

1 **Effect of high pressure homogenization and high power ultrasound on some physical properties**
2 **of tomato juices at different concentration levels**

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14

15 **Abstract**

16 The effect of high pressure homogenization (HPH) and ultrasound (US) on some physical properties
17 of tomato juices with different soluble solids content (5.0, 7.5, 10.0 °Brix) was studied. Samples were
18 subjected to HPH up to 150 MPa or US up to 30 min. The energy efficiency associated to the
19 processes was evaluated. Results showed that stress type and product concentration influenced the
20 changes of tomato juice physical properties induced by HPH and US processing. In particular, HPH
21 and US treatments led to similar increases in G' and consistency of 5.0 and 7.5 °Brix juices. These
22 changes were accompanied with redness loss and attributed to cell disruption and consequent increase
23 of inter-particle interactions. Increasing tomato juice concentration to 10.0 °Brix, HPH treatments
24 were more effective than US in changing sample consistency and gel-like properties. The process
25 energy efficiency showed that lower energy was involved in HPH in comparison to US.

26

27 *Keywords:* high pressure homogenization, high power ultrasound, tomato juice concentration,
28 physical properties, energy efficiency

29

30

31 **Highlights**

32

33 HPH and US caused physical modifications in tomato juice.

34 Tomato juice concentration greatly affected process performances.

35 HPH caused greater changes in physical properties of 10.0 °Brix tomato juice than US.

36 HPH and US were compared in terms of energy efficiency.

37

38 **1. Introduction**

39

40 High pressure homogenization (HPH) and high power ultrasound (US) are nowadays proposed as
41 novel techniques to steer desirable structure and functionality of plant-based foods. Changes in the
42 physical properties of biopolymers, such as cellulose, starch, pectin and protein, have been described
43 in fruits and vegetables (e.g. banana, mango, pineapple, peach, tomato, broccoli, carrot) derivatives
44 subjected to HPH and US (Bengtsson and Tornberg, 2011; Calligaris et al., 2012; Kubo et al., 2013;
45 Lopez-Sanchez et al., 2011a; Lopez-Sanchez et al., 2011b; Rojas et al., 2016; Silva et al., 2010).
46 These effects are attributable to the intense mechanical stresses suffering the vegetable matrix during
47 HPH and US processes. In particular, during HPH process, a fluid, which is pumped through a narrow
48 gap valve by means of a pressure intensifier, undergoes intense mechanical forces and elongational
49 stresses at the valve entrance and in the valve gap, while turbulence, cavitation and impacts with the
50 solid surface occur at the gap outlet (Floury et al., 2004a; Floury et al., 2004b). During US treatment,
51 mechanical wave propagation into a fluid may cause cavitation phenomena, which is the spontaneous
52 formation and collapse of bubbles, that leads to the generation of local extreme temperatures and
53 pressures, which in turn produce turbulence and shear stresses (Barbosa-Cánovas and Rodrigues,
54 2002; Leighton, 1995; Mason, 1998). It has been speculated that the intense stresses delivered by
55 HPH and US lead to cell disruption with leakage of plant constituents, including biopolymers, in the
56 serum. Moreover, processes would modify biopolymer structure by inducing conformational changes
57 as well as reducing polymer size. As a consequence, more polymer chain would be available for
58 bonding, giving rise to novel inter-particle interactions and a different type of network, which is
59 accompanied by a change of the rheological properties (Colle et al., 2010; Seshadri et al. 2003; Thakur
60 et al., 1995). Modification of biopolymers physical properties are reported to highly depend on matrix
61 characteristics and HPH and US intensity, that is pressure level and number of passes applied during
62 HPH, or ultrasonication time (Anese et al., 2013; Augusto et al., 2012; Augusto et al., 2013; Lopez-
63 Sanchez et al., 2011b; Tan and Kerr, 2015; Vercet et al 2002a; Yu et al., 2016). From an industrial

64 point of view, the choice between HPH and US to steer plant food material physical properties goes
65 through the evaluation of advantages and drawbacks of each technology. Final product characteristics
66 as well as energy and ownership costs would represent the driving criteria. The energy exchanges
67 involved during HPH and US processes are represented by the energy density, which is the amount
68 of energy provided to the fluid per unit volume during the process, as well as the power demand and
69 energy consumption of the equipment (Raso et al., 1999). To our knowledge, very few data are
70 available in the literature about HPH and US energy aspects (Baumann et al., 2005; Bermudez-
71 Aguirre and Barbosa, 2012; Donsì et al., 2013; Mañas et al., 2000; Stang et al., 2001). Cortés-Muñoz
72 et al. (2009) and Calligaris et al. (2016) evaluated the energy density in HPH for an oil/water emulsion
73 by considering the pressure drop. Toma et al. (2011) investigated the energy conversion efficiency in
74 US for organic solvents, and claimed that it is strongly dependent upon the fluid as well as sonication
75 equipment (e.g. its geometry), and the operation mode (e.g. temperature, amplitude of the US field).
76 Tomato is one of the most worldwide consumed crops. Due to its high versatility, tomato as raw
77 material is widely used to obtain different derivatives, such as juice, puree, pulp, paste, that can be
78 directly consumed or used as ingredients in many food formulations (Gould, 1991). Therefore, it has
79 high relevance for food industry. It has been already demonstrated that both HPH and US might
80 modify tomato physical properties (Anese et al., 2013; Colle et al., 2010; Kubo et al., 2013; Panozzo
81 et al., 2013; Tan and Kerr, 2015). In particular, parameters such as viscosity, consistency, red color
82 and particle size were observed to change in tomato products subjected to HPH and US processes
83 (Anese et al., 2013; Augusto et al., 2012; Augusto et al., 2013; Bayod et al., 2008; Kubo et al., 2013).
84 The application of US in combination with heating (thermosonication) and pressure
85 (manothermosonication) allowed to increase the sole effect of US (Vercet et al., 2002; Wu et al.,
86 2008).
87 To our knowledge, HPH and US performances in the attempt to deliver functionality of plant-based
88 foods are hardly comparable due to scarce information. Moreover, data on the role of tomato solids
89 concentration in affecting changes in physical properties as induced by HPH or US are fragmentary

90 (Bayond et al., 2008; Bayod and Tornberg, 2011; Valencia et al., 2003). Therefore, the aim of this
91 study was to investigate the effect of HPH and US on some physical properties of tomato juices with
92 different soluble solids contents. To this purpose, tomato juices with 5.0, 7.5, 10.0 °Brix were
93 subjected to HPH and US for increasing pressure levels or treatment time, respectively, and the
94 changes in their viscoelastic properties, Bostwick consistency, precipitate weight ratio, pectin
95 esterification degree and microstructure were studied. Finally, estimation of the energy density
96 transferred to the juice during processing, as well as measurement of electrical energy consumption
97 of the laboratory devices were performed to compare HPH and US processes from the point of view
98 of energy efficiency.

