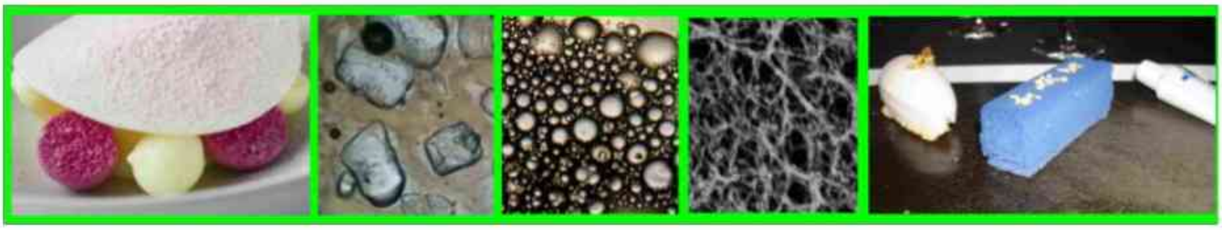

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Research Note

Modeling the structure-texture-stability relationship in apricot juices treated by high pressure homogenization

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Abstract

This study investigates how a mechanical treatment can texturize and physically stabilize juices without additives. Apricot juices with varying amount of pulp were formulated and treated by high pressure homogenization (HPH) at different pressures (0.1, 35 and 70 MPa). Physicochemical, structural, rheological properties and physical stability of the juices were assessed. HPH treatment resulted in a significant reduction in particle size and shape alterations, leading to increased viscosity (from 2 to 8-fold) and enhanced physical stability (sedimentation halted or slowed down for up to 7 days). These physical modifications were attributed to the formation of a weak interconnected particle network in treated juices that resisted sedimentation over time. These findings suggest that mechanical treatment alone can functionalize juices and that it may be applicable using domestic processes such as grinding, blending, or soup mixing.

Keywords

plant cells, diluted suspensions, weak gel, particle network, rheology, sedimentation

1. Introduction

The epidemiological evidence that links a diet rich in fruits and vegetables with reduced chronic disease (such as cardiovascular disease, stroke, type 2 diabetes or some types of cancer) risk has made fruit and vegetable derivatives like juices high-demand products (Southon, 2000; Joffe and Robertson, 2001; Benton and Young, 2019). A juice is a polydisperse system, and it is defined as a heterogeneous suspension of insoluble and deformable pulp particles (that is fruit cells, cell clusters and cell wall fragments) in a continuous serum solution that is rich in soluble compounds like pectins, saccharides, acids and minerals (Moelants *et al.*, 2014). Fruit cell wall assemblies and fragments vary in size between a few hundred micrometers and a few millimeters (Rao, 2010), and their size has been shown to be one of the major determinant of suspension stability (Reiter *et al.,* 2003 ; Augusto *et al*., 2012). Being additive-free remains the first criteria that consumers use to define “natural” foods (Rozin, 2005; Rozin *et al*., 2012), and the increasing interest in convenient yet “natural” beverages that preserve their fresh-like characteristics and nutritional value has paved the way for novel alternative processes. The presence of a sediment of phase separation in a juice is viewed as a product defect (Beveridge, 2002), and reducing pulp sedimentation through mechanical treatment could improve consumer acceptability and eliminate the need for adding hydrocolloids in juice matrices, which also has advantageous logistical and economic implications (Rojas *et al*., 2016).

Among the new non-thermal techniques investigated in the juice industry, high pressure homogenization (HPH) has recently been gaining traction as a promising unit operation that could modulate the structure and physicochemical properties of fruit-based dispersions and expand their techno-functional properties (Augusto *et al*., 2012; Zhu *et al*., 2019). In a high pressure homogenizer, the fluid is pumped into a narrow gap valve (a few tens of micrometers) *via* high pressure intensifiers, which considerably increase its velocity resulting in high shear stress (several tens or hundreds of s^{-1} depending on the fluid viscosity and on the pressure applied) followed by depressurization. The suspended cells and fragments then twist, deform and are disrupted as a consequence of turbulence and shear (Tan and Kerr, 2015; Martínez-Monteagudo *et al*., 2017).

HPH provides a higher shear input as compared to blending or grinding and maximizes cell disruption (Lopez-Sanchez *et al*., 2011), dissociating cell clusters into single cells or cell fragments (Moelants *et al*., 2013). Several juice matrices have been studied in relation to HPH over the last two decades, like for example mango (Zhou *et al*., 2017), peach (Wang *et al*., 2019), orange (Betoret *et al*., 2009; Leite *et al*., 2017), tomato (Augusto *et al*., 2012; Augusto *et al.,* 2013; Kubo *et al*., 2013), strawberry (Moscovici Joubran *et al*., 2019), apple (Leite *et al*., 2015), parsnip (Castro *et al*., 2012) and other mixed juices (Wellala *et al*., 2020). However, conclusions related to the effect of HPH on the physicochemical and rheological properties of fruit and vegetable juices are often contradictory because outcomes are pressure and product dependent (Moscovici Joubran *et al*., 2019). For example, while carrot tissue requires a more intense shear to be broken down (100 MPa), tomato cells can be disrupted at lower shear values (10 MPa) (Lopez-Sanchez *et al.,* 2011; Moelants *et al*., 2014).

The effect of concentration of insoluble matter on the behavior of many fruit-based preparations like purees and juices has been widely studied (Day *et al*., 2010; Lopez-Sanchez et al., 2011; Espinosa-Muñoz *et al*., 2012; Leverrier *et al*., 2016) and put forward as a first order parameter that controls the flow and viscoelastic properties of plant tissue-based suspensions (Ricci *et al*., 2020), and thus its physical stability against sedimentation.

The properties of the dispersed elements such as size, shape, rigidity, and surface properties, as well as the properties of the continuous phase will act as second order parameters. Mechanical treatment holds a defining role on the structure and the properties of pulp particles and so on the rheological behaviors of these suspensions (Pickardt et al., 2004; Day *et al*., 2010; Lopez-Sanchez *et al*., 2011). By modifying the number, size and morphology of fruit particles in suspension, HPH changes the interactions between them, which in turn defines the rheological and functional properties of the juices (Ouden and Vliet, 1997; Lopez-Sanchez *et al*., 2011). HPH thus allows the generation of a wide range of textures in aqueous systems, using plant cells as structuring elements to texturize foods in a clean-label way.

Apricot is a commercially significant crop, and France produces 18% of the European Union’s supply, making apricot juice of great industrial interest (Moreau-Rio, 2006). As the effect of HPH on the rheological properties and physical stability of apricot juice has not yet been studied, the aim of the present study was to validate the role of pressure, puree quantity and mechanically-induced particle-specific properties in structuring, texturizing and stabilizing apricot puree-based model juice systems, in a more global attempt to pinpoint levers that steer the properties of apricot juices (textural properties and stability over time). Even though HPH can provide very intense shear input as compared to blending or grinding and maximizes cell disruption, the pressures applied were chosen to make this work transposable to domestic tools that allow mechanical processing and therefore the modification of the particle size distribution of the juices processed, such as a hand blender or a kitchen blender.

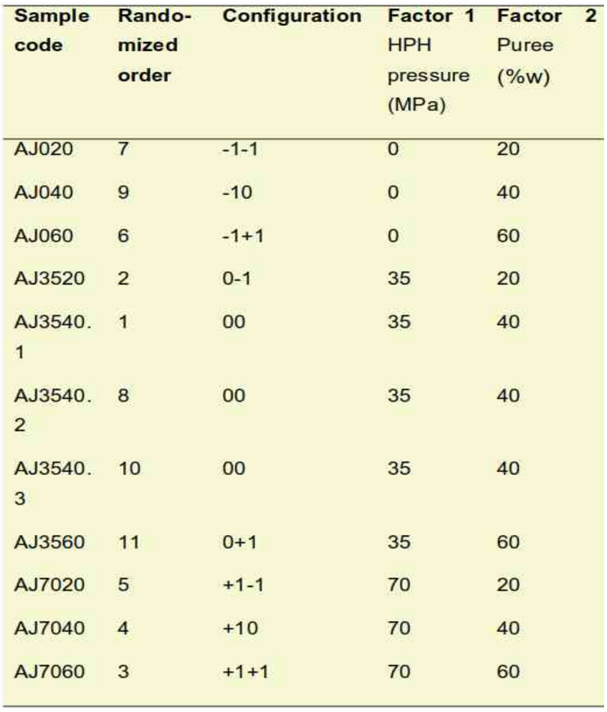
The research approach used in this work allows a quantification of the product-process interactions and the generation of models that predict the macroscopic behavior of food matrices. Rheological and stability indicators were modeled for apricot juice samples varying in puree content and subjected to different pressure treatments *via* a two-factor three-level (3²) experimental design.

