

Multi-pass high pressure homogenization (MP-HPH) of tomato juice: effect on the rheological properties

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<u>Abstract</u>

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Introduction

The high pressure homogenization (HPH) is a non-thermal technology which was initially studied for microbial inactivation in food products. In recent years, this technology has successfully been used for several other objectives, such as improving the rheological properties of fruit juices (Augusto *et al.*, 2012; Augusto *et al.*, 2013); reducing pulp sedimentation during storage (Silva *et al.*, 2010; Kubo *et al.*, 2013); stabilising juice cloudiness (Sentandreu *et al.*, 2011); increasing the activity of enzymes applied in food processing (Tribst *et al.*, 2013); and improving the properties of polysaccharides (Wang *et al.*, 2012; Wang *et al.*, 2012) and proteins (Dong *et al.*, 2011).

For both enzyme activity changes (Liu *et al.*, 2009a; Liu *et al.*, 2009b; Welti-Chanes *et al.*, 2009; Calligaris *et al.*, 2012; Tribst *et al.*, 2013) and microbial inactivation (Donsi *et al.*, 2009; Patrignani *et al.*, 2009; Maresca *et al.*, 2011), it has been shown that the multi-pass HPH processing (MP-HPH) at lower homogenization pressures (PH) was able to produce the same changes of one-pass processing at higher PH. It is important to observe that the lower the homogenization pressure, the lower the processing

The high pressure homogenization (HPH) is a promising technology that has been recently proposed as unit operation capable of improving food properties, in special rheological behaviour. It has been shown that the multi-pass HPH (MP-HPH) at lower homogenization pressures can produce the same changes on enzyme activity and microbial inactivation than processing with one-pass at higher PH, but with smaller processing costs. The present work evaluated the effect of MP-HPH on the rheological properties and particle size distribution (PSD) of tomato juice. The HPH disrupted the suspended particles and increased the juice consistency, whose rheological behaviour was described by the Herschel–Bulkley model. The asymptotic effect of the homogenization pressure was confirmed. However, the number of homogenization passes has not affected the juice rheological properties. The positive effect of the HPH process for increasing the tomato juice consistency should only be achieved by increasing the homogenization pressure.

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costs (equipment and operation). Therefore, the use of multi-passes could be of interest, aiming to optimize the HPH process (i.e., maximizing its effect with lower costs).

The HPH has been proposed to be used as a valuable tool to promote desirable changes in the physical properties of food products, in special the rheological properties of tomato juice (Augusto *et al.*, 2012). The present work evaluated the effect of the number of passes (NH) and homogenization pressures (PH), on the steady-state shear rheological properties of tomato juice (flow properties).

Material and Methods

High Pressure Homogenization (HPH) Process

A 4.5°Brix tomato juice was processed with a maximum homogenization pressure (PH) of 100 MPa, as the major rheological changes take place at this range (Augusto *et al.*, 2012; Kubo *et al.*, 2013).

The juice was homogenized at 0 MPa (control), 25 MPa, 50 MPa and 100 MPa (gauge, homogenization pressures – PH) 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 $\sim 27^{\circ}$ C (for the sample processed at 100 MPa just before the ice bath).

After passing through the valve and cooling to the same initial temperature, the samples were reintroduced in the homogenizer, in order to evaluate the effect of multi-pass processing. Thus, the 50 MPa samples were processed at 1 and 2 passes, while the 100 MPa were processed at 1, 2 and 3 passes. The experiments were carried out with three replicates.

Evaluation

Sample particle size distribution (PSD) was measured by light scattering (Malvern Mastersizer 2000 with Hydro 2000s, Malvern Instruments Ltd., UK), with three replicates. 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) and 1.0 mm of gap dimension (Augusto *et al.*, 2012). The temperature was maintained constant at 25°C using a Peltier system. Three replicates were evaluated.

The rheological evaluation was carried out with new samples, which were first placed in the rheometer and maintained at rest for 5 min before shearing. After resting, the samples were sheared at a constant shear rate (300 s⁻¹) for 10 min, in order to avoid thixotropy (Augusto *et al.*, 2012). Then, a linear decreasing stepwise protocol (300 s⁻¹ to 0.1 s⁻¹) was used to guarantee steady-state shear conditions.

The flow behaviour was modelled using the Herschel-Bulkley model (Equation 1), 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 (where σ is the shear stress, $\mathring{\gamma}$ is the shear rate, $\sigma 0$ is the yield stress, *k* is the consistency index and *n* is the flow behaviour index). The model parameters were obtained by non-linear regression using the software CurveExpert Professional (v.1.2.3, *http://www.curveexpert.net/*, *USA*) with a significant probability level of 95%.

$$\sigma = \sigma_0 + k \cdot \dot{\gamma}^n \qquad \text{(Equation 1)}$$

The effect of homogenization pressure (P_H) and number of passes (N_H) were evaluated for each Herschel-Bulkley parameter (σo , k and n). For then, the relative value of each parameter were considered, i.e., the value at each condition divided by the initial value (0 MPa). Then, the analysis of variance (ANOVA) and the Tukey test at a 95% confidence level were conducted using the Statistica

5.5 (StatSoft, Inc., USA) software.