99

100 **2. Material and Methods**

101 *2.1. Sample preparation*

102 Tomato juice at 5.0, 7.5 and 10.0 °Brix (corresponding to 5.2 ± 0.1 , 8.3 ± 0.1 and 10.7 ± 0.1 % dry matter,
103 respectively) was obtained by dilution of commercial tomato paste (21 °Brix) in distilled water. The
104 pH of the juice was 4.5 ± 0.1 .

105

106 *2.2. Treatments*

107 *2.2.1. High pressure homogenization*

108 A continuous lab-scale high-pressure homogenizer (Panda Plus 2000, GEA Niro Soavi S.p.a., Parma,
109 Italy) supplied with two Re+ type tungsten carbide homogenization valves, with a flow rate of 2.5
110 cm^3/s , was used. The first valve was the actual homogenization stage and was set at increasing
111 pressures from 20 to 150 MPa. The second valve was set at the constant value of 5 MPa. Aliquots of
112 tomato juice were introduced into the equipment at 10 ± 1 °C and cooled using an ice bath just after
113 the treatment. The maximum temperature reached by the sample was 45 ± 2 °C.

114

115 *2.2.2. High power ultrasound*

116 An ultrasonic processor (Hieschler Ultrasonics GmbH, mod. UP400S, Teltow, Germany) with a
117 titanium horn tip diameter of 22 mm was used. The instrument operated at constant frequency and
118 ultrasound amplitude of 24 kHz and 100 μm , respectively. Aliquots of 150 mL of tomato juice were
119 introduced into 250 mL capacity (110 mm height, 60 mm internal diameter) glass vessels. The tip of
120 the sonicator horn was placed in the centre of the solution, with an immersion depth in the fluid of
121 250 mm. Treatments were carried out for increasing time periods, up to 30 min. During the treatments,
122 the temperature was controlled using a cryostat set at 4 $^{\circ}\text{C}$ to dissipate the heat generated during
123 treatment. Temperature never exceeded 45 ± 2 $^{\circ}\text{C}$. Following the treatments, the samples were cooled
124 in an ice bath.

125

126 *2.3. Energy density estimation*

127 The energy density (E_v , MJ/m^3) transferred from the homogenization valve to the sample was
128 determined as described by Stang et al. (2001), according to eq. (1):

129

$$130 \quad E_v = \Delta P \quad (1)$$

131

132 where ΔP is the pressure difference operating at the nozzles.

133 The power density (P_v , kW/m^3) transferred from the ultrasound probe to the sample was determined
134 calorimetrically by recording the temperature (T , K) increase during the treatment, following eq. (2)
135 (Raso et al., 1999).

136

$$137 \quad P_v(T) = mc_p(\partial T/\partial t)/V \quad (2)$$

138

139 where m is the sample mass (kg), c_p is the sample heat capacity ($\text{kJ}/\text{kg K}$), V is the sample volume
140 (m^3), and t (s) is the time frame of treatment considered. The heat capacity was estimated on the basis
141 of sample composition and as a function of the temperature, based on the correlations by Choi and

142 Okos (1986). Power density is markedly affected by temperature, and its measurement should be
143 performed at adiabatic conditions, which however occur only at the very beginning of the treatment
144 (Raso et al., 1999). In order to achieve at least an estimation of the energy density over the whole
145 treatment while including the effect of temperature, the power density was measured as a function of
146 temperature for a separate test with thermal insulation and without temperature control, up to the
147 maximum temperature of 45 °C.

148 Later on, the energy density was estimated by integration of the power density as:

149

$$150 \quad E_v = \int P_v(T) dt = \sum(P_v(T)\Delta t) \quad (3)$$

151

152 on the whole treatment time.

153

154 *2.4. Electrical energy consumption measurement*

155 For both the HPH and US treatments the energy requirement was estimated by measuring the
156 electrical consumption at the mains supply. The high pressure homogenizer was supplied with three-
157 phase 400 V electrical power. Thus a three-phase energy logger was inserted (Kilo Box, Electrex,
158 Reggio Emilia, Italy) to measure the electrical consumption (MJ/m³) as active power, that is the
159 effective power used by the apparatus, and the power factor, that is the ratio between the “active
160 power” and the “apparent power” related to the power supplied by the net. The power factor is in the
161 range 0-1 and it should be as high as possible for optimal exploitation of the electrical energy supplied.
162 The ultrasonic processor was instead supplied with single-phase 230 V electrical power, and a power
163 meter (PC-300, Lafayette, Taiwan) was connected to measure the electrical power and thus calculate
164 the electrical energy (MJ/m³) for the whole treatment.

165

166 *2.5. Analytical determinations*

167 *2.5.1. Soluble solids (°Brix)*

168 The soluble solids (°Brix) were measured using a hand Refractometer (Unirefrax, S.A Bertuzzi,
169 Milan, Italy). Measurements were performed at 25 °C. The refractometer prism was cleaned with
170 distilled water before each analysis.

171

172 *2.5.2. Temperature measurement*

173 The sample temperature was measured just before and immediately after (i.e. before the cooling step)
174 each treatment by a copper-constantan thermocouple probe (Ellab, Hillerød, Denmark) immersed in
175 the tomato juice, connected to a portable data logger (mod. 502A1, Tersid, Milan, Italy).

176

177 *2.5.3. Colour*

178 Colour analysis was carried out using a tristimulus colorimeter (Chromameter-2 Reflectance,
179 Minolta, Osaka, Japan) equipped with a CR-300 measuring head. The instrument was standardised
180 against a white tile before measurements. Colour was expressed in L*, a* and b* scale parameters
181 and a* and b* were used to compute the hue angle ($\arctan b^*/a^*$). An increase in hue angle is an index
182 of redness loss.

