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Research Note

Effect of high pressure homogenization (HPH) on the rheological properties of a fruit juice serum model

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ABSTRACT

High pressure homogenization (HPH) is a non-thermal technology that has been widely studied as a partial or total substitute for thermal food processing. The present work evaluated the effect of HPH on the rheological properties of a fruit juice serum model, designed to be similar to tomato juice serum. Product viscosity decreased due to the increase in homogenization pressure, and could be modelled well using two functions (power-sigmoidal and exponential; $R^2 > 0.98$). The serum model processed at 200 MPa showed a viscosity decrease of 20% when compared to the original. Since fruit juice rheology is defined by the interactions occurring between the dispersed phase (suspended particles) and the solution (serum), the expected fruit juice behaviour was then discussed.

1. Introduction

High pressure homogenization (HPH) technology has been studied by many authors as a non-thermal food preservation technique, especially for fruit products. The use of HPH as a partial or total substitute for thermal food processing has been previously proposed for many vegetable juices. However, there is a lack of information in the literature regarding changes in the rheological properties of fruit products due to HPH processing.

The rheological characterization of food is important for the design of unit operations, process optimization and high quality product assurance. The study of the influence of processing on the rheological properties of food is thus essential for an efficient product and process design.

Fruit juices are composed of an insoluble phase (the pulp) dispersed in a viscous solution (the serum). The dispersed phase, or pulp, is constituted of fruit tissue cells and their fragments, cell walls and insoluble polymer clusters and chains. The serum is an aqueous solution of soluble polysaccharides, sugars, salts and acids. The fruit juice rheological properties are thus defined by the interactions within each phase and between them.

The effect of HPH on the rheological properties of fruit juices will thus be a function of the balance between the structural changes in the pulp and serum. Therefore an evaluation of the effects of processing on the serum phase is important for a better understand of the effect of HPH on the juice rheological properties. However, no such work was found in the literature describing the effect of HPH on the rheological properties of fruit juice serum.

The use of model foods for process studies enables one to carry out simple, cost-effective and continuous experiments, without significantly changing the products (Berto et al., 2003). Moreover, the main benefit of using model food systems in scientific studies is the experimental reproducibility, which minimizes the effects of inherent variations in food characteristics (Augusto et al., 2011b).

Tomato is one of the most popular and widely grown vegetables in the world. In fact, tomato is one of the most important vegetables in the food industry, and is widely consumed and included in the human diet (Augusto et al., 2010).

Tomato serum generally has a soluble solids content equivalent to 4–6°Brix, with approximately 0.2–0.3% of pectin and a pH value of 3.8–4.5 (Augusto et al., 2010; Tanglertpaibul and Rao, 1987; Takada and Nelson, 1983). A citric pectin solution was previously used by Takada and Nelson (1983) to evaluate tomato serum.

The degree of esterification (DE) of tomato pectin is a function of the process adopted, especially the blanching method used (hot or cold break), and the pectin DE of hot break tomato pulp is approximately 58–62% (Ouden et al., 1997; Chou and Kokini, 1987).

Therefore, due to its industrial importance, tomato juice serum was chosen for evaluation in this work, and the serum model was designed to be similar to this product. Thus the present work evaluated the effect of high pressure homogenization (HPH) on the

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Nomenclature

$\dot{\gamma}$	shear rate (s^{-1})	σ	shear stress (Pa)
φ	particle volume fraction (-)	σ_0	yield stress, Herschel-Bulkley's model (Pa)
η	viscosity (Pa·s)	k	consistency coefficient, Herschel-Bulkley's model (Pa s^n)
η_r	relative viscosity (Eq. (3)) (Pa·s)	n	flow behaviour index, Herschel-Bulkley's model (-)
$[\eta]$	intrinsic viscosity (Pa s)	P_H	homogenization pressure (MPa)

rheological properties of a fruit juice serum model, designed to be similar to tomato juice serum.

2. Materials and methods

2.1. Fruit juice serum model

The fruit juice serum model evaluated here was composed of 5% (w/w) glucose and 0.3% (w/w) 58–62% DE pectin (citric pectin; Grindsted Pectin XSS100, Danisco) in distilled and deionized water (Milli-Q, Millipore, São Paulo, Brazil). The solution pH was adjusted to 4.2 using 0.1 N NaOH.

