



Effect of high pressure homogenization (HPH) on the rheological properties of tomato juice: Time-dependent and steady-state shear

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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 the thermal processing of food. Although microbial inactivation has been widely studied, there are only a few works in the literature reporting the physicochemical changes caused in fruit products due to HPH, especially those regarding the rheological properties. The present work evaluated the effect of HPH (up to 150 MPa) on the time-dependent and steady-state shear rheological properties of tomato juice. HPH reduced the mean particle diameter and particle size distribution (PSD), and increased its consistency and thixotropy. The rheological results were in accordance with the PSD observed. The rheological properties of the juice were evaluated by the Herschel–Bulkley and Falguera–Ibarz models (steady-state shear) and Fignon–Shoemaker and Weltman models (time-dependent). The parameters of these equations were modelled as a function of the homogenization pressure. The models obtained described the experimental values well, and contributed to future studies on product and process development.

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1. Introduction

High pressure homogenization (HPH) technology consists of pressurizing a fluid to flow quickly through a narrow gap valve, which greatly increases its velocity, resulting in depressurization with consequent cavitation and high shear stress. Thus particles, cells and macromolecules suspended in the fluid are subjected to high mechanical stress, becoming twisted and deformed (Pinho et al., 2011; Flourey et al., 2004). Several studies have evaluated the use of HPH for microbial inactivation in fruit products. The use of HPH as a partial or total substitute for the thermal processing of foods has been studied for tomato (Corbo et al., 2010), apple (Donsì et al., 2009; Pathanibul et al., 2009; Saldo et al., 2009), mango (Tribst et al., 2009, 2011), açai (Aliberti, 2009), orange (Campos and Cristianini, 2007; Tahiri et al., 2006), carrot (Pathanibul et al., 2009; Patrignani et al., 2009, 2010), banana (Calligaris et al., 2012) and apricot (Patrignani et al., 2009, 2010) juices.

However, although microbial inactivation has been widely studied, there are only a few works in the literature regarding the physicochemical changes in fruit products due to high pressure homogenization (HPH) processing, especially regarding their rheological properties. The rheological characterization of food is

important for the design of unit operations, process optimization and high quality product assurance (Ibarz and Barbosa-Cánovas, 2003; Rao, 1999). The study of the influence of processing on the rheological properties of foods is thus essential for an efficient product and process design.

Tomato is one of the most popular and widely grown vegetables in the world (Nisha et al., *in press*). It is also one of the most important vegetables in the food industry, and widely included in the human diet. Homogenization is a commonly used unit operation in tomato processing, and it is well accepted that homogenization increases the apparent viscosity of tomato products (Bayod and Tornberg, 2011; Lopez-Sanchez et al., 2011a; Bayod et al., 2007; Ouden and Vliet, 2002, 1997; Beresovsky et al., 1995; Thakur et al., 1995; Mohr, 1987; Foda and Mccollum, 1970; Whittenberg and Nutting, 1958). However, no papers were found in the literature studying the effect of homogenization on the rheological parameters of tomato products (i.e., the steady-state shear and time-dependent rheological parameters), especially at high pressures (HPH). The present work evaluated the effect of high pressure homogenization (HPH) on the time-dependent and steady-state shear rheological properties of tomato juice.

2. Material and methods

A 4.5° Brix tomato juice was obtained by diluting a commercial 30° Brix pulp in distilled water. The commercial pulp was used to

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Nomenclature

α	slope index of the linear model for the evaluation of the experimental values versus those obtained by models (Eq. (8)) (-)	A	structural parameter in the Weltman model (Eq. (5)) (Pa)
β	intercept index of the linear model for the evaluation of the experimental values versus those obtained by models (Eq. (8)) (-)	A_{SF}	particle specific surface area (Eq. (3)) ($\text{m}^3 \cdot \text{g}^{-1}$)
$\dot{\gamma}$	shear rate (s^{-1})	B	kinetic parameter in the Weltman model (Eq. (5)) ($\text{Pa} \cdot \text{s}^{-1}$)
ϕ	particle volume fraction (-)	d	particle diameter (m)
ϕ_m	maximum packing fraction of solids (-)	$D[4,3]$	particle volume-based diameter (Eq. (1)) (m)
η	viscosity [Pa·s]	$D[3,2]$	particle area-based diameter (Eq. (2)) (m)
η_a	apparent viscosity ($= \sigma/\dot{\gamma}$) (Pa·s)	k	consistency coefficient, Herschel–Bulkley model (Eq. (6)) ($\text{Pa} \cdot \text{s}^n$)
η_r	relative viscosity (Eq. (9)) (Pa·s)	k_B	Boltzman constant ($= 1.38 \times 10^{-23} \text{ N m K}^{-1}$)
η_0	initial viscosity in the Falguera–Ibarz model (Eq. (7)) (Pa·s)	k_{FS}	kinetic parameter in the Figoni–Shoemaker model (Eq. (4)) (s^{-1})
η_∞	equilibrium viscosity in the Falguera–Ibarz model (Eq. (7)) (Pa·s)	k_{FI}	viscosity decline parameter in the Falguera–Ibarz model (Eq. (7)) (-)
$[\eta]$	intrinsic viscosity (Pa·s)	n	flow behavior index, Herschel–Bulkley model (Eq. (5)) (-)
ρ	particle density (kg m^{-3})	Pe	Peclet number (Eq. (12)) (-)
σ	shear stress (Pa)	P_H	homogenization pressure (MPa)
σ_0	yield stress, Herschel–Bulkley model (Eq. (6)) (Pa)	$\bar{r}_{\text{particle}}$	mean suspended particle radius (m)
σ_0	initial stress in the Figoni–Shoemaker model (Eq. (4)) (Pa)	V	particle volume (m^3)
σ_e	equilibrium stress in the Figoni–Shoemaker model (Eq. (4)) (Pa)	t	time (s)
		T	absolute temperature (K)

guarantee standardization and repeatability. It was obtained using the hot break process, concentrated by evaporation at 65 °C, thermally processed by the UHT method and aseptically packaged in bags.