183

184 *2.5.4. Rheological properties*

185 Rheological measurements were carried out using a controlled stress rheometer (SR5, Rheometric
186 Scientific, Germany) equipped with serrated parallel plate geometry (40 mm diameter, 2 mm gap).
187 The temperature was maintained constant at 25 °C using a Peltier system. Samples were placed
188 between the plates of the rheometer and left to rest 5 min after loading before testing. This resting
189 time was sufficient for the sample to relax and reach a constant temperature. Dynamic strain sweep
190 tests were carried out at 1 Hz between 0.1% and 100% strain to determine the linear viscoelastic
191 range. Frequency sweep tests were performed from 0.1 to 10 Hz within the linear viscoelastic range.
192 Data obtained were storage modulus (G'), loss modulus (G'') and $\tan \delta$ (G''/G'). Statistical
193 comparisons were made at 0.1 Hz.

194

195 *2.5.5. Bostwick flow index*

196 Samples were placed into Bostwick consistometer (RG Strumenti srl, Parma, Italy). This empirical
197 test consists in allowing the sample to flow under its own weight along a sloped stainless steel tray
198 for 30 s at room temperature (23 °C). The distance (cm) covered by the sample was recorded and the
199 inverse of the distance (cm⁻¹) was used to express the Bostwick consistency index. An increase of this
200 parameter is associated to high sample consistency.

201

202 *2.5.6. Precipitate weight ratio*

203 Precipitate weight ratio was determined using the method of Colle et al. (2010), with minor
204 modifications. Tomato juice (25 g) was centrifuged (Beckman, Avant J-25 centrifuge, Palo Alto,
205 California, USA) at 45000 g for 30 min at 15 °C. The percentage of precipitate weight ratio of the
206 pellet was calculated as:

207

208
$$P = (W_p/W_t) \cdot 100 \quad (4)$$

209

210 where W_p and W_t are the precipitate and tomato juice weights, respectively.

211

212 *2.5.7. Determination of degree of esterification*

213 The determination of the degree of esterification was carried out using the method of Chou and
214 Kokini (1987). 60 g of tomato juice were centrifuged (Beckman, Avant J-25 centrifuge, Palo Alto,
215 California, USA) at 7500 g for 15 min at 20 °C. The supernatant was filtered under vacuum through
216 filter paper (RPE ACS, Carlo Erba, Milano, Italy) and an equal volume of 2-propanol was added to
217 the filtrate to precipitate the isopropanol-insoluble pectins. After 15 min stirring, the suspended solids
218 in the water-isopropanol mixture were centrifuged at 7500 g for 15 min at 20 °C and isopropanol was
219 removed by means of vacuum dehydration (Laborota 4001 Efficient, Hedolph Instruments,

220 Schwabach, Germany). The water-soluble pectins were decoloured by acetone:pentane solution (2:1).
221 10 mL 1% decoloured pectin solution were titrated with 0.05 N NaOH (titration A). Afterwards,
222 20 mL 0.5 N NaOH were added to de-esterify the pectin and, after 30 min, 20 mL 0.5 N HCl were
223 added to neutralise the NaOH. This mixture was titrated with 0.1 N NaOH (titration B), using
224 phenolphthalein as indicator. The degree of esterification (DE), expressed as a percentage, was
225 calculated using the following equation:

226

$$227 \quad DE = [B/(A + B) \cdot 100] \quad (5)$$

228

229 *2.5.8. Images*

230 Tomato juice images were captured using a digital camera (Nikon D3, Nikon Corporation, Tokyo,
231 Japan) mounted on an adjustable stand positioned 50 cm above a black cardboard base where the
232 sample was placed. Light was provided by two 250 W frosted photographic floodlights in a position
233 allowing minimum shadow and glare. Images were saved in the jpg file format.

234

235 *2.5.9. Microstructure*

236 Tomato juice microstructure was analysed using an optical microscope (Leica DM 2000, Leica
237 Microsystems, Heerburg, Switzerland) connected to a Leica EC3 digital camera (Leica
238 Microsystems, Heerburg, Switzerland). The images were captured using the 200× objective
239 magnification.

240

241 *2.6. Data analysis*

242 The results are the average of at least two measurements carried out on two replicated experiments
243 ($n \geq 4$). Data are reported as mean value \pm standard error. Statistical analysis was performed using R
244 v.2.15.0 (The R foundation for Statistical Computing). Bartlett's test was used to check the

245 homogeneity of variance, one way ANOVA was carried out and Tukey test was used to determine
246 statistically significant differences among means ($p<0.05$).

247

248 **3. Results and discussion**

249 **3.1 Effect of HPH and US processing on tomato juice physical properties**

250 Fig. 1 shows the macroscopic images of 7.5 °Brix untreated as well as 150 MPa HPH and 30 min US
251 treated tomato juices. Dramatic differences in tomato appearance can be observed between the
252 untreated and treated samples.

253 Tomato juices with 5.0, 7.5 and 10.0 °Brix were subjected to HPH for increasing pressures up to 150
254 MPa or US for increasing time periods up to 30 min, and then undergone to dynamic, small
255 deformation tests to acquire information on structure. Both storage (G') and loss (G'') moduli were
256 frequency dependent and a prevalence of G' over G'' was found, indicating a weak gel-like behaviour
257 of tomato juice (Augusto et al., 2013) (data not shown). The storage modulus and $\tan \delta$ at a constant
258 frequency (0.1 Hz) were used to compare samples subjected to HPH and US processing (Fig. 2). As
259 a rule, the higher G' and the lower $\tan \delta$ the more elastic and solid-like the material. G' and $\tan \delta$
260 values of HPH and US treated tomato juices were respectively always greater and lower than those
261 of the untreated samples ($p<0.05$). This suggests a higher number of elastic interactions in processed
262 tomato juices, which resulted in a stronger structure. The extent of changes in viscoelastic properties
263 increased with the increase in juice concentration. In particular, a significant G' increase was found
264 for the 5.0 and 7.5 °Brix tomato juices subjected to HPH up to 50 MPa, while no further increase in
265 storage modulus was observed at higher pressures. By contrast, G' of the 10.0 °Brix tomato juice
266 increased progressively with the increase of pressure, reaching at 150 MPa 4 times higher values than
267 the untreated sample, in agreement with literature (Augusto et al., 2013). Similarly, the storage
268 modulus and $\tan \delta$ of the 5 min US treated tomato juices, at all concentrations, were respectively
269 higher and lower than those of the untreated samples ($p<0.05$). No significant changes in the

270 viscoelastic properties were observed among samples subjected to increasing US times ($p>0.05$).

