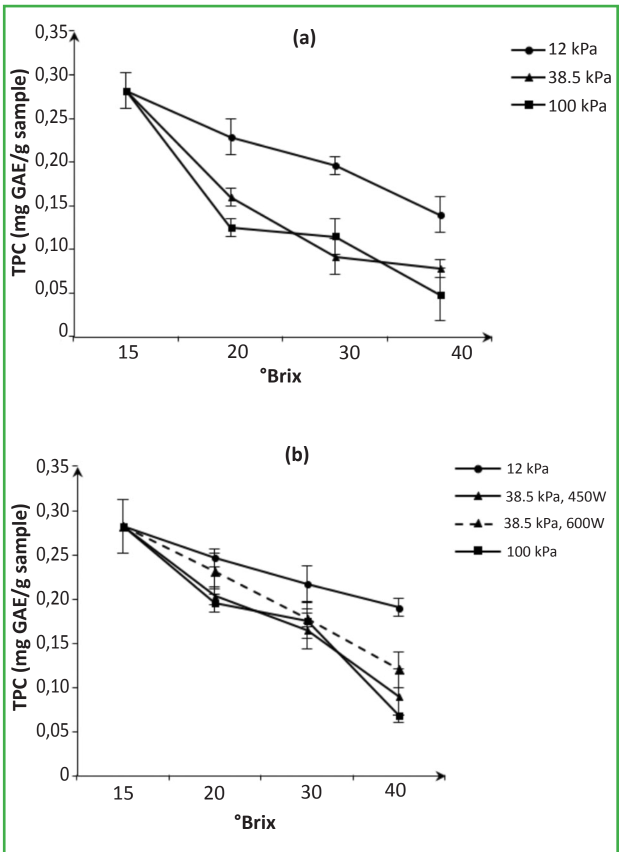

Figure 5 - Total phenolic compound (TPC) values for grape juice with different soluble solid content obtained by (a) conventional and (b) microwave heating methods at different operational pressures

Hillmann *et al.* [19] found that activation energy in the Arrhenius model used to describe the dependence of anthocyanin degradation on temperature, decreased with increasing grape juice concentration. Similar results were reported by Wang and Xu [25] for blackberry juice. As shown in Figs. 4 and 5, phenolics and anthocyanins showed lower thermal stability in juice processed at higher operational pressures. This decreasing trend of nutritional quality with increasing evaporation pressure was more pronounced with rotary evaporation than with microwave heating. Corresponding findings have been reported by Yousefi *et al.* [5] concerning pomegranate juice concentrate, Yousefi *et al.* [20] concerning raspberry juice concentrate and Hojjatpanah *et al.* [17] and Fazaeli *et al.* [18] concerning black mulberry juice concentrate. The results also revealed that microwave heating at 600 W compared to 450 W at a pressure of 38.5 kPa conserved phenolic and anthocyanin contents against thermal degradation more effectively (Figs. 4 and 5). This is consistent with the results of Yousefi *et al.* [26] who observed higher nutritional quality in raspberry juice concentrated using a microwave-assisted fluidized bed dryer at higher microwave power.

Since the antioxidant properties of fruit are mainly due to their anthocyanin and phenolic contents, it was expected that changes in the antioxidant activity of juice concentrates would reflect anthocyanin and phenolic content. Antioxidant activity decreased with increasing operational pressure and soluble solid contents (Fig. 6). Antioxidant activity was better preserved in juices concentrated with microwave heating than the conventional method. Shorter processing times at lower pressures, especially for microwave-heated samples, resulted in lower thermal degradation of the main contributors (anthocyanin and phenolic compounds) to antioxidant activity.

Viscosity

Changes in the apparent viscosity of grape juice concentrated by different methods at various operational pressures are presented in Fig. 7. Increasing the soluble solid contents of juices resulted in an increase in apparent viscosity regardless of concentration method, but the rate of viscosity increase declined when microwave heating was used. Yousefi *et al.* [5] pointed out that the increasing temperature of juices during the concentration