The pulp was fractionated into small portions in the laboratory, packaged in high density polyethylene bottles and frozen at -18 °C until used. This procedure allowed for the use of the same product for the entire experiment. The samples were thawed at 4 °C, diluted using distilled water at 50 °C to ensure better hydration (Tehrani and Ghandi, 2007), and then allowed to rest for 24 h at 5 °C to ensure complete hydration and release the incorporated air. The juice pH was 4.6.

2.1. High pressure homogenization (HPH) process

The juice was homogenized at 0 MPa (control), 50, 100 and 150 MPa using a high pressure homogenizer (Panda Plus, GEA Niro Soavi, Italy). Samples were introduced at room temperature into the equipment by suction and quickly cooled using an ice bath just after the homogenization valve. The maximum temperature reached was ~40 °C (for the sample processed at 150 MPa just before the ice bath). The experiments were carried out with three replicates.

2.2. Particle size distribution (PSD)

Sample particle size distribution (PSD) was measured by light scattering (Malvern Mastersizer 2000 with Hydro 2000s, Malvern Instruments Ltd., UK), with three replicates, for the juices processed at 0, 50 and 150 MPa. In addition to the PSD, the volume-based mean diameter was evaluated ($D[4,3]$, Eq. (1)); where n_i is the number of particles with diameter d_i and the area-based mean diameter ($D[3,2]$, Eq. (2)). Both equivalent diameters were evaluated, since the $D[4,3]$ is greatly influenced by large particles, whereas the $D[3,2]$ is more influenced by the smaller ones

(Lopez-sanchez et al., 2011a; Bengtsson and Tornberg, 2011). The particle specific surface area (A_{SF}) was also obtained, using Eq. (3).

$$D[4,3] = \frac{\sum_i n_i d_i^4}{\sum_i n_i d_i^3} \quad (1)$$

$$D[3,2] = \frac{\sum_i n_i d_i^3}{\sum_i n_i d_i^2} \quad (2)$$

$$A_{SF} = \frac{6 \sum_i \frac{V_i}{d_i}}{\rho \sum_i V_i} = \frac{6}{\rho \cdot D[3,2]} \quad (3)$$

2.3. Rheological properties

Rheological analyses were carried out using a controlled stress (σ) rheometer (AR2000ex, TA Instruments, USA) with a cross hatched plate–plate geometry (40 mm of diameter). The gap dimension (1.0 mm) was determined using a gap-independency procedure as described by Tonon et al. (2009). In this procedure, the distance between the plates was varied and the sample flow behaviour evaluated. The ideal gap dimension was observed when the sample flow behaviour was independent of variations in the gap. The temperature was maintained constant at 25 °C using a Peltier system.

The rheological evaluation was carried out with new samples, which were first placed in the rheometer and maintained at rest for 10 min before shearing. After resting, the samples were sheared at a constant shear rate (300 s^{-1}) for 10 min, while the shear stress was measured. After the time-dependent shear period, a linear decreasing stepwise protocol ($300\text{--}0.1 \text{ s}^{-1}$) was used to guarantee steady-state shear conditions (~7 min).

The time-dependent rheological properties of the product were evaluated using the first part of the protocol. The shear stress decay

was evaluated by two models widely used to describe thixotropy in foods (Ibarz and Barbosa-Cánovas, 2003) and previously evaluated for tomato juice (Augusto et al., *in press*). The models evaluated were the Fighi and Shoemaker (1983), (Eq. (4)) and Weltman (1943), (Eq. (5)) models.

$$\sigma = \sigma_e + (\sigma_0 - \sigma_e) \cdot \exp(-k_{FS} \cdot t) \quad (4)$$

$$\sigma = A - B \cdot \ln t \quad (5)$$

The steady-state shear rheological properties of the product were evaluated using the second part of the protocol. The product flow behaviour was modelled using the Herschel–Bulkley model (Eq. (6)), which comprises the Newton, Bingham and Ostwald–de-Waele (power law) models, and has been widely used to describe the rheological properties of food products. Moreover, the flow behaviour of the tomato juice was also evaluated using another rheological model recently proposed by Falguera and Ibarz (2010). In the Falguera–Ibarz model (Eq. (7)), the decline in apparent viscosity ($\eta_a = \sigma/\dot{\gamma}$) with the shear rate is described by a power equation, from an initial value (η_0) to an equilibrium one (η_∞).

$$\sigma = \sigma_0 + k \cdot \dot{\gamma}^n \quad (6)$$

$$\eta_a = \eta_\infty + (\eta_0 - \eta_\infty) \cdot \dot{\gamma}^{(-k_H)} \quad (7)$$

Each parameter in Eqs. (4)–(7) was then modelled as a function of the homogenization pressure (P_H) and the rheological parameter (“RP”, i.e., σ for the Fighi–Shoemaker, Weltman and Herschel–Bulkley models, and η_a for the Falguera–Ibarz model) obtained by the models (RP_{model}) plotted as a function of the experimental values ($RP_{\text{experimental}}$). The regression of these data to a linear function (Eq. (8)) results in three parameters that can be used to evaluate the description of the experimental values by the models, i.e. the linear slope (α ; that must be as close as possible to the unity), the intercept (β ; that must be as close as possible to zero) and the coefficient of determination (R^2 ; that must be as close as possible to the unity).

$$RP_{\text{model}} = \alpha \cdot RP_{\text{experimental}} + \beta \quad (8)$$

The parameters for each model were obtained by linear or non-linear regression using the software CurveExpert Professional (v.1.2.0, <http://www.curveexpert.net/>, USA) with a significant probability level of 95%.

3. Results and discussion

3.1. Particle size distribution (PSD)

Fig. 1 shows the effect of HPH (0–150 MPa) on the tomato juice particle size distribution (PSD). As expected, the homogenization processing reduced the mean particle diameter, as previously observed for various tomato products (up to 9 MPa, Bayod and Tornberg, 2011; Bengtsson and Tornberg, 2011; Bayod et al., 2008; up to 60 MPa, Lopez-Sanchez et al., 2011a) and other vegetable products, such as passion fruit juice (up to 28 MPa, Okoth et al., 2000), citrus juices (up to 30 MPa, Betoret et al., 2009; Sentandreu et al., 2011; up to 170 MPa, Lacroix et al., 2005), apple juice (up to 200 MPa, Donsi et al., 2009), apple, broccoli, carrot and potato sauces (up to 9 MPa, Bengtsson and Tornberg, 2011; up to 60 MPa, Lopez-Sanchez et al., 2011a).