271 Tomato juice consistency was also evaluated by Bostwick consistometer, which is a widely used tool

272 for quality control at the industrial level. HPH and US induced a significant increase in juice

273 consistency, in agreement with the data relevant to the storage modulus ($p<0.05$) (Fig. 3). Results

274 suggest that both HPH and US were responsible for modifications in the physical properties of tomato

275 samples, which are attributable to cell rupture. As shown in Fig. 4, HPH caused a progressive cell

276 disruption and no intact cells were found upon 150 MPa, while some intact cells were still present in

277 the 30 min US treated sample. As a consequence of cell rupture, the surface area of the suspended

278 particles increased and tomato constituents were released in the medium. In these conditions, novel

279 inter-particle interactions could have been favoured. These events were accompanied by sample

280 colour bleaching (Table 1). Both technologies induced a significant decrease in tomato juice redness

281 at the lesser pressure or time. By increasing the HPH pressure, the colour fading progressively

282 increased, whereas US treatment times higher than 5 min did not cause further colour modifications.

283 No differences in colour bleaching were found among samples with different solids content. ~~It can be~~

284 ~~inferred that carotenoids were no more protected by cell integrity and underwent to isomerization and~~

285 ~~oxidation due to cell membrane disruption and their dispersion in the medium (Colle et al., 2010). It~~

286 can be inferred that HPH and US by disrupting cell membrane caused carotenoids dispersion in the

287 medium, where they underwent to isomerization and oxidation being not more protected by cell

288 integrity (Colle et al., 2010).

289 To support this hypothesis, the precipitate weight ratio of untreated and treated tomato juices was

290 determined (Table 2). This measure provides an indication of the capacity of the matrix to hold water

291 in the macromolecular network (Colle et al., 2010). The higher the precipitate weight ratio the

292 stronger the network and its water holding capacity. Specifically, increases from 10% to 22% and

293 from 14% to 36% were found for 5.0 and 7.5 °Brix HPH processed tomato juices, respectively. A

294 greater increase up to 46% in the precipitate weight ratio was obtained for the 10.0 °Brix sample

295 subjected to HPH for increasing pressures. Similarly, US treatments caused significant changes of

296 this indicator compared to the untreated samples in all the tomato juices considered. Treatment times
297 higher than 5 min did not induce further changes of precipitate weight ratio, in agreement with the
298 other indexes previously described for the US processed samples.

299 Measurement of the pectin esterification degree of the HPH or US processed tomato juices showed
300 that this parameter did not change in comparison with that of the untreated samples, regardless the
301 tomato juice concentration and intensity of process applied (data not shown). This result is in
302 agreement with the literature in the framework of the effect of HPH and US on pectin molecule
303 (Shpigelman et al., 2015; Anese et al., 2013). Thus, the modifications of colour, rheological properties
304 and water holding capacity of tomato juice upon HPH and US treatments can be mainly attributed to
305 physical events than to chemical ones.

306 Discrepancies in the observed physical properties between samples subjected to HPH and US are
307 likely attributable to differences in modalities of force transmission during processing and thus in
308 mechanical stresses generated by the two technologies. Considering the 5.0 and 7.5 °Brix tomato
309 juices, similar structure modifications were obtained by applying HPH or US, regardless the process
310 intensity. By subjecting to HPH the tomato juice with the highest solids concentration (10.0 °Brix), a
311 dramatic change in the viscoelastic properties was observed. On the contrary, these modifications
312 were not found for the 10.0 °Brix US treated sample. The changes in physical properties of the HPH
313 processed tomato juice suggests that higher particle-particle interactions took place leading to the
314 formation of a stronger network. It can be inferred that the crowding in the homogenization valve
315 increased by increasing the number of suspended material in the sample, thus favouring interactions
316 and, consequently, inducing an increase in consistency as well as gel-like properties. In these
317 conditions the magnitude of mechanical stresses acting on the particles during the flow through the
318 valve could have become much higher, thus increasing the extent of cell disruption. Our findings
319 highlight that stress magnitude in combination with product concentration is crucial for structure
320 development using HPH. Differently, the particle number increase is expected to reduce the efficacy

321 of ultrasonication, because the high initial product consistency could hinder wave propagation
322 (Earnshaw, 1998).

323

324 **3.2 Energy density and electrical energy consumption of HPH and US lab equipment**

325 To estimate HPH and US processes efficiency the energy density and electrical energy consumption
326 were evaluated. Table 3 shows the power density measured at ambient temperature at the beginning
327 of the US treatment, as well as the energy density values for both processes. It appears that the energy
328 density values for the US process were higher than those for the HPH treatment, even if the former
329 are possibly underestimated because of their calculation procedure.

330 Values of the electrical energy consumption for HPH and US devices are summarised in Table 4. As
331 far as energy consumption of HPH is concerned, the electrical energy values were calculated
332 considering that the lab-scale high pressure homogenizer was capable to treat 2.5 cm³/s of product.
333 An almost linear correlation between pressure and both electrical energy and power factor was found.
334 Moreover, sample concentration did not affect these parameters. This result was also confirmed by
335 treating deionised water in the high pressure homogenizer. Energy consumption of the US treatment
336 was estimated by integrating the measurements of the instantaneous electric power supplied during
337 the whole treatment. According to Table 4, also US energy consumption did not change significantly
338 with sample solids concentration.

339 These results show that at laboratory scale the high pressure homogenizer presents quite lower energy
340 consumption than the US device, even if the HPH apparatus employed in this case gave rise to
341 significantly low power factors which call for correction in order to comply with the requirements
342 from the energy supplier.

343

344 **4. Conclusions**

345 Results of this study highlighted the influence of stress type and food solids concentration on the
346 changes in tomato juice physical properties induced by HPH and US processing.

347 From an industrial feasibility perspective, these results provide information useful to select the most
348 appropriate process to steer physical properties of tomato derivatives. For tomato juices with
349 concentration equal or lower than 7.5 °Brix, the choice between HPH and US should not be performed
350 on the basis of the induced structure modifications because both technologies led to comparable
351 effects. In this context, equipment and total ownership costs would drive the choice. Despite the study
352 was performed on lab-scale equipment, the estimated energy density transferred to the juice during
353 processing and the equipment electrical energy consumption here reported can be used to compare
354 HPH and US processes from the point of view of operating costs, being higher those relevant to the
355 US technology.