2. Materials and methods

2.1. Preparation of apricot juice models

Juices with a concentration of 11.5 ± 0.2 °Brix were obtained by diluting 20%, 40%, or 60% (w/w) of a 20 ± 1 °Brix apricot puree (Les Vergers Boiron, France) in distilled water and adding sucrose under gentle magnetic stirring (Table 1). The addition of sucrose allowed for the establishment of an initial pre-treatment °Brix in the range of 11.5 ± 0.2° for all juices. This step was taken to standardize the quantity of soluble materials in the system and adhere to the requirements of the EU Directive 2001/112/EC for apricot juices. Subsequently, the juices underwent immediate high-pressure homogenization (HPH) treatments.

Table 1. Pressure treatment, puree content and naming of the samples.



Sample code	Rando-mized order	Configuration	Factor 1 HPH pressure (MPa)	Factor 2 Puree (%w)
AJ020	7	-1-1	0	20
AJ040	9	-10	0	40
AJ060	6	-1+1	0	60
AJ3520	2	0-1	35	20
AJ3540.1	1	00	35	40
AJ3540.2	8	00	35	40
AJ3540.3	10	00	35	40
AJ3560	11	0+1	35	60
AJ7020	5	+1-1	70	20
AJ7040	4	+10	70	40
AJ7060	3	+1+1	70	60

2.2. High pressure homogenization

The process was carried out at three pressures: 0.1 MPa (atmospheric pressure, meaning that no pressure was applied in the equipment during HPH treatment), 35 and 70 MPa in a bench-top high pressure homogenizer (Panda Plus, GEA, Italy). Preliminary tests carried out at 70 MPa revealed significant alterations in particle size and shape. Subsequently, a lower pressure level (35 MPa) was chosen for comparative analysis.

Similar pressure levels have been used for other fruits and vegetables (Leite *et al.,* 2015; Yu *et al*., 2016; Yi *et al.,* 2018; Wellala *et al.*, 2020). For enhanced readability and to emphasize that the sample treated at 0.1 MPa did not undergo pressurization during the high-pressure homogenization treatment, the samples will be denoted as "0", 35, and 70 MPa in the subsequent discussion. The aliquots of apricot juice (600 mL) were introduced into the equipment at 20 °C for one cycle, then covered and cooled at 4 °C until analyses were carried out. The highest recorded temperature post-treatment was 31 °C at 70 MPa, and the thermal effect was considered negligible.

2.3. Physicochemical and structural characterizations

Soluble solids and pH: The soluble solids of the juices pre and post-treatment were obtained using a pocket refractometer (Atago, PAL-1, Japan) at 20 °C. The refractometer prism was washed with distilled water prior to each measurement (Bot *et al.*, 2017). The pH of the juices was measured using a benchtop pH meter (pHenomenal, Avantar VWR, United States) at 20 °C under magnetic stirring. The pH meter was previously calibrated with three buffers (pH 4, 7 and 10). All measurements were made in triplicate.

Pulp content: The amount of pulp was obtained by centrifuging 30 g of juice at 5000 g for 2 hours at 20 °C (centrifuge Sigma 3K 12, Bioblock Scientific). All measurements were made in triplicate according to Espinosa-Muñoz *et al.* (2012). The serum was collected for dry matter and rheological analyses.

Dry matter: 3 g of juice or serum were weighed on an aluminum cup and dried in an oven at 105 °C for 8 hours until a constant weight was reached (Espinosa-Muñoz *et al.,* 2012).

Particle size distribution: The particle size distribution of the samples was acquired by laser diffraction (Mastersizer 2000 with Hydro 2000s, Malvern Instruments, UK). Drops of juice were slowly introduced into the sample dispersion unit which is filled with distilled water until obscurity was around 12. Refractive indices of 1.46 and 1.33 were fixed for the fruit cells and the dispersion phase (water) respectively. The absorption was set at 0.1 and the measurements were made in triplicate.

Light microscopy: 2 µL of well-stirred juice was placed on a glass slide and covered with a cover slip. Microstructure observations were made with a 10X objective lens using an Olympus BX51 microscope (Japan) in phase contrast connected to a digital camera.

Rheological characterization: Measurements were performed with a stress controlled rheometer (MCR301, Anton Paar, Austria) equipped with large gap coaxial cylinders (CC18.92, inner radius: 9.46 mm; outer radius: 14.46 mm; gap: 5 mm, gap length: 40 mm) at 20 °C (controlled with a Peltier system). Dynamic followed by steady-state measurements were carried out following an adapted procedure of Leverrier *et al.* (2016). The protocols were adapted for diluted products (no sedimentation within the highest total measurement time including resting time ~12 min). A resting time of 5 minutes was allowed before every measurement in order to control the loaded sample history. The rheological tests were carried out once at day 0, 1 and 7. The juices were brought from 4 °C back to room temperature prior to analysis, and directly poured into the measuring cylinder. Serum viscosity was measured using double gap cylinders (DG26.7).

Dynamic measurements: Strain sweep. A strain ranging from 0.01 to 150% was applied at constant angular frequency (10 rad.s^{-1}), measuring 22 points. Average values for the elastic (*G'*) and loss (*G"*) moduli were taken in the viscoelastic region (LVR). The cross-over value of *G'* and *G"* beyond the LVR was taken as the yield stress value.

Steady-state measurements: Juice flow curve: a logarithmic shear rate ramp was applied from 1 to 250 s^{-1} for a total duration of 6.5 min. The apparent viscosity was taken at 9.82 s^{-1} for all samples since for highly diluted ones, a Newtonian plateau was observed at low shear rates (9.82 s^{-1} being the highest accessible shear rate within the Newtonian plateau). The data were fitted to the Ostwald–de-Waele model allowing to obtain the consistency (*k*) and flow behavior (*n*) indices.

Serum viscosity: a logarithmic ramp from 1 to 500 s^{-1} was applied for a total duration of 5 min. The average apparent viscosity was taken in the Newtonian plateau between 10.6 and 58.7 s^{-1}.

Stability against sedimentation with time: Pulp sedimentation was evaluated via image analysis (ImageJ software, NIH), by comparing non-diluted and 1:8 diluted samples stored in glass tubes at 4 °C at day 0 and day 7. The sedimentation index was calculated as follows:

$$SI = \frac{Sediment\ height\ at\ day\ 7}{Initial\ height\ at\ day\ 0} \qquad [1].$$

Experimental design and statistical analysis: In order to evaluate the effect of homogenization pressure (factor 1) and puree content (factor 2) on the behavior of the juices, a 3² full factorial design was set-up. A total of 11 samples were produced, including the center point which was repeated an additional two times (Table 1). Significance was determined at 95% confidence level (*p*-value < 0.05) and the postulated quadratic model was the following:

$y_i = a\, x_{12} + b\, x_{22} + c\, x_1 + d\, x_2 + e\, x_1 x_2 + f.$ [2].

Analyses were done using JMP 14.1 (SAS, USA) and XLSTAT (Addinsoft, France) software. The analysis of variance (ANOVA) was carried out with a significant probability level of 95% and significant differences between means were determined by Tukey's test.

3. Results and discussion

3.1 The effect of HPH on physicochemical properties

The pulp content, which is proportional to the percentage (% w) of puree incorporated into the system, more than doubled after HPH for all samples. No significant difference was observed between 35 and 70 MPa (Table 2). For example, AJ040 pulp content increased by a factor of 2.6 at 35 MPa, and by 2.5 at 70 MPa.