Moreover, as can be seen in Fig. 1, not only was the mean diameter affected by the HPH but also the particle size distribution (PSD). The control juice showed a monomodal distribution, with particle diameters ranging between ~ 100 and ~ 1000 μm . When the juice was processed at 50 MPa, a broader distribution was observed, with particles ranging between ~ 10 and ~ 1000 μm .

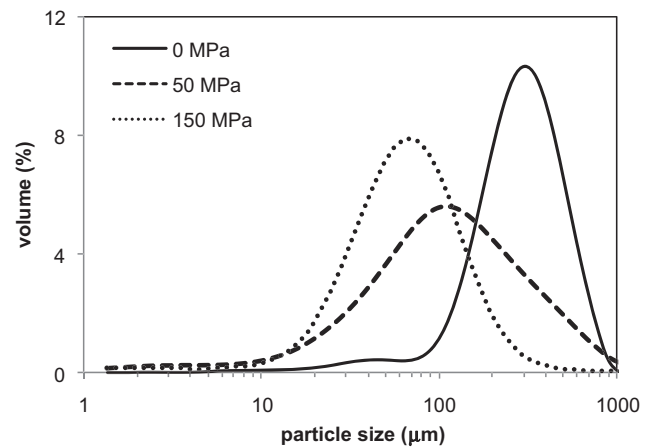


Fig. 1. Effect of HPH (0–150 MPa) on the tomato juice particle size distribution (PSD). Mean of three replicates.

Finally, when the juice processed at 150 MPa was evaluated, a further reduction in particle diameter and narrower distribution (~ 10 to ~ 300 μm) were seen. Moreover, the changes in particle diameter between 50 and 150 MPa were less pronounced than those between 0 MPa and 50 MPa. Similar behaviour was observed by Silva et al. (2010) for pineapple pulp homogenized at pressures up to 70 MPa.

Thus the effect of homogenization pressure (P_H) on the disruption of suspended particles seems to follow an asymptotic behaviour, i.e., increases in P_H have reduced effects at higher P_H values. In fact this can be observed even in the D[4,3] and D[3,2] values (Fig. 2a) and in the following evaluation of the rheological behaviour.

Fig. 2a shows the reduction in the volume-based mean diameter (D[4,3], Eq. (1)) and in the area-based mean diameter (D[3,2], Eq. (2)) due to the homogenization pressure. Although the equivalent diameters were both reduced during HPH, the reduction in D[3,2] between the samples treated at 0 and 50 MPa (79%) was higher than in D[4,3] (45%). Since the D[4,3] is greatly influenced by large particles and the D[3,2] more influenced by smaller ones (Lopez-sanchez et al., 2011a; Bengtsson and Tornberg, 2011), this result indicated a considerable increase in the number of small particles when the juice was processed at 50 MPa. Moreover, the reduction in D[3,2] between 50 and 150 MPa (20%) was smaller than in D[4,3] (53%), which indicates that the following disruptions occurred preferentially in the larger suspended particles, in accordance with the PSD values shown in Fig. 1.

Becker et al. (1972) studied the tomato cell dimensions, and found values between 400×600 μm and 600×1000 μm . Thus it is to be expected that the control juice, with particle diameters ranging between ~ 100 and ~ 1000 μm , would be constituted of some whole cells and their fragments, obtained during tomato pulp processing. The homogenization process disrupts the remaining cells and breaks the fragments up into small suspended particles, and it is to be expected that the small fragments would be less susceptible breakage during processing than the larger ones or the whole cells, which explains the effect observed for P_H on the disruption behaviour of suspended particles.

3.2. Rheological properties

Fruit juices are composed of an insoluble phase (the pulp) dispersed in a viscous solution (the serum). The dispersed phase or pulp is formed of fruit tissue cells and their fragments, cell walls

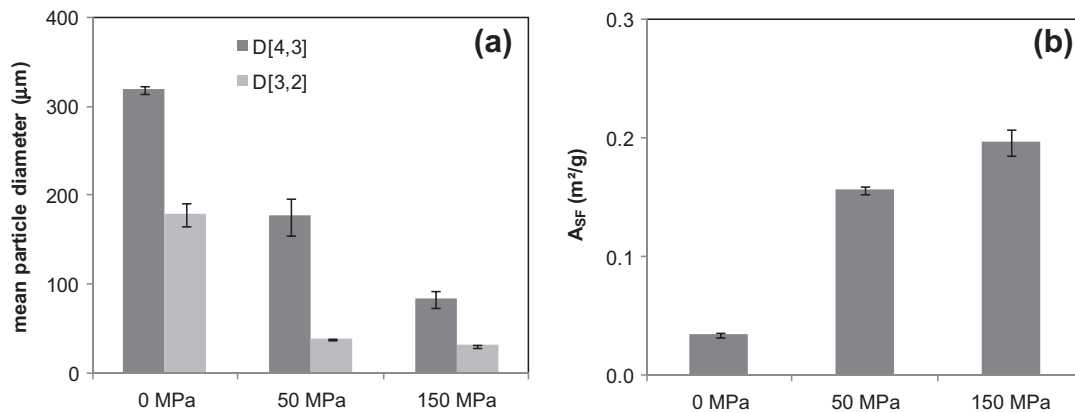


Fig. 2. Effect of HPH (0–150 MPa) on tomato juice particles mean diameter (D[4,3] and D[3,2]; a) and specific surface area (A_{sf} , b). Mean of three replicates; vertical bars represent the standard deviation in each value.

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 inside each phase and between them.

The tomato juice serum is a Newtonian fluid, which becomes a Herschel–Bulkley fluid due to the dispersion of pulp particles (Augusto et al., 2012b; Augusto et al., *inpress*; Tanglertpaibul and Rao, 1987). The tomato juice particle diameters ranged between 10 and 1000 μm in the present work (0–150 MPa). In this range, the product is classified as a noncolloidal dispersion, where hydrodynamic forces govern its rheological properties and Brownian motion is negligible (Fischer et al., 2009; Genovese et al., 2007; Tsai and Zammouri, 1988).