356 On the contrary, for tomato juices with higher concentration (10.0 °Brix), HPH treatments resulted
357 very effective in changing sample consistency and gel-like properties, in an extent that was not
358 achievable by applying the US process. Thus, the criteria for technology selection should be based
359 on a product perspective rather than on process costs.

360

361 **Acknowledgments**

362 MA and SC conceived the study and carried out the experiments in conjunction with FB and FN.
363 DP carried out the rheological measurements in conjunction with FN. GC carried out energy
364 computations. All authors participated in manuscript revision and discussion, coordinated and
365 critiqued by MA and SC.

366 **Captions for Figures**

367

368 **Fig. 1.** Images of 7.5 °Brix untreated (A), 150 MPa HPH (B) and 30 min US (C) treated tomato juices.

369

370 **Fig. 2.** Storage modulus (G') and $\tan \delta$ at 0.1 Hz of 5.0, 7.5 and 10.0 °Brix tomato juices subjected to
371 high pressure homogenization (HPH) (A, C) and ultrasound (US) (B, D) processes.

372

373 **Fig. 3.** Bostwick consistency of 5.0, 7.5 and 10.0 °Brix tomato juices subjected to high pressure
374 homogenization (HPH) (A) and ultrasound (US) (B) processes.

375 **Fig. 4.** Micrographs of 5.0 °Brix untreated (A), and HPH (B: 20 MPa, C: 50 MPa, D: 100 MPa, E:150
376 MPa) and US (F: 5 min, G: 15 min, H: 30 min) processed tomato juices.

377

378

379 **References**

380 Anese, M., Mirolo, G., Beraldo, P., & Lippe, G. (2013). Effect of ultrasound treatments of tomato
381 pulp on microstructure and lycopene *in vitro* bioaccessibility. *Food Chemistry*, 136, 458-463.

382 Augusto, P.E.D., Ibarz, A., & Cristianini, M. (2012). Effect of high pressure homogenization (HPH)
383 on the rheological properties of tomato juice: Time-dependent and steady-state shear. *Journal*
384 *of Food Engineering*, 111, 570-579.

385 Augusto, P. E. D., Ibarz, A., & Cristianini, M. (2013). Effect of high pressure homogenization (HPH)
386 on the rheological properties of tomato juice: Viscoelastic properties and the Cox–Merz rule.
387 *Journal of Food Engineering*, 114, 57-63.

388 Barbosa-Cánovas, G.V., & Rodriguez J.J. (2002). Update on non-thermal food processing
389 technologies: Pulsed electric field, high hydrostatic pressure, irradiation and ultrasound. *Food*
390 *Australia*, 54, 513-518.

391 Baumann, A.R., Martin, S.E., & Feng, H. (2005). Power ultrasound treatment of *Listeria*
392 *monocytogenes* in apple cider. *Journal of Food Protection*, 68(11), 2333-2340.

393 Bayod, E., Willers, E. P., & Tornberg, E. (2008). Rheological and structural characterization of
394 tomato paste and its influence on the quality of ketchup. *LWT-Food Science and Technology*,
395 41, 1289-1300.

396 Bayod, E., & Tornberg, E. (2011). Microstructure of highly concentrated tomato suspensions on
397 homogenization and subsequent shearing. *Food Research International*, 44, 755-764.

398 Bengtsson, H., & Tornberg, E.V.A. (2011). Physicochemical characterization of fruit and vegetable
399 fiber suspensions. I: effect of homogenization. *Journal of Texture Studies*, 42, 268-280.

400 Bermúdez-Aguirre, D., & Barbosa-Cánovas, G.V. (2012). Inactivation of *Saccharomyces cerevisiae*
401 in pineapple, grape and cranberry juices under pulsed and continuous thermo-sonication
402 treatments. *Journal of Food Engineering*, 108, 383-392

403 Calligaris, S., Foschia, M., Bartolomeoli, I., Maifreni, M., & Manzocco, L. (2012). Study on the
404 applicability of high-pressure homogenization for the production of banana juices. *LWT-Food*
405 *Science and Technology*, 45, 117-121.

406 Calligaris, S., Plazzotta, S., Bot, F., Grasselli, S., Malchiodi, A., & Anese, M. (2016). Nanoemulsion
407 preparation by combining high pressure homogenization and high power ultrasound at low
408 energy densities. *Food Research International*, 83, 25-30.

409 Choi Y., & Okos M.R. (1987). Effects of temperature and composition on the thermal properties of
410 foods. *Food Engineering and Process Applications*, 1, 93-101.

411 Chou, D., & Kokini, J.L. (1987). Rheological properties and conformation of tomato paste pectins,
412 citrus and apple pectins. *Journal of Food Science*, 52, 1658-1664.

413 Colle, I., Van Buggenhout, S., Van Loey, A., & Hendrickx, M. (2010). High pressure homogenization
414 followed by thermal processing of tomato pulp: Influence on microstructure and lycopene in
415 vitro bioaccessibility. *Food Research International*, 43, 2193-2200.

416 Cortés-Muñoz, M., Chevalier-Lucia, D., Dumay, E. (2009). Characteristics of submicron emulsions
417 prepared by ultra-high pressure homogenisation: Effect of chilled or frozen storage. *Food*
418 *Hydrocolloids*, 23, 640-654.

419 Donsì, F., Annunziata, M., & Ferrari, G. (2013). Microbial inactivation by high pressure
420 homogenization: Effect of the disruption valve geometry. *Journal of Food Engineering*, 115,
421 362-370.

422 Earnshaw, R.G. (1998) Ultrasound: A new opportunity for food preservation, In: M.J.W Povey and
423 T.J., Mason (Eds.), *Ultrasound in Food Processing* (pp.183-192). Blackie Academic and
424 Professional, Glasgow, UK.

425 Flourey, J., Bellettre, J., Legrand, J., & Desrumaux, A. (2004a). Analysis of a new type of high pressure
426 homogeniser. A study of the flow pattern. *Chemical Engineering Science*, 59, 843-853.

427 Flourey, J., Legrand, J., & Desrumaux, A. (2004b). Analysis of a new type of high pressure
428 homogeniser. Part B. study of droplet break-up and recoalescence phenomena. *Chemical*
429 *Engineering Science*, 59, 1285-1294.

430 Gould, W.A. (1991). Tomato production, processing and technology (3rd ed.). CTI Publications Inc.,
431 Timonium, ML, USA.

432 Kubo, M.T.K., Augusto, P.E.D., & Cristianini, M. (2013). Effect of high pressure homogenization
433 (HPH) on the physical stability of tomato juice. *Food Research International*, 51, 170-179.