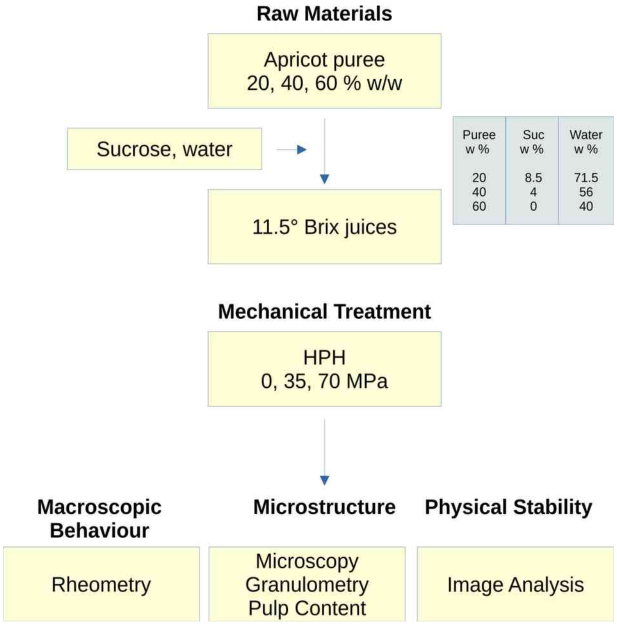
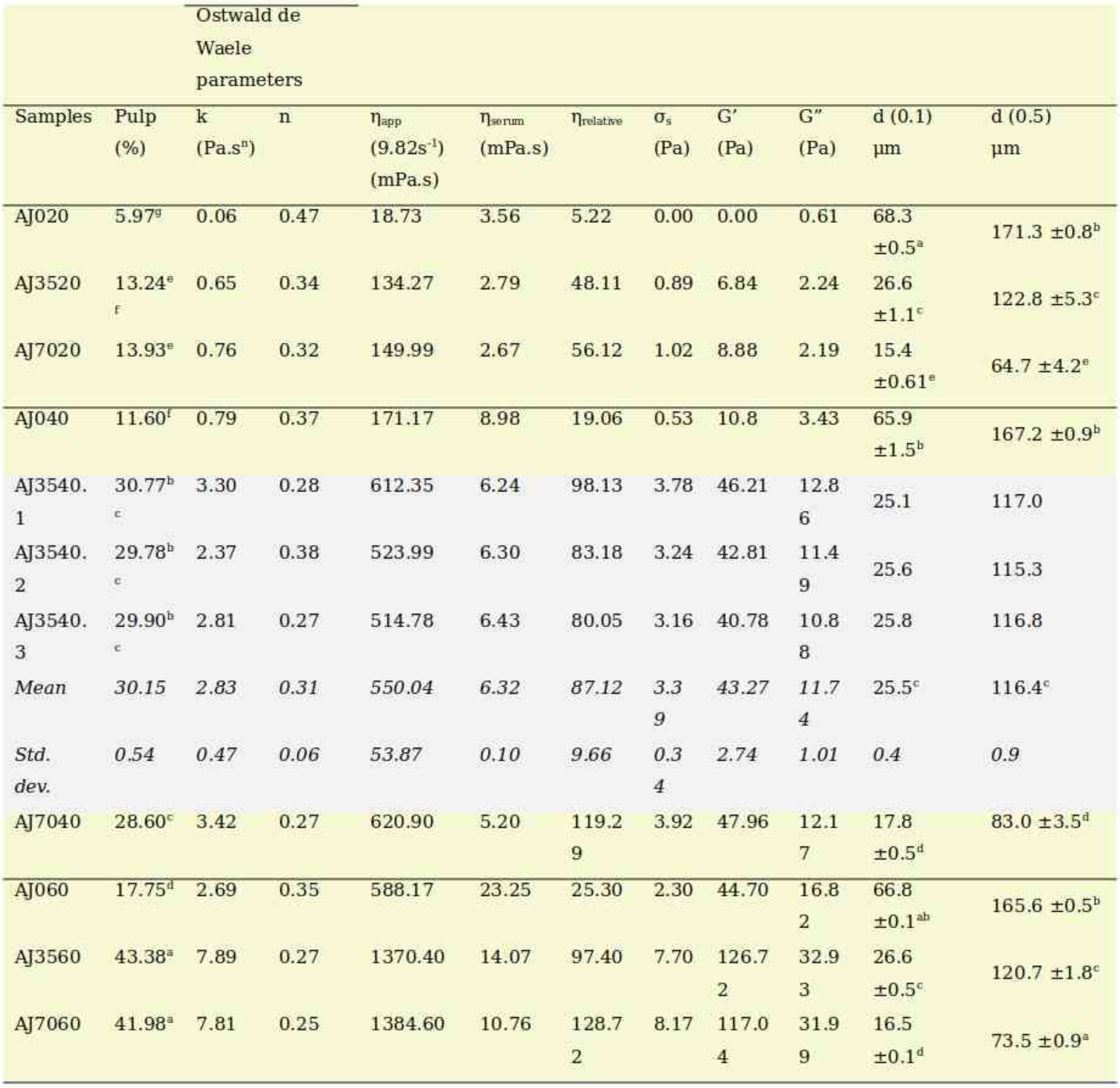


Figure 1. Juice formulation, processing parameters and analyses.

The pulp content typically varies with the shape, size and amount of particles in the sample (Espinosa-Muñoz *et al.*, 2013), and the increase in the pulp content with pressure across all samples confirms the effect of pressure on particle morphology and hence the packing or volume occupied by the particles post-centrifugation (Leverrier *et al.*, 2016). Pulp content is then a key measured variable that validates the effect of HPH on plant cells first-hand.

HPH did not significantly modify the °Brix of the juices (data not shown), which suggests that treatment within the range of pressures employed did not lead to an additional solubilization of materials (that is insoluble pectin) in the matrix. This is also confirmed by the dry matter measurements, with an average of 11.3 ± 0.1% post-treatment. The dry matter content is composed of cell wall particles, soluble pectin, sugars and minerals (Moelants *et al.*, 2014) as well as

Tableau 2. Pulp content, rheological and particle size properties measured at Day 0. The %pulp standard deviations for all samples are below 0.01. Different letters within each column represent significant differences.



		Ostwald de Waele parameters									
Samples	Pulp (%)	k (Pa.s^{n})	n	η_{app} (9.82s^{-1}) (mPa.s)	η_{serum} (mPa.s)	$\eta_{relative}$	σ_s (Pa)	G' (Pa)	G" (Pa)	d (0.1) µm	d (0.5) µm
AJ020	5.97^{g}	0.06	0.47	18.73	3.56	5.22	0.00	0.00	0.61	68.3 ±0.5^{a}	171.3 ±0.8^{b}
AJ3520	13.24^{ef}	0.65	0.34	134.27	2.79	48.11	0.89	6.84	2.24	26.6 ±1.1^{c}	122.8 ±5.3^{c}
AJ7020	13.93^{e}	0.76	0.32	149.99	2.67	56.12	1.02	8.88	2.19	15.4 ±0.61^{e}	64.7 ±4.2^{e}
AJ040	11.60^{f}	0.79	0.37	171.17	8.98	19.06	0.53	10.8	3.43	65.9 ±1.5^{b}	167.2 ±0.9^{b}
AJ3540.1	30.77^{bc}	3.30	0.28	612.35	6.24	98.13	3.78	46.21	12.86	25.1	117.0
AJ3540.2	29.78^{bc}	2.37	0.38	523.99	6.30	83.18	3.24	42.81	11.49	25.6	115.3
AJ3540.3	29.90^{bc}	2.81	0.27	514.78	6.43	80.05	3.16	40.78	10.88	25.8	116.8
Mean	*30.15*	*2.83*	*0.31*	*550.04*	*6.32*	*87.12*	*3.39*	*43.27*	*11.74*	25.5^{c}	116.4^{c}
Std. dev.	*0.54*	*0.47*	*0.06*	*53.87*	*0.10*	*9.66*	*0.34*	*2.74*	*1.01*	*0.4*	*0.9*
AJ7040	28.60^{c}	3.42	0.27	620.90	5.20	119.29	3.92	47.96	12.17	17.8 ±0.5^{d}	83.0 ±3.5^{d}
AJ060	17.75^{d}	2.69	0.35	588.17	23.25	25.30	2.30	44.70	16.82	66.8 ±0.1^{ab}	165.6 ±0.5^{b}
AJ3560	43.38^{a}	7.89	0.27	1370.40	14.07	97.40	7.70	126.72	32.93	26.6 ±0.5^{c}	120.7 ±1.8^{c}
AJ7060	41.98^{a}	7.81	0.25	1384.60	10.76	128.72	8.17	117.04	31.99	16.5 ±0.1^{d}	73.5 ±0.9^{a}

40% w puree juices (Figure 1). The pH of the juices averaged 3.5 ± 0.1 and was not affected by the treatment (Lopez-Sanchez, Nijsse *et al.*, 2011).