The relative viscosity (η_r , Eq. (9)) of dilute solid particles dispersed in a liquid medium is described by the Einstein Equation (Eq. (10); Genovese et al., 2007; Loveday et al., 2007; Guyot et al., 2002; Metzner, 1985). Moreover, one of the most used equations derived for concentrated dispersions is the Krieger–Dougherty equation (Eq. (11); Fischer et al., 2009; Genovese et al., 2007; Loveday et al., 2007; Guyot et al., 2002).

$$\eta_r = \frac{\eta_{\text{dispersion}}}{\eta_{\text{continuous_phase}}} \quad (9)$$

$$\eta_r = 1 + [\eta] \cdot \phi \quad (10)$$

$$\eta_r = \left(1 - \frac{\phi}{\phi_m}\right)^{-[\eta] \cdot \phi_m} \quad (11)$$

According to these equations, the viscosity of the dispersion is affected by the continuous phase viscosity (the juice serum), the particle intrinsic viscosity (which depends on particle shape) and the relative volume occupied by the particles (expressed by its volume fraction – ϕ – and the maximum packing fraction of solids – ϕ_m). Although more complex and in most cases more concentrated than the Einstein proposed fluid, a qualitative evaluation can be carried out based on Eqs. (10) and (11).

The HPH reduced the diameters of the tomato juice suspended particles, increasing its volume fraction (ϕ). Thus the observed behaviour suggests an increase in apparent viscosity of the tomato juice when processed by HPH. However, the final juice behaviour will also be a function of the serum viscosity and particle intrinsic viscosity.

Augusto et al. (2012b) studied the effect of HPH on the viscosity of a tomato juice serum model, describing a viscosity reduction due to HPH (~5% at 50 MPa and ~15% at 150 MPa). Thus, HPH reduces the viscosity of the tomato juice continuous phase (i.e.,

the serum) ($\eta_{\text{continuous_phase}}$), which would reduce the product apparent viscosity ($\eta_{\text{dispersion}}$).

Moreover, it is to be expected that the HPH would also change the particle shapes, polydispersity, volume fraction (ϕ), maximum packing fraction of solids (ϕ_m) and intrinsic viscosity ($[\eta]$).

However, the final product rheology cannot be simply described by the hydrodynamic forces. The reduction in the diameters of the tomato juice suspended particles can improve interparticle interaction, since the particle surface area is greatly increased (Fig. 2b). The interaction of small particles can be due to van der Waals forces (Genovese et al., 2007; Tsai and Zammouri, 1988) and/or electrostatic forces due to the interaction between the negatively charged pectins and the positively charged proteins (Berevovsky et al., 1995; Takada and Nelson, 1983). Cell disruption and further fragmentation not only increase the surface area of the suspended particles, but also change the properties of the particles and serum. Cell fragmentation exposes and releases wall constituents, such as pectins and proteins, improving the particle–particle and particle–serum interactions. Thus, the non-hydrodynamic forces can be important in systems with smaller suspended particles, such as the HPH tomato juice.

Thus as observed by Guyot et al. (2002), it is still not possible to predict the rheological behaviour of a dispersed system just from its PSD. The final tomato juice behaviour in relation to the HPH will be a function of the changes in both particles and serum.

3.3. Steady-state shear rheological properties

Fig. 3 shows the flow curves ($\sigma \times \dot{\gamma}$) of the tomato juice processed by HPH (0–150 MPa). Fig. 4 shows the corresponding behaviour of the apparent viscosity in relation to the shear rate ($\eta_a \times \dot{\gamma}$).

As expected (Augusto et al., *inpress*), the tomato juice flow behaviour was well described by the Herschel–Bulkley model (Eq. (6); $R^2 > 0.99$), while the apparent viscosity behaviour in relation to the shear rate was well described by the Falguera–Ibarz model (Eq. (7); $R^2 > 0.99$). Moreover, the parameters obtained for both models were in accordance with those previously described for other fruit products (Tables 1 and 2).

Figs. 3 and 4 show that HPH improves tomato juice consistency, i.e., increases the shear stress (σ) and apparent viscosity (η_a) related to each shear rate $\dot{\gamma}$. Moreover, it can be seen that the main changes take place between 0 and 50 MPa, being smaller between 50 and 100 MPa and tending to an asymptotic behaviour between 100 and 150 MPa. The changes are due to the reduction in the

suspended particles, and are in accordance with the values observed for the PSD.

Figs. 5 and 6 show the Herschel–Bulkley (Eq. (6)) and Falguera–Ibarz (Eq. (7)) model parameters as a function of the homogeniza-

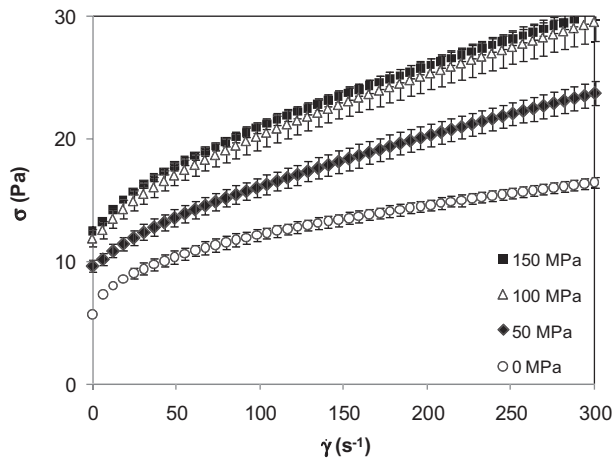


Fig. 3. Flow curves ($\sigma \times \dot{\gamma}$) of tomato juice processed by HPH (0–150 MPa). Mean of three replicates at 25 °C; vertical bars represent the standard deviation for each value.