The glucose and pectin were dispersed in water vigorously agitated at 1000 rpm using a mechanical stirrer (Fisatrom 713D, São Paulo, Brazil). Then, it was allowed to rest overnight at 5 °C for complete hydration and release of air bubbles.

2.2. High pressure homogenization (HPH) process

HPH processes were carried out using a high pressure homogenizer (Panda Plus, GEA Niro Soavi, Italy), with nominal maximum pressure operation up to 200 MPa. Samples were introduced into the equipment at 10 °C by suction, and quickly cooled using an ice bath just after the homogenization valve. The maximum temperature reached was 37 °C (sample processed at 200 MPa, just before the ice bath).

The serum model was processed at 0, 25, 50, 75, 100, 150 and 200 MPa. The processed samples were refrigerated (5 °C) and their rheological properties evaluated on the same day.

2.3. Rheological properties

Rheological measurements were carried out with a controlled stress ($\sigma\sigma$) rheometer (AR2000ex, TA Instruments, USA), with cone-plate geometry (2°, 60 mm of diameter). The temperature was maintained constant at 25 °C by a Peltier system.

The steady-state shear experiments were carried out in the shear rate ($\dot{\gamma}$) range of 0.1–100 s^{-1}. Samples were submitted to shear at 300 s^{-1} for 5 min to avoid thixotropy (data not shown). Product flow behaviour was modelled using the best fit with the Newton, Bingham, Ostwald-de-Waele (power law) and Herschel-Bulkley models.

The experiments were carried out with three replicates of freshly prepared samples, and the regression calculated for each replicate. The parameters for each model were obtained by linear or non-linear regression using the software CurveExpert Professional v.1.0.1, and a significant probability level of 95%.

3. Results and discussions

Fig. 1 shows the flow curves (σversus $\dot{\gamma}$) for the fruit juice serum model homogenised at 0, 25, 50, 75, 100, 150 and 200 MPa. As expected, the serum model showed Newtonian behaviour ($R^2 > 0.99$), with a viscosity (η) close to the values reported for tomato serum (Table 1). Moreover, the serum model viscosity was close to that of other vegetable juices such as carrot, red raspberry, blueberry, pineapple and pomegranate juices, and clarified cashew, grape, peach, orange, cherry, pomegranate and *Malus floribunda* juices (Table 1).

The product viscosity showed a tendency to decrease with increasing homogenization pressure (P_H; Fig. 2). Similar results were reported for pectin solutions (Corredig and Wicker, 2001) and other biopolymers such as carboxymethylcellulose (CMC, Floury et al., 2002), flaxseed gum (Wang et al., 2011), xanthan gum (Harte and Venegas, 2010; Lagoueyte and Paquin, 1998), alginate and κ-carrageenan (Harte and Venegas, 2010).

Corredig and Wicker (2001) evaluated the effect of homogenization at 124 MPa on the viscosity of three pectins (0.3 mg mL^{-1}) with different DE values. While the homogenization process did not change the viscosity of two of the pectins, it did change the viscosity of the 70% DE pectin from 7 mPa·s (0 MPa) to 5 mPa·s (124 MPa). Moreover, the authors evaluated the change in molecular weight of the pectins, and showed that sample polydispersity had increased, while the average molecular weight had decreased, due to the homogenization pressure, thus explaining the results obtained.

A reduction in average molecular weight of the carbohydrates due to homogenization was also observed by Floury et al. (2002) for carboxymethylcellulose (CMC) and by Lagoueyte and Paquin (1998) for xantan gum. In both cases, the homogenization process reduced the pseudoplastic behaviour (i.e. increased the flow behaviour index – n) and consistency (i.e. decreased the consistency index – k) of the products. Floury et al. (2002) suggested that the accentuated shear stress encountered by the polymer chain at the homogenization valve might produce sufficient energy to disrupt covalent bonds. Lagoueyte and Paquin (1998) suggested that the turbulence, cavitation and high shear stress produced during processing had an effect on the molecular conformation; first inducing the molecule to an ordered-disordered conformation