3.2. The effect of HPH on particle size distribution and microstructure

Figure 2 shows the effect of homogenization

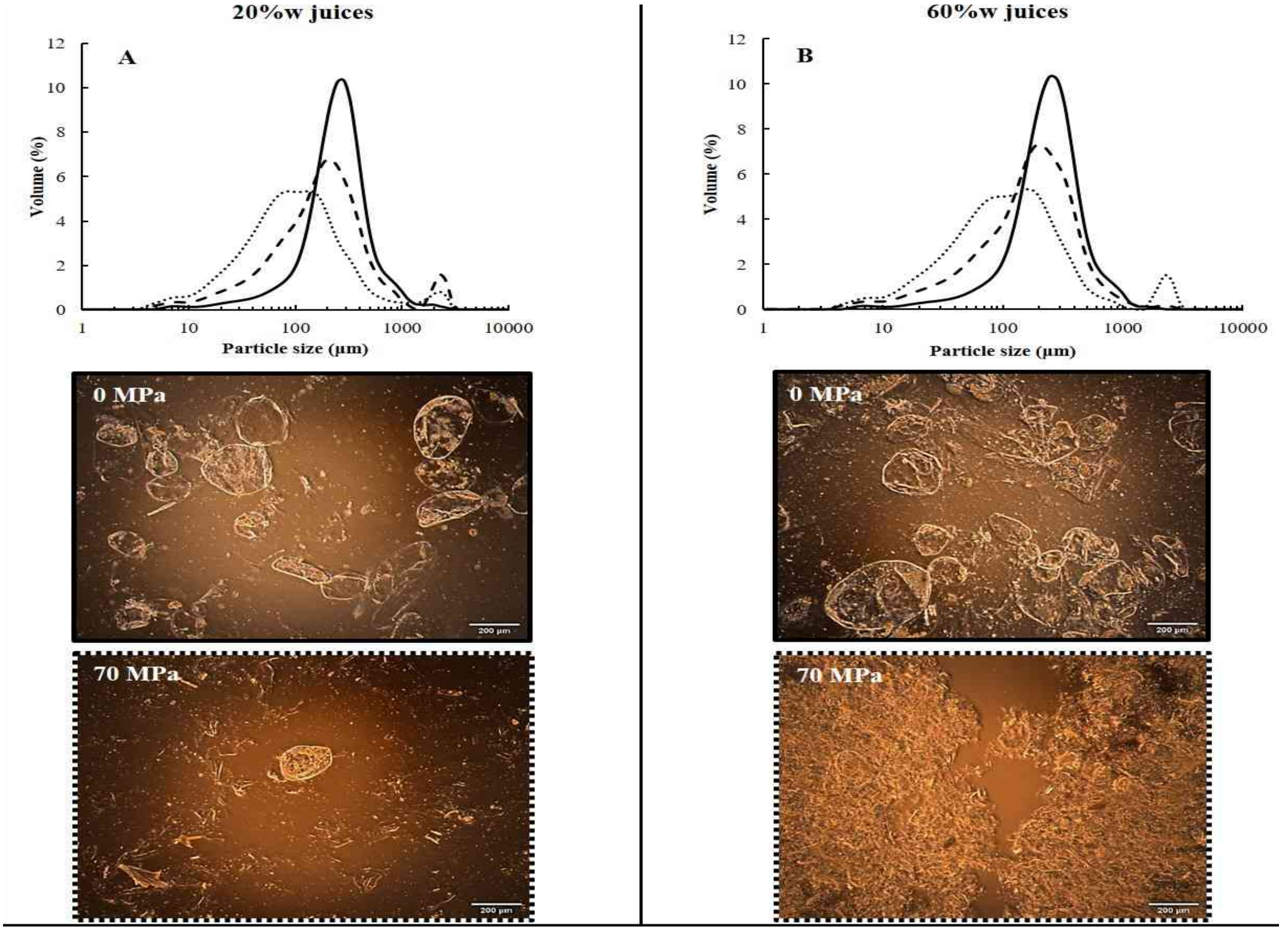


Figure 2. The effect of HPH pressure ("0" MPa continuous line; 35 MPa dashed line; and 70 MPa dotted line) on the particle size distributions and microstructures of apricot juices made with 20% puree (A) and 60% puree (B).

pressure on the particle size distribution (PSD) of two representative juice formulations. Particle size significantly decreased with increasing pressure independent of puree concentration in the juice (Table 2) The PSD curves shifted to the left, indicating the generation of smaller particle at the expense of mechanically disrupted larger ones. The mean particle size of the juice made with 60% puree for example was halved, decreasing significantly from 165 ± 0.52 µm at "0" MPa to 73 ± 0.91 µm at 70 MPa (p = 0.00) Table 2).

The particle size reduction effect of HPH has also been observed by several authors for tomato (Augusto *et al.*, 2012), passion fruit (Okoth *et al.*, 2000), apple (Zhu *et al.*, 2019) and citrus juices (Betoret *et al.*, 2009), as well as broccoli, tomato and carrot-based suspensions among others (Lopez-Sanchez *et* al., 2011). The untreated juices ("0" MPa) presented monomodal distributions with particle diameters extending in majority from 100 to 1000 µm. Processing at 35 MPa led to a broader distribution of particles, falling in the range of 10 to 1000 µm. At 70 MPa, the PSD curve further widens and presents a plateau of smaller particles with sizes roughly falling between 10 and 400 µm. The same behavior was observed for all puree percentages with increasing pressure. Peaks of 1 to 2% in volume occasionally appear beyond 1000 µm

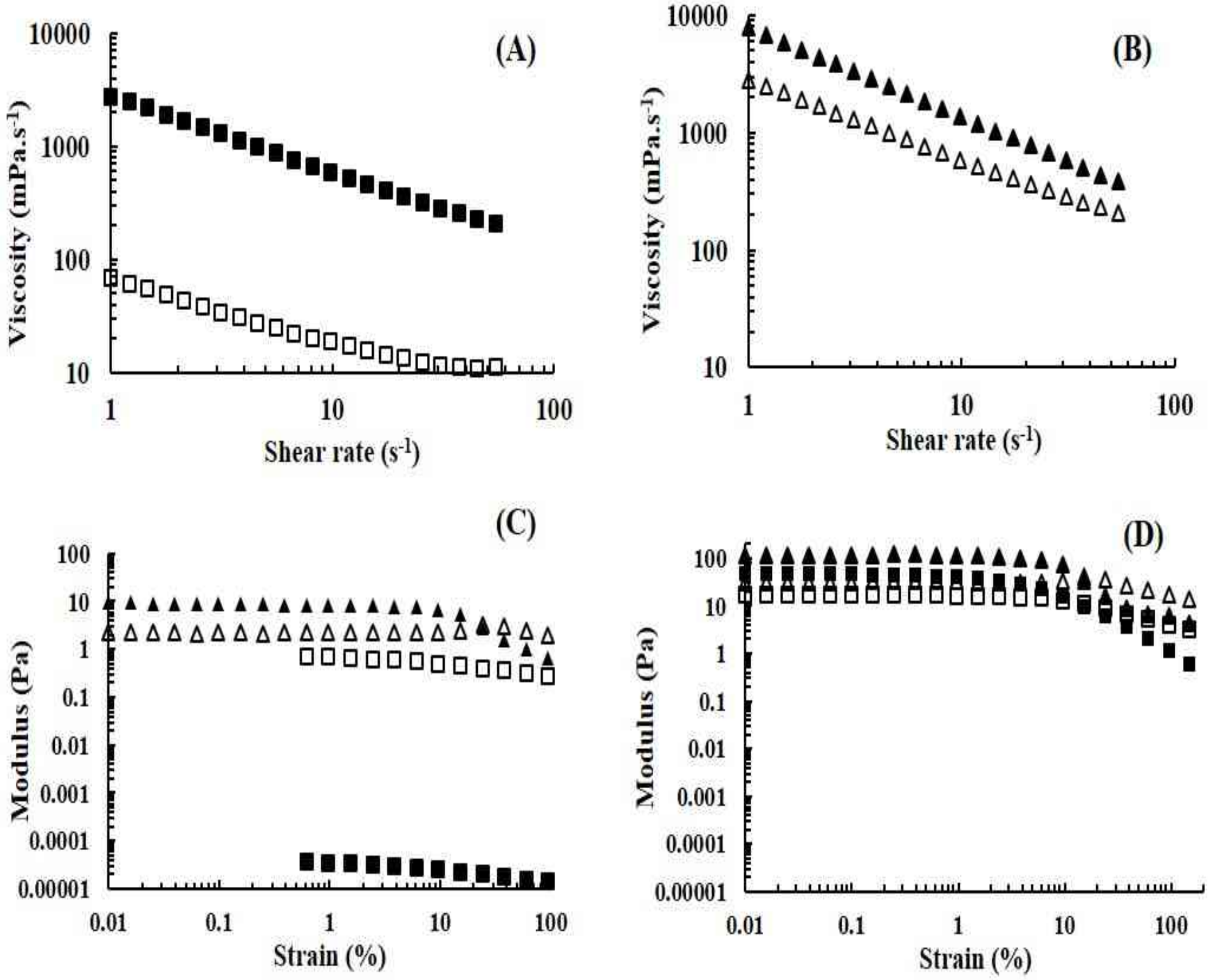


Figure 3. Viscosity as a function of shear rate for 20% (A) *and 60%* (B) *puree juices at "0" MPa* (white)*, and 70 MPa* (black) *at Day 0. Elastic* (black) *and viscous* (white) *moduli as a function of strain for 20% (C) (points below 1% strain for this sample not shown).*