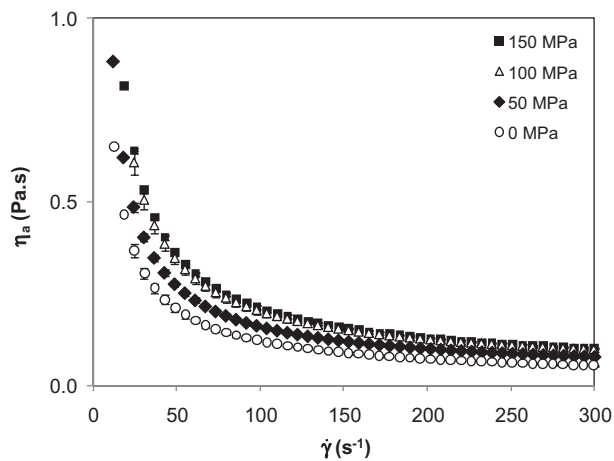


Fig. 4. Apparent viscosity ($\eta_a \times \dot{\gamma}$) of tomato juice processed by HPH (0–150 MPa). Mean of three replicates at 25 °C; vertical bars represent the standard deviation for each value.

Table 1
Values for the parameters of Herschel–Bulkley model for fruit products.

Product	T (°C)	σ_0 (Pa)	k (Pa·s ⁿ)	n	Refs.
Tomato juice (0 MPa)	25	5.38	0.92	0.44	Present work
Tomato juice	20	0.94	0.19	0.56	Augusto et al. (in press)
Concentrated tamarind juice (71° Brix)	30	1.46	4.32	0.59	Ahmed et al. (2007)
Jabuticaba pulp	25	1.55	0.48	0.6	Sato and Cunha (2009)
Concentrated orange juice	25	2.2	3.13	0.64	Falguera and Ibarz (2010)
Peach juice (10% fiber)	20	3.26	13.1	0.46	Augusto et al. (2011)
Açai pulp	25	4.35	0.17	0.78	Tonon et al. (2009)
Butia pulp	20	32.54	0.15	0.86	Haminiuk et al. (2006)
Peach puree (12–21° Brix)	25	1.11–25.3	2.46–11.3	0.32–0.34	Massa et al. (2010)
Umbu pulp (10–25° Brix)	20	3.18–8.06	14.00–37.74	0.29–0.31	Pereira et al. (2007, 2008)
Apple, apricot and banana based baby food	20	3.38–7.41	4.76–13.7	0.18–0.38	Ahmed and Ramaswamy (2007)
Mango pulp	20	3.81–6.24	8.82–11.33	0.31–0.34	Ahmed et al. (2005)
<i>S. purpurea</i> L. pulp	20	13.26	15.22	0.3	Augusto et al. (2012a)

tion pressure (P_H). The increase in homogenization pressure (P_H) decreased the consistency index in the Herschel–Bulkley model (k), and increased the other Herschel–Bulkley (σ_0 , n) and Falguera–Ibarz model parameters (η_0 , η_∞ , k_{FI}), as discussed below. Moreover, the Herschel–Bulkley yield stress (σ_0) and the Falguera–Ibarz initial viscosity (η_0) increased systematically due to the P_H .

Bengtsson and Tornberg (2011), Lopez-Sanchez et al. (2011a), Bayod et al. (2008) and Ouden and Vliet (1997) observed that the yield stress (σ_0) of tomato products increased due to homogenization ($P_H < 60$ MPa). However, the authors did not model it as a function of the homogenization pressure (P_H), or study the other steady-state shear rheological parameters (i.e., the consistency index – k , and the flow behaviour index – n) or the time-dependent rheological properties.

Shijvens et al. (1998) observed that a reduction in the suspended particle diameter increased the yield stress (σ_0) and apparent viscosity (η_a) of apple sauce. Moreover, Tsai and Zammouri (1988) showed that a decrease in particle size resulted in increases in both the flow behaviour index (n) and apparent viscosity (η_a) in shear thinning fluids. Thus the rheological and PSD results described here are in accordance with those reported by Shijvens et al. (1998) and Tsai and Zammouri (1988).

Servais et al. (2002) described the effect of PSD on the Herschel–Bulkley model parameters. According to these authors, the yield stress (σ_0) mostly depends on the amount of small particles (i.e., the specific surface area) and on the interactions between them (due to mechanical friction or chemical interactions). Moreover, the flow behaviour index (n) depends on the distribution of small and large particles and the rheology of the suspending fluid; while the consistency index (k) depends on the maximum packing fraction (ϕ_m) and the distribution of small and large particles.

Silva et al. (2010) studied the HPH of pineapple pulp up to 70 MPa. The product consistency index (k) and the flow behaviour index (n) showed the same behaviour as those reported here. Similar behaviour was shown by Floury et al. (2002) studying the HPH of methylcellulose up to 350 MPa. However, Silva et al. (2010) observed that the apparent viscosity of pineapple pulp decreased with increase in homogenization pressure, as observed by Lopez-Sanchez et al. (2011a) for carrot and broccoli homogenized at 60 MPa.

In fact, Lopez-Sanchez et al. (2011b) showed that each vegetable cell wall had a different behaviour when processed by HPH. While carrot tissue requires higher shears to be disrupted, the cell walls of tomato cells were broken even at moderate shear values. The increases and decreases in the viscosities of homogenized tomato, carrot and broccoli were directly related to their volume fraction (ϕ), highlighting the importance of hydrodynamic forces in product rheology.

Table 2
Values for the parameters of Falguera–Ibarz model for fruit products.

Product	T (°C)	η_{∞} (Pa·s)	η_0 (Pa·s)	k_{FI}	Refs.
Tomato juice (0 MPa)	25	0.016	5.68	0.866	Present work
Tomato juice	20	0.008	1.11	0.807	Augusto et al. (in press)
Concentrated orange juice	25	0.214	6.08	0.584	Falguera and Ibarz (2010)

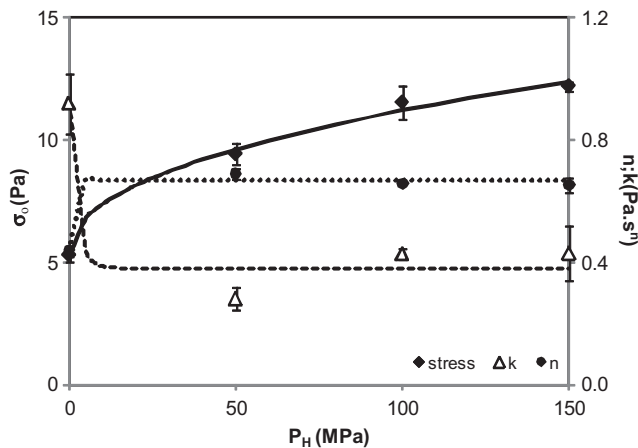


Fig. 5. Parameters of the Herschel–Bulkley model as a function of homogenization pressure (P_H). Vertical bars are the standard deviation for each mark, and the curves are empirical regressions of Table 3.