434 Leighton T.G. (1995). Bubble population phenomena in acoustic cavitation. *Ultrasound*
435 *Sonochemistry*, 2, S123–S136.

436 Lopez-Sanchez, P., Nijssse, J., Blonk, H.C.G., Bialek, L., Schumm, S., & Langton, M. (2011a). Effect
437 of mechanical and thermal treatments on the microstructure and rheological properties of
438 carrot, broccoli and tomato dispersions. *Journal of the Science of Food and Agriculture*, 91,
439 207-217.

440 Lopez-Sanchez, P., Svelander, C., Bialek, L., Schumm, S., & Langton, M. (2011b). Rheology and
441 microstructure of carrot and tomato emulsions as a result of high-pressure homogenization
442 conditions. *Journal of Food Science*, 76, E130-E140.

443 Mañas, P., Pagan, R., & Raso, J. (2000). Predicting lethal effect of ultrasonic waves under pressure
444 treatments on *Listeria monocytogenes* ATCC 15313 by power measurements. *Journal of Food*
445 *Science*, 65(4), 663-667.

446 Mason T.J. (1998). Power ultrasound in food processing: The way forward. In M.J.W. Povey, T.J.
447 Mason (Eds.), *Ultrasound in Food Processing* (pp.105-126). Blackie Academic and
448 Professional, Glasgow, UK.

449 Panozzo, A., Lemmens, L., Van Loey, A., Manzocco, L., Nicoli, M. C., & Hendrickx, M. (2013).
450 Microstructure and bioaccessibility of different carotenoid species as affected by high
451 pressure homogenisation: A case study on differently coloured tomatoes. *Food Chemistry*,
452 141, 4094-4100.

453 Rojas, M.L., Leite, T.S., Cristianini, M., Alvim, I.D., & Augusto, P.E.D. (2016). Peach juice
454 processed by the ultrasound technology: Changes in its microstructure improve its physical
455 properties and stability. *Food Research International*, 82, 22-33.

456 Raso, J., Manas, P., Pagan, R., & Sala, F.J. (1999). Influence of different factors on the output power
457 transferred into medium by ultrasound. *Ultrasonics Sonochemistry*, 5, 157-162

458 Seshadri, R., Weiss, J., Hulbert, G.J., & Mount, J. (2003). Ultrasonic processing influences
459 rheological and optical properties of high-methoxyl pectin dispersions. *Food Hydrocolloids*,
460 17, 191-197.

461 Shpigelman, A., Kyomugasho, C., Christiaens, S., Van Loey, A. M., & Hendrickx, M. E. (2015). The
462 effect of high pressure homogenization on pectin: Importance of pectin source and pH. *Food*
463 *Hydrocolloids*, 43, 189-198.

464 Silva, V.M., Sato, A.C.K., Barbosa, G., Dacanal, G., Ciro-Velásquez, H. J., & Cunha, R.L. (2010).
465 The effect of homogenisation on the stability of pineapple pulp. *International Journal of Food*
466 *Science and Technology*, 45, 2127-2133.

467 Stang, M., Schschmann, H., & Schubert, H. (2001). Emulsification in high-pressure homogenizers.
468 *Engineering in Life Science*, 1, 151-157.

469 Tan, J., & Kerr, W. L. (2015). Rheological properties and microstructure of tomato puree subject to
470 continuous high pressure homogenization. *Journal of Food Engineering*, 166, 45-54.

- 471 Thakur, B.R., Singh, R.K., & Handa, A.K. (1995). Effect of homogenization pressure on consistency
472 of tomato juice. *Journal of Food Quality*, 18, 389-396.
- 473 Toma, M., Fukutomi, S. Asakura, Y., Koda, S. (2010). A calorimetric study of energy conversion
474 efficiency of a sonochemical reactor at 500 kHz for organic solvents. *International Journal of*
475 *Food Microbiology*, 137, 116-120.
- 476 Valencia, C., Sánchez, M. C., Ciruelos, A., Latorre, A., Madiedo, J.M., & Gallegos, C. (2003). Non-
477 linear viscoelasticity modeling of tomato paste products. *Food Research International*, 36,
478 911-919.
- 479 Vercet, A., Sánchez, C., Burgos, J., Montanes, L., López-Buesa P. (2002). The effects of
480 manothermosonication on tomato pectin enzymes and tomato paste rheological properties.
481 *Journal of Food Engineering*, 53, 273-278.
- 482 Yu, Z.-Y., Jiang, S.-W., Cao, X.-M., Jiang, S.-T., & Pan, L.-J. (2016). Effect of high pressure
483 homogenization (HPH) on the physical properties of taro (*Colocasia esculenta* (L). Schott)
484 pulp. *Journal of Food Engineering*, 177, 1-8.
- 485 Wu, J., Gamage, T.V., Vilku, K.S., Simons, L.K., & Mawson. R. (2008). Effect of thermosonication
486 on quality improvement of tomato juice. *Innovative Food Science and Emerging*
487 *Technologies*, 9, 186-195.

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490 **Table 1.** Hue angle (arctan b*/a*) of 5.0, 7.5, 10.0 °Brix tomato juices subjected to high pressure
491 homogenization (HPH) and ultrasound (US) processes. Data relevant to untreated samples are also
492 shown.

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Total soluble		HPH				US		
solids content		Pressure (MPa)				Time (min)		
(°Brix)	Untreated	20	50	100	150	5	15	30
5.0	21 ^e	24 ^d	27 ^c	29 ^b	30 ^a	25 ^d	24 ^d	24 ^d
7.5	23 ^c	27 ^b	29 ^a	29 ^a	29 ^a	23 ^c	24 ^c	23 ^c
10.0	22 ^d	23 ^c	24 ^b	26 ^a	26 ^a	23 ^{dc}	22 ^{cd}	22 ^{dc}

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495 a, b, c, d, e: means with different letters in the same row are significantly different (p<0.05)

496 Standard error<1

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499

500 **Table 2.** Precipitate weight ratio (%) of 5.0, 7.5, 10.0 °Brix tomato juice at subjected to high pressure
 501 homogenization (HPH) and ultrasound (US) treatments. Data relevant to untreated samples are also
 502 shown.