with increasing pressure, a phenomena that could be attributed to particle aggregation occurring immediately after treatment (Kubo *et al.*, 2013). The reduction in *d* (0.1) particle diameter was more pronounced between "0" and 35 MPa than between 35 and 70 MPa, which may be caused by the physical limitation of the equipment in providing the higher shear required to disrupt smaller particles. A number of authors have emphasized the latter, whereby higher pressures above a certain point did not, or only minimally impacted the PSD of tomato juice (Augusto, Ibarz *et al.*, 2012; Kubo *et al.*, 2013), orange juice (Leite *et al.*, 2014), strawberry nectar (Moscovici Joubran *et al.*, 2019), mango juice (Zhou *et al.*, 2017), and pineapple pulp (Silva *et al.*, 2010).

The micrographs in Figure 2 are consistent with the PSD results. In the untreated ("0" MPa) samples, individual or clusters of large intact cells are discernable, along with some cell fragments obtained during the processing of the commercial apricot puree. Homogenization pressure breaks the whole cells and cell fragments, releasing cell content into the dispersed phase (Salehi, 2020), resulting in a larger number of cell fragments (Day *et al.*, 2010; Lopez-Sanchez *et al.*, 2011; Augusto *et al.*, 2012). The images taken post-treatment confirm the disruption of apricot cells caused by turbulence and shear (Tan and Kerr, 2015). After homogenization, the samples consist of dispersed ruptured cell wall materials and membranes that, depending on the insoluble solids content, are more or less interconnected. The particle network is suggested to be brought by an increased surface area of the suspended particles and a possible alteration of the composition of the serum (pectin and proteins), both of which are implicated in enhancing particle-particle interactions and driving more pronounced rheological properties like viscosity (Augusto *et al.*, 2012).

This is similar to what was observed by Augusto *et al.* (2012) and Moelants *et al.* (2013) for tomato juice and carrot suspensions respectively. The microstructures also reveal the effect of mechanical treatment on the shape and arrangement of particles (Espinosa-Muñoz *et al.*, 2013; Moelants *et al.*, 2013) which have been linked to the rheological properties of the product. Whereas the control samples mostly comprised smooth regularly shaped individual cells, the cell-wall fragments in treated ones appear to be irregular and rough, playing a role in product texture and physical stability.

3.3 The effect of HPH on rheological properties

Due to experimental design constraints, rheological measurements were repeated for the central point only (AJ3540); mean values and standard deviations are shown in Table 2; variations are assumed to be low for all other samples. Viscosity decreased with increasing shear rate for all samples, and the shear-thinning behavior of the samples was enhanced at higher pressures and with higher puree content, as seen by reduced flow behavior index values (n) (Table 2). Compared to the untreated samples (“0” MPa), the treated ones presented higher viscosities over the entire range of shear rates, highlighting an increase of particle-particle interactions in the HPH-treated samples (Figure 3). Having smaller particles after the HPH treatment indicates that there is a larger surface area, a lower mean distance between the particles and consequently higher particle-particle interactions, which explains the higher viscosities (Augusto *et al.*, 2013). The main viscosity increment occurred between “0” and 35 MPa, being smaller between 35 and 70 MPa for all samples; for example, AJ020 and AJ060 saw their viscosities increase by 617 and 133% respectively at 35 MPa and only negligibly thereafter (12 and 1% between 35 and 70 MPa, respectively). The changes are in accordance with the PSD results, whereby a higher particle size reduction was observed between “0” and 35 MPa. The flow behavior was well-described using the Ostwald de Waele model ($R^2 > 0.98$) for all juices, and pressure was demonstrated to also improve the consistency (k) of the products. Similar results were reported by Augusto *et al.* (2012) and Zhou *et al.* (2017) for tomato and mango juices respectively.

The increase in pulp percentage with pressure for the same amount of puree led to an increase in the viscosity of the suspensions, so the viscosity increase brought by the particles is directly linked to the volume that they occupy in the solution. The flow behavior of plant tissue-based suspensions has repeatedly been shown to predominantly be dictated by the particles (Espinosa-Muñoz *et al.,* 2013; Moelants et al., 2013; Leverrier *et al.*, 2016;), and the significant correlation found between the percentage of pulp and juice viscosity (0.943) and relative viscosity (0.886) validate the above. Particle shape is likely to also be impacting the pulp percentage which is linked to the particles' stacking capacity. Leverrier *et al.* (2016) established that smaller and smoother apple puree particles following different levels of grinding led to a decrease in pulp percentage

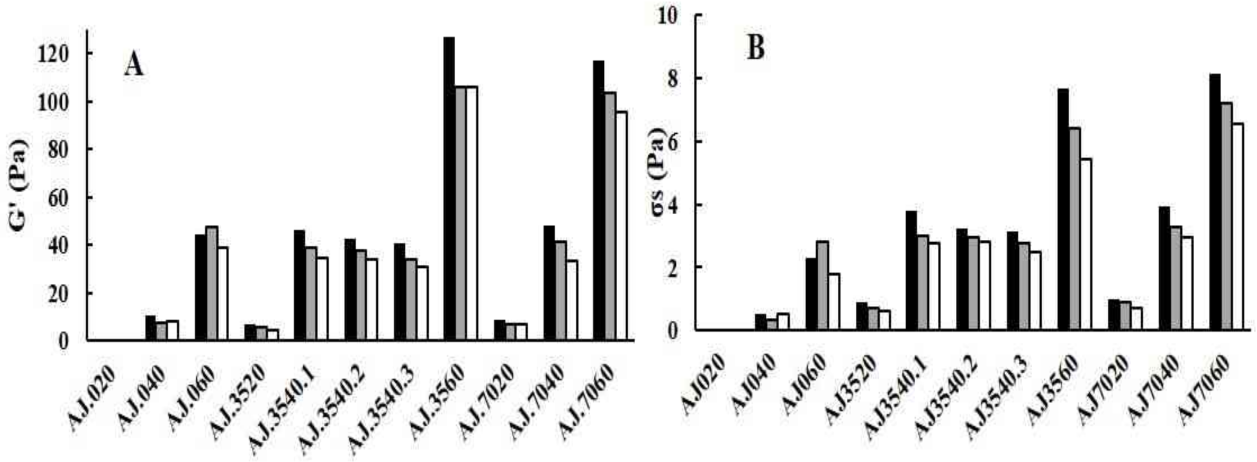


Figure 4. Evolution of G' *(A) and yield stress (B) with time (day 0: black; day 1: grey, day 7: white).*

and a subsequent decrease in viscosity. The opposite is found here for apricot cells, where the smaller but more irregularly-shaped particles resulting from HPH treatment led to an increase in the pulp percentage, causing an increase in viscosity and the increase of the elastic modulus *G'*. We therefore hypothesize that the shape of the particles directly impacts the rheology of the plant particle suspensions even if the plant particles are soft because it allows the percolation threshold to be reached at a lower amount of insoluble material. Getting away from the sphere, the maximum volume fraction becomes smaller, which means that for the same amount of material, aspherical particles will occupy more space than spherical particles, thus allowing the creation of a percolated network of particles (or its reinforcement).

HPH-treated samples also see their serum viscosities decrease post-treatment (Table 2), a phenomenon that can be attributed to pressure-induced pectin degradation (Moelants *et al.*, 2014). The effect of the viscosity of the continuous phase on the particle network is minimal, however serum composition (quantity and molecular weight of pectins) has been shown to modify particle-particle interactions (Moelants *et al.,* 2013).

The viscoelastic properties provide an indication of the strength of the inter-particle interactions within the system (Dahdouh *et al*., 2016), and the increase in the elastic modulus (*G'*) and yield stress (*σs*) post-treatment confirm the existence of a network of interacting particles. With the exception of the untreated and most diluted sample AJ020, the ten others exhibited the typical behavior of weak gels at constant angular frequency and at low strain (< 10%), with *G'* consistently higher than *G"* (Table 2, Figure 4) corresponding to a solid-like behavior in the linear viscoelastic region (LVR) (Day *et al.*, 2010).