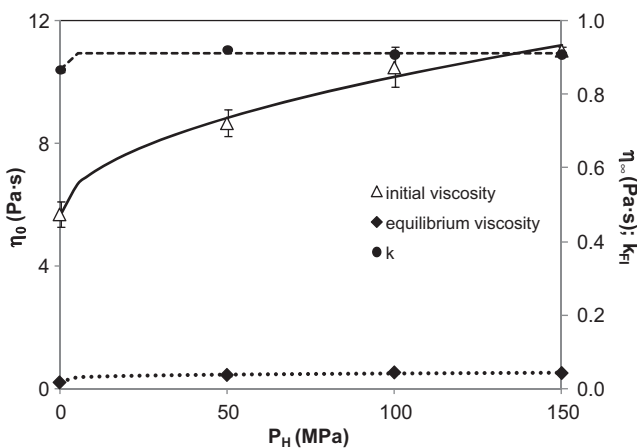


Fig. 6. Parameters of the Falguera–Ibarz model as a function of homogenization pressure (P_H). Vertical bars are the standard deviation for each mark, and the curves are empirical regressions of Table 3.

This suggests that the effect of HPH processing is different for each fruit product, and highlight the need for better understanding of this process.

The Herschel–Bulkley (Eq. (6)) and Falguera–Ibarz (Eq. (7)) model parameters were then modelled as a function of the homogenization pressure (P_H ; Figs. 5 and 6, Table 3). Due to the behaviour observed (Figs. 5 and 6), a power-type function with an initial value (related to original properties of the tomato juice, i.e., those processed at 0 MPa) was used to model the parameters, with the exception of the consistency index in the Herschel–Bulkley model (k), due to its reduction with increase in P_H .

The shear stress and apparent viscosity obtained using the models (Table 3) were compared with the experimental values. Using Eq. (8), it is observed that the models obtained described the experimental values well (Table 4).

The Peclet Number is related to the particle transport due to shearing (non-Brownian systems) and diffusion (Brownian systems) (Fischer et al., 2009; Rao, 1999). Therefore, as the particle size is reduced, the Pe decreases and the system approximates to the Brownian domain.

At small Pe values Brownian motion dominates, while at higher Pe values, structure distortions due to shear flow are more pronounced, and Brownian motion cannot restore the structure of the suspension to its equilibrium state; therefore shear thinning and shear thickening will occur (Fischer et al., 2009).

Yoo and Rao (1994) and Tsai and Zammouri (1988) described a decreasing linear relationship between the logarithm of the relative viscosity ($\ln(\eta_r)$) and the logarithm of the Peclet number ($\ln(Pe)$) for shear thinning fluids. The Peclet number (Pe) is the product of the Reynolds number (Re) and the Schmidt number (Sc), being described by Eq. (12) (Fischer et al., 2009; Yoo and Rao, 1994; Tsai and Zammouri, 1988).

$$Pe = \frac{\eta_{\text{continuous phase}} \cdot \bar{r}_{\text{particle}}^3 \cdot \dot{\gamma}}{k_B \cdot T} \quad (12)$$

If the reduction in tomato juice serum model viscosity described by Augusto et al. (2012b) is considered ($\sim 15\%$ at 150 MPa), as well as the observed reduction in $D[4,3]$ ($\sim 85\%$ at 150 MPa), it can be seen that HPH reduces the Peclet number of the product (Pe , considering the $\bar{r}_{\text{particle}}$ based on the $D[4,3]$ values), as presented in Fig. 7. As described by Yoo and Rao (1994) and Tsai and Zammouri (1988), a reduction in Pe increases the relative viscosity (η_r) of the product, and consequently its apparent viscosity (η_a). The linear correlation between the Pe and the relative viscosity (η_r) can also be used as an approach to estimate the η_r as a function of Pe and P_H .

Once again, the results obtained for PSD described the effect of HPH on the rheological properties of the tomato juice well.

3.4. Time-dependent rheological properties

The time-dependent rheological behaviour is related to the structural change due to shear (Ramos and Ibarz 1998), i.e., destruction of the internal structure during flow (Cepeda et al. 1999). Consequently, the time-dependent rheological characterization is important to understand the product changes during processing (Augusto et al., in press). In the original product, the internal structure formed by the insoluble pulp dispersed in the serum showed greater resistance to deformation, resulting in higher shear stress. When shearing is carried out, this structure is broken, which can be noticed by the decline in stress.

Fig. 8 shows the decline in shear stress of the samples when sheared at 300 s^{-1} for 10 min. As expected (Augusto et al., in press), the tomato juice showed thixotropic behaviour, and even the Fignon–Shoemaker (Eq. (4)) and Weltman (Eq. (5)) models could describe its time-dependent rheological behaviour well ($R^2 > 0.92$). Moreover, the parameters obtained for both models are in agreement with those previously described by Augusto et al. (in press) for a refined commercial tomato juice (Table 5) and for other fruit products.

In concentrated orange juice the k_{FS} value of the Fignon–Shoemaker model varied from 0.073 to 0.600 s^{-1} ($7.2 \leq \dot{\gamma} (\text{s}^{-1}) \leq 57.6$;

Table 3

Parameters of Figoni–Shoemaker, Weltman, Herschel–Bulkley and Falguera–Ibarz models as function of homogenization pressure (P_H in MPa). Tomato juice, 25 °C, 0 MPa $\leq P_H \leq 150$ MPa.