Total soluble		HPH				US		
solids content		Pressure (MPa)				Time (min)		
(°Brix)	Untreated	20	50	100	150	5	15	30
5.0	10±2 ^d	14±0 ^{cd}	18±1 ^{bc}	20±2 ^{ab}	22±1 ^a	14±0 ^{bc}	15±0 ^{bc}	17±1 ^{bc}
7.5	14±1 ^f	23±1 ^{cd}	27±1 ^{bc}	30±2 ^b	36±1 ^a	20±0 ^{de}	22±1 ^{ef}	19±1 ^{de}
10.0	19±0 ^e	29±1 ^d	37±1 ^c	42±3 ^{ab}	46±2 ^a	23±0 ^{de}	23±0 ^{de}	22±0 ^{de}

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504 a, b, c, d, e, f: means with different letters in the same row are significantly different (p<0.05)

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507 **Table 3.** Energy density values generated during HPH and US of 5.0, 7.5 and 10.0 °Brix tomato
508 juices, and power density at ambient temperature relevant to the US treatment.

Total soluble solids content (°Brix)	HPH			US	
	Energy density (MJ/m ³)			Power density (kW/m ³)	Energy density (MJ/m ³)
	50 MPa	100 MPa	150 MPa		
5.0	50	100	150	533	612
7.5	50	100	150	916	635
10.0	50	100	150	1316	659

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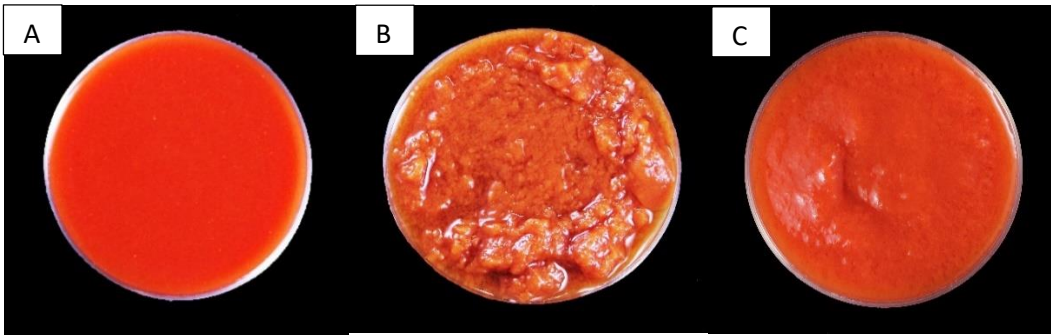
511 **Table 4.** Electrical energy consumption of the HPH and US lab devices, during tomato juice
 512 processing.

Total soluble solids content (°Brix)	HPH						US
	Electrical energy (MJ/m ³)			Power factor (-)			Electrical energy (MJ/m ³)
	50 MPa	100 MPa	150 MPa	50 MPa	100 MPa	150 MPa	
5.0	317	480	644	0.29	0.41	0.51	1369
7.5	310	464	648	0.28	0.40	0.52	1171
10.0	312	462	645	0.28	0.40	0.51	1250

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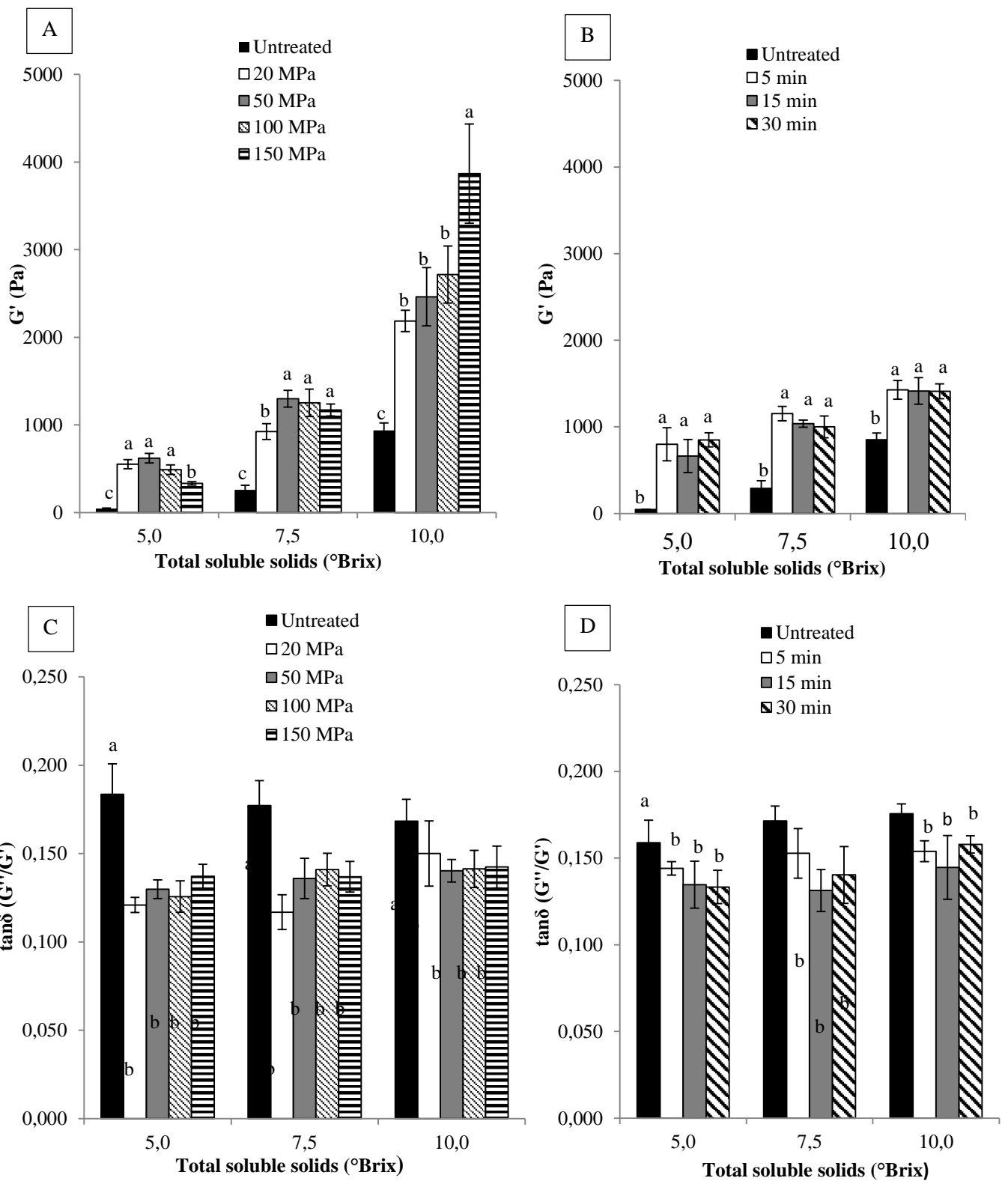


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517 **Fig. 1.** Images of 7.5 °Brix untreated (A) and 150 MPa HPH (B) and 30 min US (C) treated tomato
518 juices.