Although to a lesser extent than by puree content, this behavior is also driven by pressure (p = 0.0099). For instance, applying a 35 MPa pressure led to 301 and 183% increases in *G'* for 40 and 60% puree juices respectively, again with less pronounced increases occurring between 35 and 70 MPa. AJ020's initial fluid-like behavior (*G"* > *G'*) evolved to a weak gel with rising pressure, although considerably lower in strength in comparison to the other products. Zhou *et al.* (2017) and Augusto *et al.* (2013) attributed the increase in *G'* values for mango and tomato juices respectively after a single HPH pass to enhanced suspended solid interactions induced by particle size reduction,

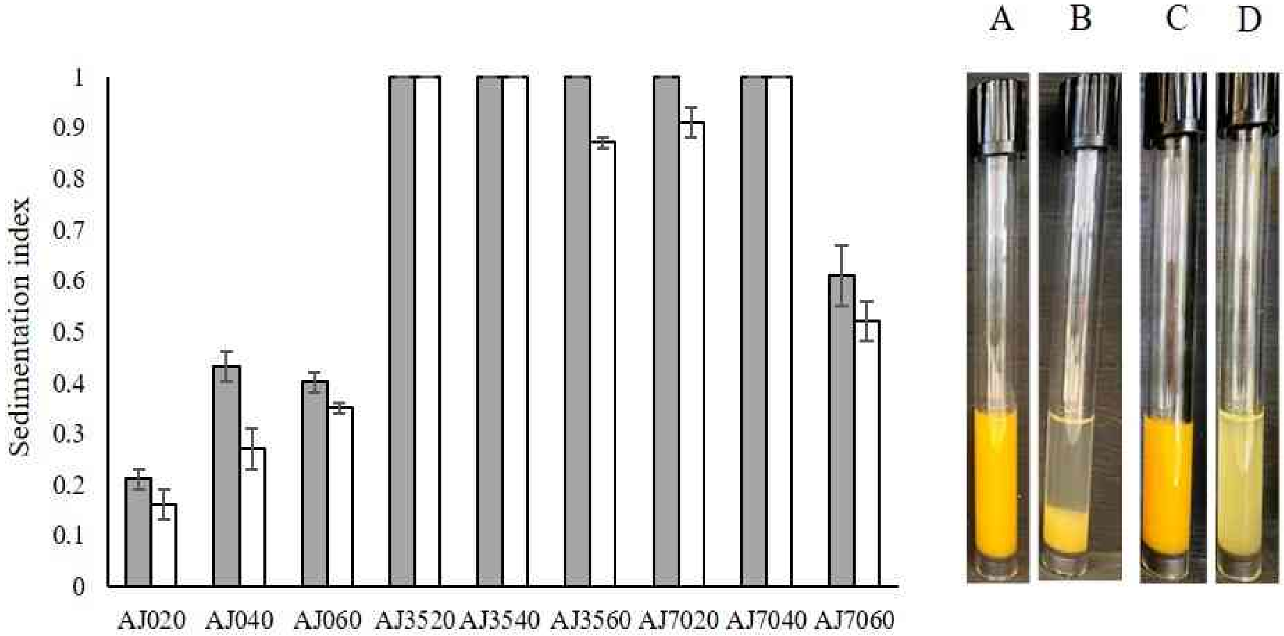


*Figure 5. Effect of HPH on apricot juice sedimentation at day 1 (*grey*) and day 7 (*white*). The samples shown are 1:8 dilutions of the original samples. The sedimentation index is assumed to be equal to 1 for all samples at day 0. Vertical bars are standard deviations. 40% puree juice samples at day 7 (A) undiluted untreated, (B) diluted untreated, (C) undiluted 35 MPa-treated, (D) diluted 35MPa treated.*

but Leverrier *et al.* (2016) and Dahdouh *et al.* (2015) also mention particle shape as a determinant of network strength; irregular broken cells and cell wall fragments have a higher tendency to build structure in rest conditions (Moelants *et al.,* 2013).

This is related to the volume fraction, defined as the volume occupied by the particles in the total volume. While hard spheres can randomly pack with a maximum volume fraction close to a value of 0.66 (Barnes, 2000), irregular particles will have a less effective packing and therefore a lower maximum volume fraction. This is observed here with the percentage of pulp: in products that have not undergone HPH treatment, particles have almost spherical shapes and the percentage of pulp goes from 5.97 to 17.75% for samples containing 20 to 60% puree (Table 2). After HPH treatment, the shapes of the particles move away from the sphere (Figure 2) and the percentage of pulp increases up to 13.93 to 41.94% for the same samples, without addition of insoluble content. Therefore, it seems that the shape of the apricot juice particles has a direct impact on their packing. The same tendency was observed by Leverrier *et al.* (2016), were mechanical treatment on apple purees resulted in more spherical particles and a lower pulp percentage. However, plant particles being soft, when mechanical treatment induces a modification of the morphology, it also modifies the architecture of the particles and thus their rigidity, the latter also being involved in different packing abilities: highly deformable and compressible particles are able to pack more densely.

When strain is increased, *G'* decreases while *G"* starts to increase, going beyond a cross-over point where *G"* is higher than *G'* and the dispersion flows. The yield stress suggests that the juices possess a network structure that requires a given stress for flow to occur (Moelants *et al.*, 2014), and the values for yield

stress increase with pressure (Figure 4, Table 2).
This network structure is a result of direct contact between the non-colloidal particles that make up the large fraction of the samples. With time and at day 7 post-treatment, the *G’* and *σs* values for all samples followed a downward trend, declining by a maximum of 33% and 31% respectively for AJ3520, but retaining their weak gels. This weak gel structure, which is essential for the physical stability of fruit juices containing pulp particles, allows the product to remain pourable.

3.4 The effect of HPH on physical stability

Besides AJ020, no sedimentation was observed within 7 days of refrigerated storage for all original undiluted samples. Figure 5 shows the sedimentation indices for the 1:8 dilutions of the original samples at 4 °C. Diluting the samples emphasizes the role of reduced particle size in enhancing their interactions, beyond their concentration in the system. The formation of an interconnected network depends on the quantity of insoluble particles in suspension, an insufficient quantity of which would slow but not stop sedimentation.
Untreated samples with larger particles were unstable, compared to pressure-treated ones whose particles were smaller and did not or only minimally exhibited sedimentation. For example, AJ040 sediments while its pressure-treated counterparts, AJ3540 and AJ7040 are stable over the same duration. The tendency for large particles to sediment within only one day of storage was higher, attributable to the lower volume that they occupy in the system and the weaker interactions between them (Kubo *et al.*, 2013; Leverrier *et al.*, 2016). In the absence of an interconnected network, particles of larger diameters sediment at a faster rate, driven by gravitational forces. Even diluted, AJ3520, AJ3540 and AJ7040 were stable throughout the storage period, supporting the hypothesis that enhanced particle-particle interactions resulting from size and shape alterations during treatment play a role in stabilizing the juices. This was supported by the significance of the quadratic effect term (Pressure2) (p = 0.0358) on the sedimentation index for diluted samples stored at 4 °C. Repetitions are needed to further confirm the results and to explain the instability of AJ7020 for example. AJ7060 and AJ3560 have the highest pulp contents (41.98 and 43.38% respectively) and both exhibit sedimentation. An optimal pulp content may eventually be deduced for an enhanced system stability.