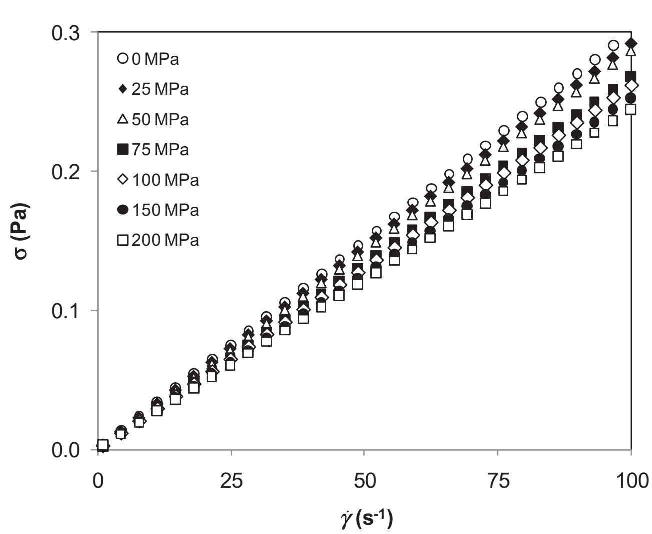

Fig. 1. Fruit juice serum model flow curve (25 °C) as a function of P_H. Mean of three replicates; vertical bars represent the standard deviation for each value.

Table 1
Viscosity values for fruit products.

Product	T (°C)	η(mPa·s)	Refs.
Fruit juice serum model	25	3.0	Present work
Tomato serum	20–25	1.3–6.0	Beresovsky et al. (1995), Tanglertpaibul and Rao (1987)
Carrot juice (7.6–8.4°Brix)	25	1.3–3.5	Vandresen et al. (2009)
Clarified cashew juice (12.1°Brix)	25	1.3	Cianci et al. (2005)
Red raspberry juice (10°Brix)	20	1.8	Nindo et al. (2005)
Clarified grape juice (22.9°Brix)	25	2.0	Zuritz et al. (2005)
Clarified peach juice (12.3°Brix)	20	2	Augusto et al. (2011a)
Blueberry juice (10°Brix)	20	2.9	Nindo et al. (2005)
Clarified orange juice (10–40°Brix)	25	2.4–5.8	Ibarz et al. (2009)
Clarified cherry juices (22–35°Brix)	20	2.9–5.4	Giner et al. (1996)
Clarified pomegranate juice (45.3°Brix)	20	3.5	Kaya and Sözer (2005)
Pineapple juice (4.3°Brix)	25	4	Shamsudin et al. (2009)
Clarified *M. floribunda* juice (44–45°Brix)	25	5–6	Cepeda and Villarán (1999), Cepeda et al. (1999)
Pomegranate juice (17.5°Brix)	25	7.4	Altan and Maskan (2005)

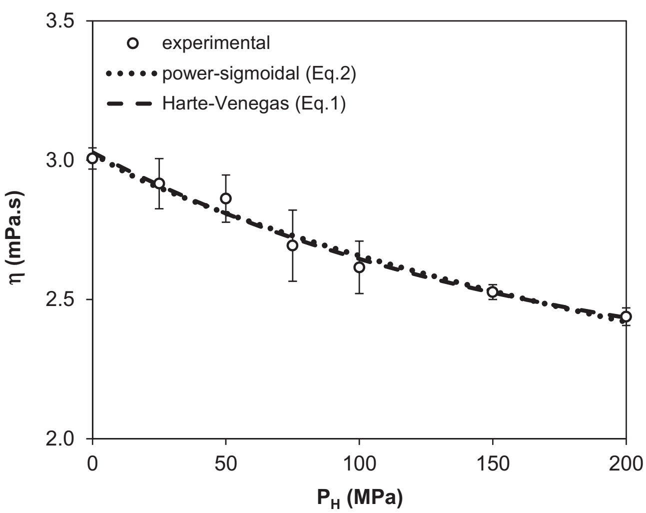

Fig. 2. Fruit juice serum model viscosity (25 °C) as a function of P_H. Mean of three replicates; vertical bars represent the standard deviation for each value; curves are the evaluated models.

transition, and then degrading the molecule. Similar behaviour was expected for the serum model evaluated here.