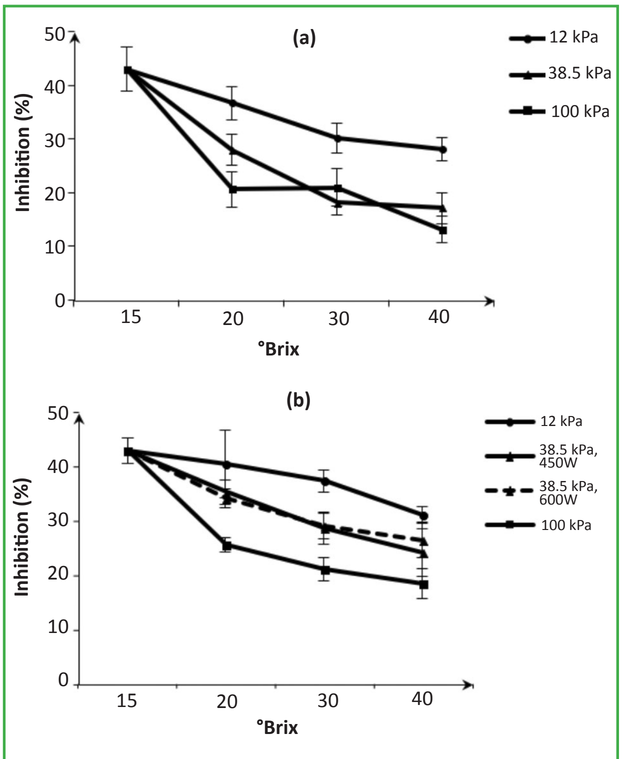

Figure 6 - % Inhibition values for grape juice with different soluble solid content obtained by (a) conventional and (b) microwave heating methods at different operational pressures

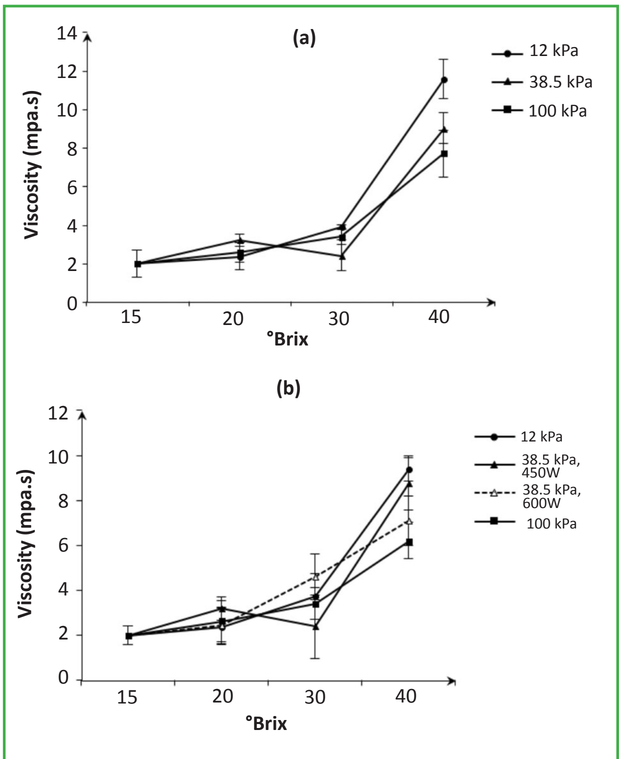

Figure 7 - Apparent viscosity values for grape juice with different soluble solid content obtained by (a) conventional and (b) microwave heating methods at different operational pressures

process was more noticeable for the microwave heating method compared with conventional thermal processing. Kaya and Sözer [27] reported decreased pomegranate juice concentrate viscosity with increasing process temperature. It is noteworthy that fruit juice concentrates processed at higher evaporation pressures had a lower apparent viscosity, possibly due to the higher temperature of the concentration process. All treatments obeyed Newton's laws (data not shown) and the heating method did not influence the rheological behaviour of the juice concentrates. Our results agree with those of Kaya and Sözer [27] who demonstrated that pomegranate juice concentrate in the range of 45.7–71°Brix followed Newton's laws. Similarly, Altan and Maskan [28] found no difference between the rheological behaviour of pomegranate juice concentrated with different heating methods.

Conclusion

Operational pressure played a key role in the quality of grape concentrate obtained by conventional and microwave heating methods. The best concentrate quality was obtained when the lowest evaporation pressure (12 kPa) was applied. Microwave treatment has potential as an alternative to traditional heating methods for the concentration of grape juice. Microwave heating at higher microwave power (600 W) resulted in less loss of both nutritional quality and Hunter colour parameters during the concentration process. No differences were noted between the rheological behaviour of juice concentrates produced by the different heating methods.

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