3.5 The effect of pulp content on rheological properties and overall stability

The upward evolution of pulp content with pressure (Table 2) is attributable to HPH-induced modifications in particle morphology. For the same puree quantity, irregularly-shaped interconnected cell wall fragments in pressure-treated samples occupy a higher volume as compared to round-shaped individual or clustered cells in untreated samples, the maximum volume fraction of which is lower. For example, AJ7060 has a pellet that is 2.3 times heavier than its untreated counterpart (AJ060), as its irregularly shaped particles are able to stack more densely after HPH. The pulp content parameter then plays a role in structuring and stabilizing the system, which is why the rheological behavior of the apricot juices is modelled as a function of pulp percentage. Although it is a measured variable rather than a factor of the experimental plan, the pulp % represents the interaction of the two factors (pressure level and puree quantity) and is strongly correlated to *G’*, yield stress and viscosity with Pearson correlation coefficients > 0.93. The rheological parameters as a quadratic function of the pulp % are shown in Figure 6, with R^2 regression values consistently higher than 0.92. The elastic modulus, yield stress, viscosity and consistency indices increased when the pulp content increased. With the same amount of pulp but different initial puree contents and treatments, AJ040 undergoes sedimentation while AJ3520 remains

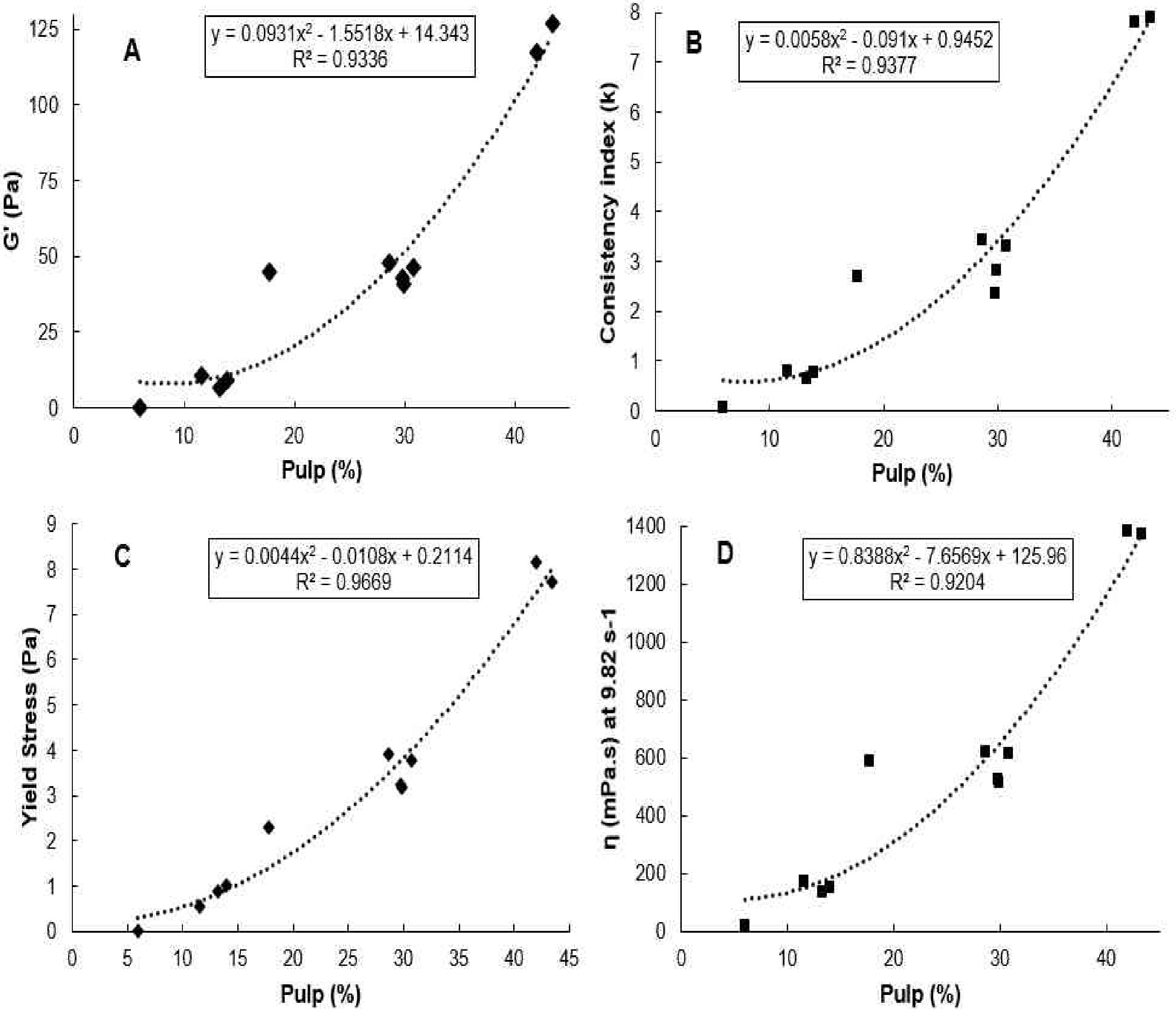


*Figure 6. Dynamic (*A, C*) and steady-state (*B, D*) rheological parameters as a function of pulp content.*

stable. This suggests that the particles post-HPH stack less densely compared to untreated ones in the same volume. This is related to the particle size reduction and particle geometry modifications (aspherical) brought by HPH, which affect the maximum volume fraction (Barnes, 2000). AJ060 can be distinguished from the other juices on the basis of its relatively low pulp content but higher rheological properties, mainly driven by puree concentration and larger particles that have a higher resistance to flow (Leverrier *et al*., 2016). Its gel-like behavior, similar to other samples, confirms the first order effect of suspended particles on the viscoelastic properties. With comparable *G'*, viscosity and *k* values to AJ3540 and AJ7040, but considerably less pulp (Table 2), AJ060 destabilizes while the other samples do not, underlining the hypothesis that pulp content is a principal determinant of the macroscopic behavior. The network of irregularly-shaped particles created in the pressure-treated samples enhances particle-

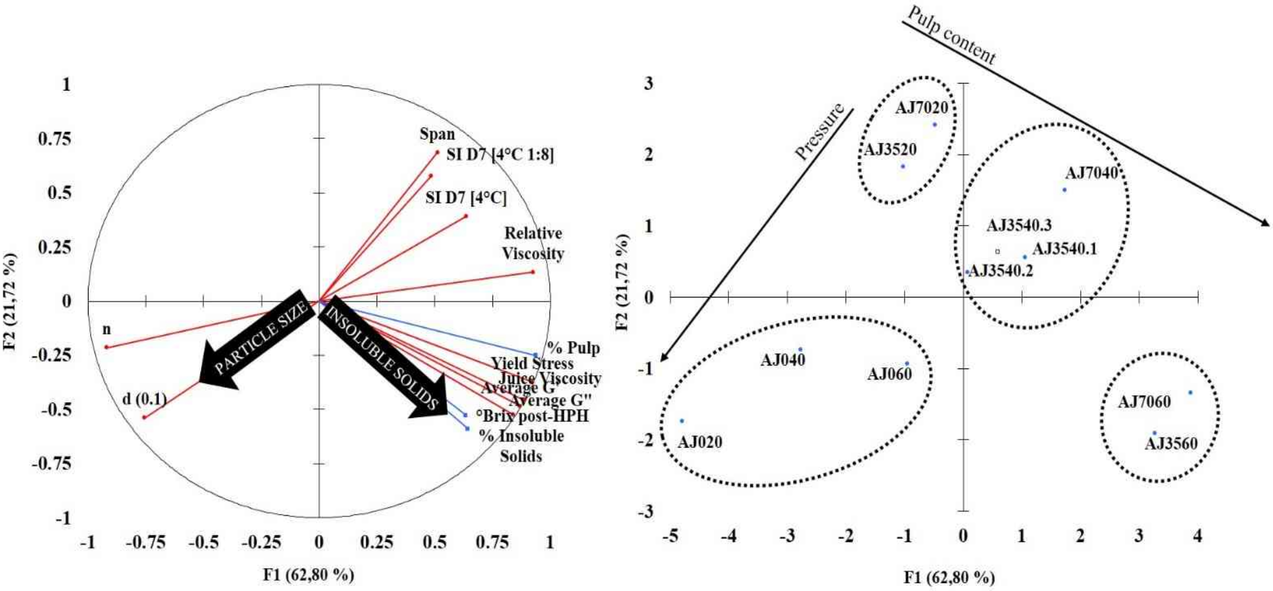


*Figure 7. Correlation loading plot (*A*) of the structural (*d *(0.1), span), rheological (*G', G"*, yield stress, juice viscosity and relative viscosity) and stability properties (SI D7 4°C) (*main variables in red*) and compositional properties (°Brix, % insoluble so solids, % pulp) (*supplementary variables in blue*), and corresponding score plot of the 11 juices (*B*).*

particle interactions, the particles of which stack less densely. The set of rheological parameters modeled in Figure 6 is closely linked to product texture (Espinosa-Muñoz *et al.,* 2012) and physical stability over time. Indeed, a negative correlation of - 0.836 between *d* (0.1) and relative viscosity was found. Higher juice viscosity and viscoelastic moduli brought by smaller particle sizes have been suggested to govern physical stability (Augusto *et al.*, 2012; Salehi, 2020), and the data obtained for apricot juices are consistent with the latter. The models presented here can be of industrial use, in product and process optimization, or used in cooking and kitchen applications.