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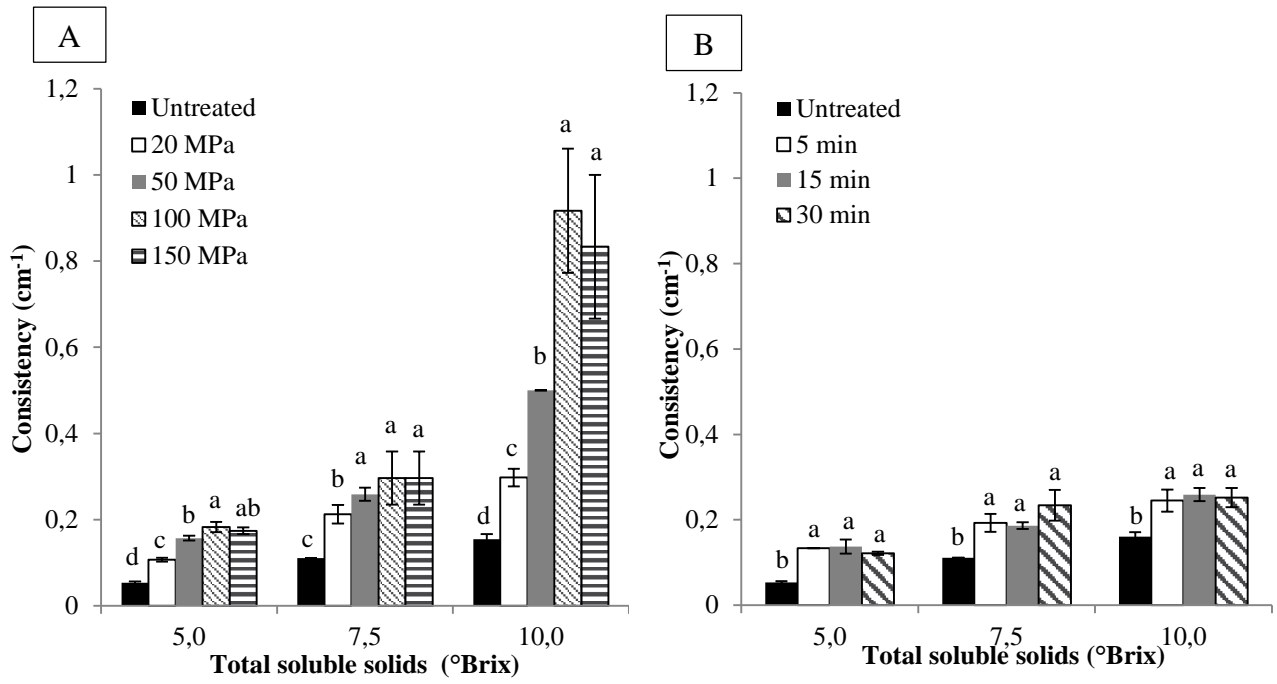
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533 **Fig. 2.** Storage modulus (G') and $\tan \delta$ at 0.1 Hz of 5.0, 7.5, 10.0 °Brix tomato juices subjected to
 534 high pressure homogenization (HPH) (A, C) and ultrasound (US) (B, D) processes.

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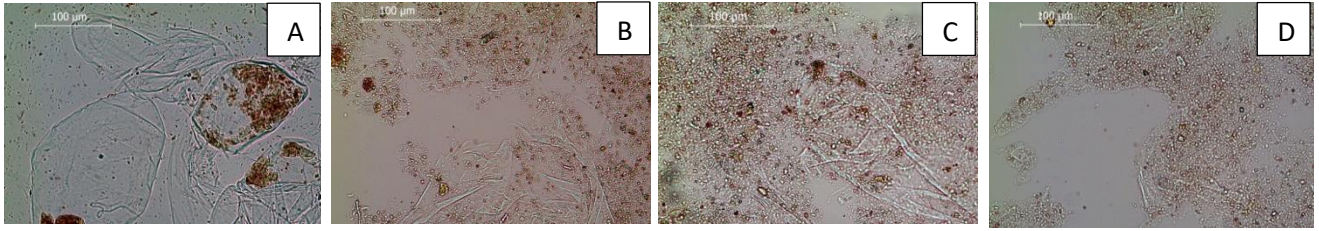


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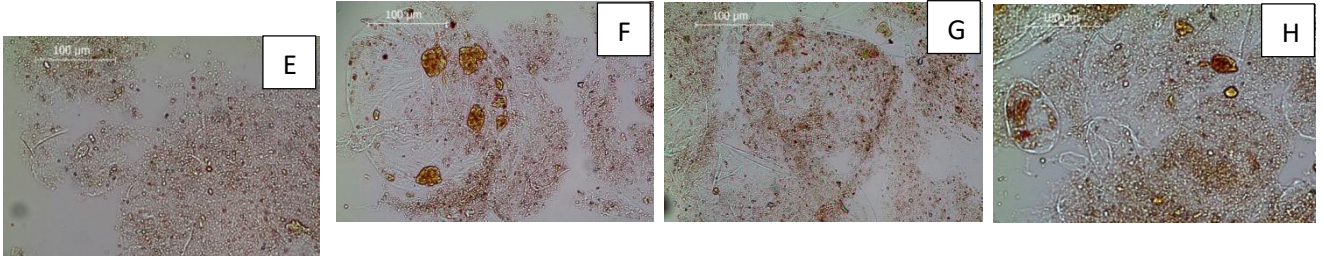
538 **Fig. 3.** Bostwick consistency of 5.0, 7.5, 10.0 °Brix tomato juices subjected to high pressure
 539 homogenization (HPH) (A) and ultrasound (B) processes.

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546 **Fig. 4.** Micrographs of untreated (A) and 20 MPa (B), 50 MPa (C), 100 MPa (D), 150 MPa (E) HPH

547 processed and 5 min (F), 15 min (G), 30 min (H) US processed 5.0 °Brix tomato juices.

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