Results and Discussion

As expected, tomato juice rheological behaviour could be described by the Herschel-Bulkley model (Equation 1; $R^2 > 0.99$), showing a shear thinning behaviour with yield stress. The control sample properties were $\sigma 0 = 4.61\pm0.39$ Pa, k = 0.82 ± 0.03 Pa•sn and n = 0.44 ± 0.005 (at 25°C), values very close to those reported by Augusto *et al.* (2012).

Figure 1 shows the effect of HPH on the tomato juice particle size distribution (PSD; to allow better visualization of the curves only the processes at 0, 50 and 100 MPa are shown), as well as the effect of HPH (0-100 MPa; 1-3 cycles) on the tomato juice rheological properties. Each rheological parameter is shown in relation to its initial value, i.e., by analysing the relative values of yield stress ($\sigma 0/\sigma 0$ 0MPa), consistency index (k/k0MPa) and flow behaviour index (n/n0MPa).

As expected, the increase in homogenization pressure (PH) decreased the consistency index in the Herschel-Bulkley model (k), and increased both the yield stress (σ_0) and the flow behaviour index (n) parameters, with an asymptotic behaviour. As discussed by Augusto et al. (2012) the main effects of HPH are related to changes on the juice suspended particles (pulp), which is formed by fruit tissue cells and their fragments, cell walls and insoluble polymer clusters and chains. When the product is homogenized, those particles are broken, which increases its surface area and the interaction forces between them. As described by Tsai and Zammouri (1988) and Genovese et al. (2007), the van der Waals and electrostatic forces only dictate interparticle interactions between small particles at low shear rates (\dot{r}), while the hydrodynamic forces dictate the rheological properties at higher shear rates $(\not\sim)$. Therefore, the smaller particles are aggregated, forming a network, increasing the yield stress ($\sigma 0$) value. On the other hand, since the particles structures are broken by shear, interparticle interaction is low and hence the consistency index (k) is also low. Finally, the smaller particles are easier aligned to the flow field, decreasing the juice shear thinning behaviour (i.e., approximating the flow behaviour index to the unit: $n \rightarrow 1$).

Thus, it can be seen that the main changes take place between 0 MPa and 25 MPa, being smaller and tending to an asymptotic behaviour between 50 MPa and 100 MPa. The relative yield stress ($\sigma 0/\sigma 0$ 0MPa) increased ~300% of its initial value between 0 and 100 MPa, being the changes up to 25 MPa ~230%.



Figure 1. Effect of high pressure homogenization on the rheological and microstructural properties of tomato juice. Parameters of Herschel–Bulkley ($\sigma 0/\sigma 0$ 0MPa; k/k0MPa; n/n0MPa) model and particle size distribution (PSD) as a function of homogenization pressure (PH) and number of passes (ϕ : N_H = 1; \blacksquare : N_H = 2; \blacktriangle : N_H = 3). Vertical bars are the standard deviation for each condition.

The relative consistency index (k/k0MPa) decreased to ~50% of its initial value between 0 and 100 MPa, while the changes up to 25 MPa was close to 70% of its initial value. The relative flow behaviour index (n/n0MPa) showed small variation, increasing to ~150% of its initial value between 0 and 100 MPa, with changes of ~130% up to 25 MPa.

On the other hand, it can be seen that the number of homogenization cycles (N_H, 1-3) have not affected the product rheological properties, without statistical differences among cycles for each homogenization pressure (P>0.05). Those values of $\sigma 0/\sigma 0$ 0MPa, k/k0MPa and n/n0MPa were close for 1, 2 and 3 consecutive processes (Figure 1).

It has been shown that the multi-pass HPH processing can change the enzyme activity (Liu *et al.*, 2009a; Liu *et al.*, 2009b; Welti-Chanes *et al.*, 2009; Calligaris *et al.*, 2012; Tribst *et al.*, 2013) and microbial inactivation (Donsi *et al.*, 2009; Patrignani *et al.*, 2009; Maresca *et al.*, 2011). However, the present work demonstrated that the multi-pass processing was not able to promote any additional changes on the tomato juice rheological properties, even so a small change on its PSD are shown (Figure 1).

When the food product is processed by the HPH, it is pressurized to quickly flow through a

narrow gap valve, whose dimension is in the order of just some micrometres. As a result, its velocity is greatly increased, resulting in depressurization with consequent cavitation and high shear stress. The flow regime during the fluid passage through the gap is laminar (Re \sim 300-500), being transitional to turbulent after that (Floury *et al.*, 2002; Pinho *et al.*, 2011; Lee *et al.*, 2013). Consequently, the velocity profile inside the gap is parabolic, with a stress profile across the gap width.