Fig. 2 shows the viscosity of the fruit juice serum model viscosity (25 °C) as a function of the homogenization pressure (P_H). The falling tendency was observed, and was modelled using two functions. Harte and Venegas (2010) proposed an exponential function (Eq. (1)) to describe the effect of homogenization pressure (P_H) on the viscosity (or apparent viscosity) of polymer suspensions. This function was used to model the present results (Eq. (1), P_H in MPa, $R^2 > 0.98$). Moreover, due to the sigmoidal shape of the curve (Fig. 2), a power-sigmoidal function was used (Eq. (2), P_H in MPa, $R^2 > 0.98$) to model the effect of homogenization pressure (P_H) on the serum model viscosity. The two equations explain the reduction in viscosity due to the homogenization pressure (P_H) in the evaluated serum model well, which is of interest for the evaluation and design of HPH processing.

$$\eta(\text{mPa}\cdot\text{s}) = 3.03\left[\frac{2.55+\exp(-0.0059\cdot \text{P}_\text{H})}{2.55+1}\right] \quad (1)$$

$$\eta(\text{mPa}\cdot\text{s}) = \frac{3.02}{1+(0.0099\cdot \text{P}_\text{H})^{0.856}} \quad (2)$$

The rheological properties of fruit juices are determined by interactions amongst the suspended particles and between these particles and the serum. The relative viscosity η_r of a dilute dispersion of solid particles in a liquid medium is described by the Einstein Equation (Eq. (3), Genovese et al., 2007; Metzner, 1985), where $[\eta]$ is the intrinsic viscosity and φ is the particle volume fraction.

$$\eta_r = \frac{\eta_{\text{dispersion}}}{\eta_{\text{continuous\_phase}}} = 1 + [\eta]\cdot\varphi \quad (3)$$

According to this equation, the dispersion viscosity is affected by the continuous phase viscosity, the particle intrinsic viscosity (which depends on the particle shape) and the relative volume occupied by the particles. For fruit juices, the dispersed phase (pulp) is formed of fruit tissue cells and their fragments, cell walls and insoluble polymer clusters and chains. Although more complex and in most cases more concentrated than the fluid proposed in the Einstein Equation, a qualitative evaluation can be carried out based on this equation.

In the present work, the serum model viscosity represented a fruit juice continuous phase, and its viscosity decreased due to the HPH. Thus, the behaviour observed suggests a reduction in juice viscosity when processed by HPH. However, the final juice behaviour will also be a function of the particle volume fraction and particle intrinsic viscosity.

Bayod and Tornberg (2011) studied the microstructure of tomato suspensions homogenized at 9 MPa. The authors observed an increase in the surface area covered by particles and in its volume fraction, with a reduction on particle size due to the homogenization process. In fact, Bayod et al. (2007) observed increases in consistency and yield stress (σ_0) of the tomato concentrate when homogenized at 9 MPa. Yoo and Rao (1994) observed that the smaller the suspended particles were, the smaller the distance between them and the greater the interaction amongst them.

Silva et al. (2010) studied the effect of homogenization (up to 70 MPa) on pineapple pulp properties. Although the authors observed the same reduction in particle size, the product consistency showed the opposite behaviour of that reported for tomato products: the homogenization process reduced the pseudoplastic behaviour (i.e. increased the flow behaviour index – n) and consistency (i.e. decreased the consistency index – k) of the pulp. Similar behaviour was observed by Donsì et al. (2009) for apple juice, where the viscosity and suspended particle size were reduced by HPH processing.

Considering the reduction in viscosity due to HPH observed here and those reported for other carbohydrates (Wang et al., 2011; Harte and Venegas, 2010; Floury et al., 2002; Corredig and Wicker, 2001; Lagoueyte and Paquin, 1998), the different results obtained for tomato, apple and pineapple juices cannot be assigned to the serum phase and must be associated with differences in the dispersed phase.

In fact, Lopez-Sanchez et al. (2011) showed that the cell walls of each vegetable have different behaviours when processed by HPH. While carrot tissue requires high shear values to be disrupted, tomato cell walls were broken with moderate shear values. This suggests that the effect of HPH processing is different for each fruit product, and highlights the need for better understanding of this process.

Once again this highlights the fact that the effect of HPH on the rheological properties of fruit juices is a function of the balance between the structural changes in the pulp and serum. The present work showed the behaviour expected for the serum phase using a tomato juice serum model. The results obtained are potentially useful for understanding juice behaviour, but further work is necessary for a better understanding of the effect of high pressure homogenization on the rheological properties of tomato juice. In fact it is the subject of our next works.

4. Conclusions

The present work evaluated the effect of high pressure homogenization (HPH) on the rheological properties of a fruit juice serum model. The serum model was chosen and designed to be similar to tomato serum, and showed Newtonian behaviour. Product viscosity decreased with increasing homogenization pressure, and could be modelled well using two different functions (exponential and sigmoidal). Based on the results obtained, the expected behaviour of the fruit juice was discussed. The results obtained are potentially useful for future studies on product and process development.

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