Model		Equation	R^2
Figoni–Shoemaker ($\dot{\gamma} = 300 \text{ s}^{-1}$)	σ_0 (Pa)	$\sigma_0 = 19.58 + 2.298P_H^{0.6230}$	0.98
	σ_e (Pa)	$\sigma_e = 16.31 + 1.101P_H^{0.5460}$	0.99
	k_{FS} (s^{-1})	$k_{FS} = 0.048 + 0.0027P_H^{0.2495}$	0.99
Weltman ($\dot{\gamma} = 300 \text{ s}^{-1}$)	A (Pa)	$A = 22.52 + 4.305P_H^{0.5839}$	0.99
	B ($\text{Pa}\cdot\text{s}^{-1}$)	$B = 0.9381 + 0.5405P_H^{0.5962}$	0.99
Herschel–Bulkley	σ_0 (Pa)	$\sigma_0 = 5.320 + 0.7254P_H^{0.04538}$	0.99
	k ($\text{Pa}\cdot\text{s}^n$)	$k = 0.380 + (0.920 - 0.380) \cdot e^{-0.4636P_H}$	0.94
	n	$n = 0.4402 + 0.2263 \cdot P_H^{4.83 \cdot 10^{-9}}$	0.98
Falguera–Ibarz	η_0 ($\text{Pa}\cdot\text{s}$)	$\eta_0 = 5.668 + 0.4347P_H^{0.5078}$	0.99
	η_∞ ($\text{Pa}\cdot\text{s}$)	$\eta_\infty = 0.0160 + 0.0093 \cdot P_H^{0.2063}$	0.99
	k_{FI}	$k_{FI} = 0.8664 + 0.0465 \cdot P_H^{1.12 \cdot 10^{-8}}$	0.94

Table 4

Figoni–Shoemaker, Weltman, Herschel–Bulkley and Falguera–Ibarz models parameters as function of homogenization pressure (P_H , Table 3): accuracy evaluation by using Eq. (8).

Models	α	β	R^2
Figoni–Shoemaker	0.98	0.65	0.98
Weltman	0.97	1.05	0.97
Herschel–Bulkley	0.94	1.25	0.96
Falguera–Ibarz	0.99	0.00	0.99

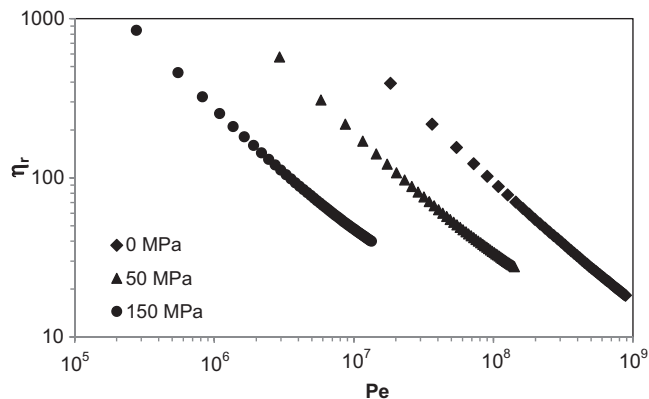


Fig. 7. Effect of HPH (0–150 MPa) on the tomato juice Peclet number (Pe) and relative viscosity (η_r). Mean of three replicates at 25 °C.

Ramos and Ibarz, 1998), while in gilaboru juice at 43° Brix, it varied from 0.0027 to 0.0031 s^{-1} ($50 \leq \dot{\gamma} (\text{s}^{-1}) \leq 150$; Altan et al., 2005). Under the same conditions, the value of B (Weltman model) varied from 0.89 to 1.17 Pa s^{-1} . Abu-Jdayil et al. (2004) used the Weltman model to model the time dependent rheological behaviour of tomato paste (5.7% solids). The value of B varied from 10^{-14} to 0.0187 Pa s^{-1} in the shear rate range of 2.2–79 s^{-1} .

Fig. 8 shows that HPH improves tomato juice thixotropy, since the difference between the initial and equilibrium stress in relation to the homogenization pressure (P_H) was bigger, as well as the time taken to stabilize (~ 200 – 300 s for 0 MPa and ~ 500 – 600 s for 100–150 MPa). Moreover, as observed in the results for the steady-state shear properties and PSD, the main changes take place between 0 and 50 MPa, being smaller between 50 and 100 MPa and tending to stabilize between 100 and 150 MPa.

Bayod and Tornberg (2011) studied the effect of homogenization (9 MPa) on the properties of the suspended particles in a

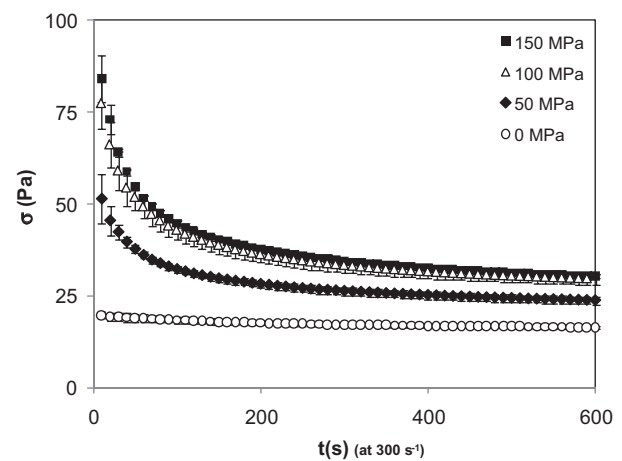


Fig. 8. HPH (0–150 MPa) tomato juice stress decline during shearing at 300 s^{-1} for 10 min. Mean of three replicates at 25 °C; vertical bars represent the standard deviation for each value.

tomato suspension containing salt, sugar and acetic acid. Using micrographs, the authors observed that the structure of the suspension formed a network due to processing by homogenization, which could be disrupted by shearing.

The PSD analysis showed that HPH reduced the diameter of the suspended particles in the tomato juice, increasing the particle surface area (Fig. 2b) and the interaction forces between them. Thus, it is to be expected that HPH processing would result in small particles aggregating to form a network, as described by Bayod and Tornberg (2011), resulting in a more thixotropic fluid.

However, as described by Genovese et al. (2007) and Tsai and Zammouri (1988), the van der Waals and electrostatic forces only dictate interparticle interactions between small particles at low shear rates ($\dot{\gamma}$). At higher shear rates ($\dot{\gamma}$), the hydrodynamic forces dictate the rheological properties of the fluid.