Therefore, for small particles or molecules (i.e., dimension << gap width), each subsequently passage through the homogenizer can promote a new change, as it can be placed in a new position of specific velocity and stress. On the other hand, for particles whose dimension is close to the gap width, any subsequent cycle can only submit it to the same shear stress, delivering the same amount of energy. In this case, a small effect of multi-pass processing is expected.

Further, as the stress distribution is more uniform in small particles, it is expected that the small fragments would be less susceptible to break during processing than the larger ones or the whole cells. Moelants *et al.* (2013) described different disruption behaviour in relation to small and large particles of carrot, observing that the cell breakage occurred during mechanical breakup rather than cell separation along the middle lamella. For the evaluated tomato juice, however, the initial juice showed the presence of whole cells but not of tomato tissues, reinforcing the initial proposition (Kubo *et al.*, 2013).

Therefore, the observed important effect of homogenization pressure (0-100 MPa), but negligible effect of multi-pass processing (1-3) on the tomato juice rheology, is explained. Tribst et al. (2013) studied the effect of multi-pass HPH on the activity of amyloglucosidase, glucose oxidase and a neutral protease. The authors observed that each enzyme showed a different behaviour in relation to the multi-pass processing. For amyloglucosidase and neutral protease, the main effects of homogenization were observed after only one pass, indicating that the energy delivered to the molecule under this condition was sufficient to cause the maximum molecular changes. On the other hand, the glucose oxidase activity was increased up to three cycles, which could be attributed to the additional molecular change caused by each homogenization pass.

Harte and Venegas (2010) evaluated the effect of HPH on the rheological behaviour of alginate, κ -carrageenan and xanthan gum dispersions. Only a slight decrease on the suspension's consistency was observed due to the number of passages through the homogenizer (up to 6 passages at 300 MPa). Further, the effect of homogenization pressure was much higher than the number of passages.

Lopez-Sanchez et al. (2011) evaluated the effect of homogenization on carrot and tomato emulsions, considering the storage (G') and loss (G") modules at the oscillatory frequency of 1 Hz, as well as the yield stress (σ_0). The homogenization process was conducted at 10 MPa (1-10 cycles) and 100 MPa. The authors observed that the flow behaviours of all processed tomato samples were similar, with no statistical difference on the yield stress when processed up to 10 cycles at 10 MPa. On the other hand, the carrot emulsions showed ~68% of difference on the yield stress when the samples of one and 10 cycles at 10 MPa were compared. In fact, the authors also showed that each vegetable cell wall had a different behaviour when processed by HPH.

Patrignani *et al.* (2009) and Patrignani *et al.* (2010) evaluated the carrot and apricot juices homogenized up to 8 cycles at 100 MPa. The product apparent viscosity was obtained using a falling ball viscometer. For both juices, no differences were observed among the processed samples.

Bengtsson and Tornberg (2011) and Bayod and Tornberg (2011) observed small changes on tomato suspension and concentrate viscoelastic behaviour after being homogenizes up to three cycles. However, the results were obtained for a very small homogenization pressure (9 MPa), and only considering its viscoelastic behaviour.

In the present work, the tomato juice flow properties were evaluated, as it better describes the product behaviour during processing. As the viscoelastic behaviour importance is more related to conditions of low shear rates (\nearrow), the small differences on the particle disruption during multipass processing could be sufficient to promote the changes observed by Bengtsson and Tornberg (2011) and Bayod and Tornberg (2011).

The positive effect of HPH on the tomato juice properties was recently demonstrated, increasing the juice consistency, thixotropy, viscous and elastic behaviour, being possible to improve its sensory acceptance, reducing the need for adding hydrocolloids and reducing particle sedimentation and serum separation (Augusto et al., 2012; Kubo et al., 2013; Augusto et al., 2013). The initial purpose of the present works was to evaluate the possibility of using multi-pass HPH processes, as smaller homogenization pressures (P_H) in consecutives cycles could result in smaller processing costs (equipment and operation). Nevertheless, it must be highlighted that the multi-pass HPH is not a practical processing, being interesting just if a small number of cycles are effective. However, it was shown that the number of HPH passes $(N_{H}; 1-3)$ have not affected the steadystate shear rheological properties of tomato juice. Consequently, the positive effect of this technology for the evaluated product should only be achieved by increasing the homogenization pressure (P_{μ}) .

Conclusions

The effect of multi-pass high pressure homogenization (MP-HPH) on the steady-state shear rheological properties of tomato juice was evaluated. The increase in the juice consistency, with an asymptotic effect of the PH was confirmed. However, the number of homogenization passes (up to three passes at 100 MPa) have not affected the juice rheological properties. Consequently, the positive effect of this technology for the evaluated product should only be achieved by increasing the homogenization pressure (P_H).

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