3.6 Product mapping: a direct representation of the relationship between product properties and process/formulation

The principal component analysis (PCA) plotted in Figure 7 maps the 11 products as a function of rheological, structural and stability indicators. The selected 2D projection (F1, F2) displays more than 84% of the total information. Looking at the correlation loading plot (Figure 7), the F1 axis holds close to 63% of the information, and is related to the rheological and composition indicators, confirming the correlation between pulp percentage and rheological properties. F2 explains the rest of the information mainly through particle size and sedimentation index. The samples are well-dispersed on the score plot (Figure 7B), which means that they are well discriminated, indicating that a wide range of particle sizes, as well as rheological and stability profiles were generated. Different groups can be defined according to both F1 and F2, and based on the experimental design factors: pressure and puree percentage. Samples clustered together have similar attributes and *vice versa*. The center point triplicates AJ3540 are very closely positioned, which ratifies the repeatability of the results.

Untreated samples fall close to each other, while

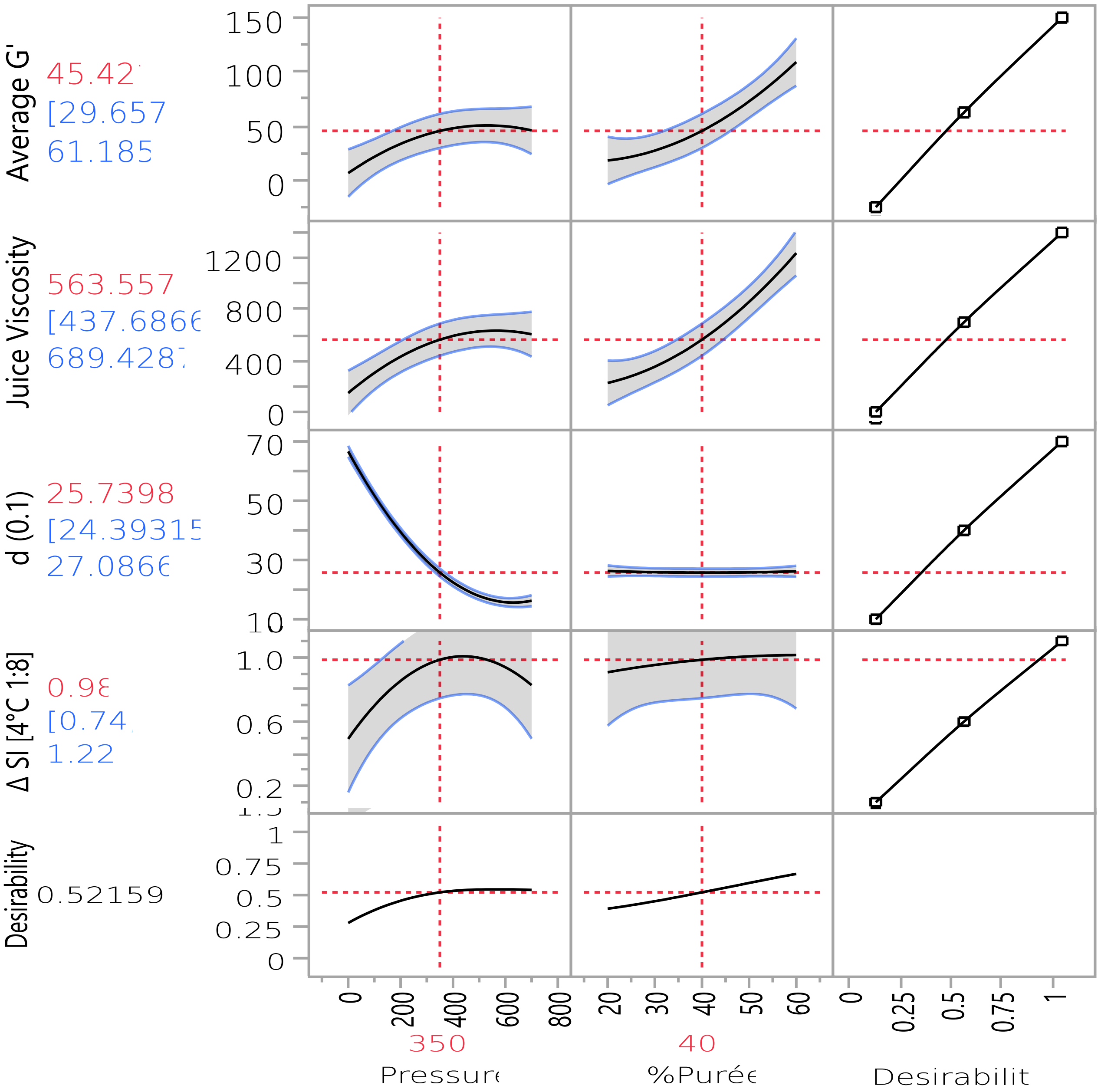


Figure 8. Prediction models of the measured variables (Y axes) as a function of the experimental plan factors (X axes).

pressure-treated ones can be clustered according to their puree percentage. As previously shown, a pressure of 70 MPa did not alter product properties much, beyond what has already been brought by applying a 35 MPa pressure. Along F1, products are mainly organized according to their rheological properties and their pulp content, with the ones on the right, namely AJ7060, AJ3560 and the center-points having high values for pulp content, viscosity, elastic and viscous modulus, and those falling on the left (that is AJ7020, AJ3520) having the lowest values of the same parameters. Products with large coordinates on F1 possess strong networks of smaller particles, and are more stable. Along F2, products are mainly grouped as to their particle size and stability. Untreated samples (AJ020, AJ040, and AJ060) have larger particles and exhibit more sedimentation. Similar results were reported by Espinosa *et al.* (2012), where apple purees produced from different varieties of apple and by three different grinding treatments were essentially discriminated according to pulp/insoluble material content and particle size. These key structural parameters were found to be the determinants of the texture and sensory properties of the purees (Espinosa-Muñoz *et al.*, 2012).

3.7 Tailoring texture by modifyng formulation and process parameters

Figure 8 displays the profiles of the measured variables plotted as a function of the experimental design factors (pressure and puree percentage). These prediction profiles allowed the selection of the most significant indicators that govern the structure-function relationship in this system. Strong correlations could be underlined between a higher pressure for example and a lower particle size. Puree percentage governs mainly the texture, and the higher it is, the higher the viscosity and viscoelastic properties of the products. The prediction profiles also constitute a valuable and practical desirability-optimization tool for the industry, with the possibility of setting target values for the measured variables and acquiring the needed optimal levels of pressure and puree percentage to obtain them.

4. Conclusion

Thanks to an experimental design, a wide diversity of apricot juices varying in rheological properties and physical stability was produced, highlighting the principal factors that influence the macroscopic behavior of this type of suspensions. The volume occupied by the insoluble particles in the system emerged as a major determinant of juice texture and stability. This occupied volume was monitored solely through the use of mechanical treatment, as non-spherical particles post-HPH were found to occupy a greater volume in the system. This work thus confirms the ability of apricot cell wall to withstand sedimentation during storage and validates HPH as a promising tool in juice stabilization, linking its particle size reduction capacity to greater stability.

We propose to see the pulp content as an indicator of the particles' volume fraction, which governs the rheological behavior and consequently the stability of the juice. Thanks to the mechanical process applied, a low quantity of apricot puree or particles was sufficient to structure the medium and create a network. The pressure-dependent structural changes were shown to intensify particle-particle interactions. The network structure confers the characteristic weak gel behavior to the juices, which consists of an elevated elastic modulus and a yield stress, essential to ensure physical stability over time. In that way, mechanical processing can be considered as a valuable tool to control and tailor the texture and the stability of plant particle-based matrices. Within the studied product space, it was possible to model the physical properties of the juices as a function of both pressure and pulp content, and to elaborate a set of texture and stability indicators that could be directly exploited by the industry.

The findings pin down the fundamental levers for the formulation of innovative clean-label products with a broad spectrum of textures: HPH as a tool to bring desirable structural changes to pulpy juice systems, and plant cell walls as structuring materials. The versatility of plant cells could lead to innovative applications in the fruit-based beverage industry or in the culinary field. It would be interesting to continue investigating the impact of mechanical processing on the functionalization of the plant cell walls present in fruit juices and purées by diversifying the mechanical processing tools used (in particular through the use of domestic tools such as grinders, blenders or mixers) on a wider panel of fruit and vegetables, as the reaction to mechanical processing is closely linked to the raw material used.

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