This explains the results obtained for yield stress (σ_0) and consistency index (k) in the Herschel–Bulkley model and the thixotropic behaviour. When the tomato juice is at rest or submitted to low shear rates ($\dot{\gamma}$), the inter-particle interactions result in a network structure. It characterizes the increasing in the yield stress values (σ_0) and thixotropy of the product due to HPH. Since the particle structure is broken by shear, interparticle interaction is low and hence the consistency index (k) is also low.

The Figoni–Shoemaker (Eq. (4)) and Weltman (Eq. (5)) parameters were then modelled as a function of the homogenization

Table 5

Values for the parameters of Fignon–Shoemaker and Weltman models for fruit products ($\dot{\gamma} = 300\text{s}^{-1}$).

Model	Tomato juice (0 MPa) (25 °C, present work)	Commercial tomato juice (20 °C, Augusto et al., in press)
Fignon–Shoemaker	σ_0 (Pa)	19.82
	σ_e (Pa)	16.37
	k_{FS} (s^{-1})	0.0048
Weltman	A (Pa)	22.81
	B ($\text{Pa}\cdot\text{s}^{-1}$)	0.976

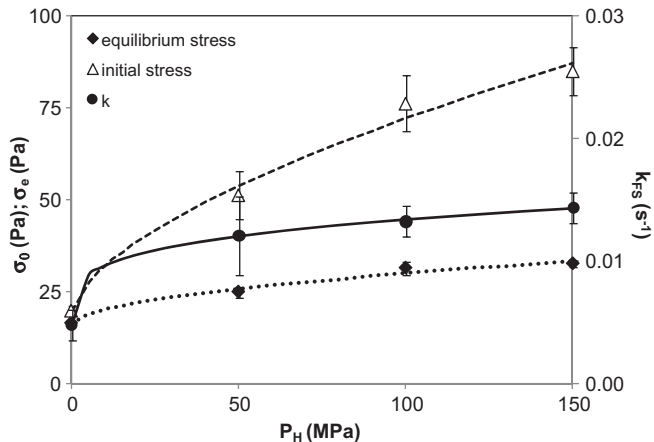


Fig. 9. Parameters of the Fignon and Shoemaker model as a function of homogenization pressure (P_H). Vertical bars are the standard deviation for each mark, and the curves are empirical regressions of Table 3.

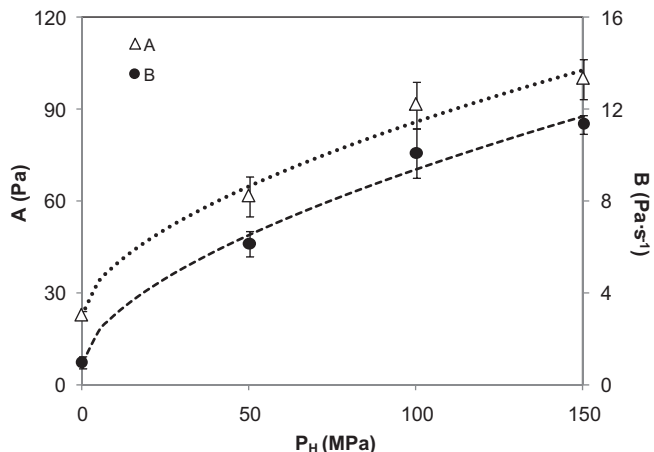


Fig. 10. Parameters of the Weltman model as a function of homogenization pressure (P_H). Vertical bars are the standard deviation for each mark, and the curves are empirical regressions of Table 3.

pressure (P_H ; Figs. 9 and 10, Table 3). Due to the behaviour observed, a power-type function with an initial value (related to the original properties of the tomato juice, i.e., those processed at 0 MPa) was used. The values obtained for shear stress using the models were compared with the experimental values. Using Eq. (8), it is observed that the models obtained described the experimental values well (Table 4).

3.5. General discussion and importance

High pressure homogenization (HPH) is a non-thermal technique widely studied to obtain safe, stable products such as fruit juices. Its importance is growing due to increasing scientific research associated with food processing and even industrial applications. However, there are still only a few papers in the literature regarding the physicochemical changes in fruit products due to this technique, especially regarding their rheological properties.

In the present work, the effect of HPH (up to 150 MPa) on the time-dependent and steady-state shear rheological properties of tomato juice was studied. HPH reduced the suspended particle diameters and the particle size distribution (PSD), increasing product consistency and thixotropy.

Moreover, the results obtained indicated that HPH could be used to increase tomato juice consistency, improving its sensory acceptance, reducing the need for the addition of hydrocolloids, and reducing particle sedimentation and serum separation.

Thus, the effect of homogenization pressure (P_H) on the tomato juice rheological parameters was modelled. Using the models obtained (Table 3), the tomato juice flow properties could be predicted well as a function of homogenization pressure (P_H), shear rate ($\dot{\gamma}$) and time of shearing (t). This could be useful in the design of equipment and processes for food products.

The main effect was observed at low homogenization pressures ($P_H \sim 50$ MPa), followed by asymptotic behaviour. This indicates that simpler and less expensive equipment could be used to obtain the desirable effects of HPH in tomato juice.

The results obtained highlighted the possible applications of high pressure homogenization (HPH) as a valuable tool to promote changes in the physical properties of food products.

4. Conclusions

The present work evaluated the effect of high pressure homogenization (HPH) on the time-dependent and steady-state shear rheological properties of tomato juice. The tomato juice particle size distribution (PSD) and rheological properties were evaluated at homogenization pressures (P_H) of up to 150 MPa. HPH reduced the mean particle diameter and the particle size distribution (PSD), increasing the tomato juice consistency. Moreover, HPH increased tomato juice thixotropy, in agreement with the observed PSD. The steady-state shear rheological properties of the juice were evaluated by the Herschel–Bulkley and Falguera–Ibarz models, whose parameters were modelled as a function of the homogenization pressure (P_H). The tomato juice time-dependent rheological properties were evaluated by the Fignon–Shoemaker and Weltman models, whose parameters were then modelled as a function of the P_H . The models obtained described the experimental values well.

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