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Received: 22 March 2022 | Revised: 10 June 2022 | Accepted: 11 August 2022

DOI: 10.1111/jfpp.17049

Journal of Food Processing and Preservation | Institute of Food Science + Technology | ifst | WILEY

ORIGINAL ARTICLE

Effect of high-pressure homogenization on the sensory, nutritional and physical characteristics of mango nectar (*Mangifera indica* L.)

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Abstract

High-pressure homogenization (HPH) was applied within a range of 300 to 1000 bar for 2 to 8 cycles. At constant dry matter (6.2%), pulp mass increased from 3.7 to 8%, indicating a change in particle organization. D_{90} varied from 197 to 20 μm depending on the pressure and number of cycles. The aggregation of particles, then destruction of the aggregates depending on the number of cycles, explained viscosity behavior. HPH delayed phase separation by an average 28.5 h. Sedimentation velocity was 10 times higher at 8 cycles than at 3 cycles, at 1000 bar. At 1000 bar, browning was observed, resulting from Maillard reactions, and was linked to the furan-2-pentyl content (10-fold higher) and a 45% loss of carotenoids. This study will help in understanding changes induced in mango nectar by HPH and will be of use in developing processing technologies to preserve mango-based products.

Novelty impact statement: This study provides some additional scientific indications for understanding the subtility of HPH impacts in stabilizing mango nectar. The optimum pressure treatment for delaying phase separation (1000 bar, 3–6 cycles), i.e., reducing particle size and forming adequate re-aggregation, also causes a Maillard reaction (2-fold color difference, 10-fold furan increase, etc.), hence browning through an increase in local temperature. It will therefore be necessary to find a compromise to limit degradation reactions caused by the process, and to guarantee greater consumer acceptance (phase separation and sensory quality).

1 | INTRODUCTION

Mango (*Mangifera Indica* L.) is a tropical fruit grown in 85 countries and ranks fifth in world fruit production (Marin Castro, Pallet, et al., 2021). Given their perishable nature and limited shelf life, these fruits have high potential for processing and export, as the market for value-added mango products, such as juice, puree and nectar, has steadily grown (Olivier-Simancas et al., 2021). Mango nectar is a popular drink with consumers of all ages, among other things because of its strong aroma, attractive color, pleasant taste and high nutritional value, with significant amounts of ascorbic acid, polyphenols, and carotenoids (Marin Castro, Garcia-Alvarado, et al., 2021; Marin Castro, Pallet, et al., 2021; Yildiz & Aadil, 2022).

High-pressure homogenization (HPH) can be considered one of the most promising non-thermal technologies, because of the recent improvements made in high-pressure homogenizers and increasing

acceptance by consumers of pressure-processed foods. It is particularly suitable for the continuous production of liquid foods, limiting thermal damage and promoting "freshness" (Aadil, 2022; Roobab, Raheem, et al., 2021; Roobab, Shabbir, et al., 2021). The HPH pressures commonly used range between 200 and 500 bar. However, the available homogenizers reach much higher pressures, sometimes up to 4000 bar. Depending on the nominal pressure level, the technology is called either HPH or ultra-high-pressure homogenization (UHPH, up to 3500–4000 bar) (Dumay et al., 2013; Roobab, Afzal, et al., 2022).

HPH has already been studied for fruit juice stabilization. Color change (enzymatic or non-enzymatic browning) has been widely studied. For instance, HPH treatment would seem to preserve the natural color of mango nectar (Uranga-soto et al., 2022). Likewise, the color parameters of apple juice were not significantly affected after 3 cycles at 1500 bar (Maresca et al., 2011), while the color of banana juice (from 1500 to 4000 bar/4°C/1 cycle) and apple juice (3000 bar/4°C, or 20°C/1 cycle) was significantly improved by HPH treatment, with an increase in *L* values and a decrease in *a* values (Calligaris et al., 2012; Saldo et al., 2009). Enzymes responsible for browning can be inactivated by HPH, but the impact of HPH on enzymes has been shown to be unpredictable (Roobab, Abida, et al., 2022). Ahmed et al. (2002) studied the increased browning of mango puree during thermal processing and they reported that it followed first-order reaction kinetics. It has also been reported that high pressures cause an increased browning reaction in some vegetable products (Zhou et al., 2017; Zhu et al., 2022), but Garcia et al. (2001) reported no effect of pressurization on tomato puree pigments. Maresca et al. (2011) and Suárez-Jacobo et al. (2010) found that HPH treatment did not change the pH, total soluble solids (TSS) and titratable acid (TA) in apple juice. Similarly, no significant effect on these parameters was found in mango nectar, even though a slight change was seen during storage (Guan et al., 2016; Zhou et al., 2017). Velázquez-Estrada et al. (2013) reported that HPH treatment (2000 and 3000 bar/6°C/1 cycle) caused a 5% and an 11% reduction in vitamin C in orange juice, respectively, which were lower values than for heat treatment (HT) (90°C/1 min, 17.4% loss). HPH treatment with different pressures and numbers of cycles did not have an impact on vitamin C in orange juice (500–2500 bar/22°C/1 cycle) and apple juice (1000–3000 bar/4°C, or 20°C/1 cycle) (Suárez-Jacobo et al., 2011; Welti-Chanes et al., 2009). Among carotenoids, β-carotene is the major pigment of ripe mango (50–64%) (Marin Castro, Pallet, et al., 2021). Lemmens et al. (2013) showed that the main degradation of β-carotene in mango during HPH was due to isomerization, hence the increase of cis-forms, caused by heat intake. The authors also found that the selection of mango material seemed to have more influence than the process itself. At low temperature, the authors highlighted the negligible modification of β-carotene. Dars et al. (2019) also found that high pressure had no impact on mango juice quality, while Kaushik et al. (2016) proposed optimized HPP conditions at 600 MPa with thermal assistance at 52°C for 10 min for preserving mango pulp quality, taking into account color, ascorbic acid and phenolic differences associated with antioxidant activity.

Lastly, a previous study showed that HPH could modify certain physical characteristics of the fluid, such as rheology, particle size, phase separation and texture uniformity (Augusto et al., 2013; Leite et al., 2015). For example, HPH was successfully applied to reduce the consistency of concentrated orange juice, with a decrease of up to 50% in its apparent viscosity (Leite et al., 2014). Zhou et al. (2017) observed that HPH treatment significantly decreased the particle size of mango nectar by shifting the volume peak from 138 to 6 μm after one cycle from 400 to 1900 bar at 20°C. In a review, Jolvis Pou (2021) found that HPH processing preserved the sensory qualities of food compared to thermal processing, and that HPH might be better than heat treatment for preserving color, flavor and vitamins.

The current work focused on how the level of HPH treatment at pressures of 300 to 1000 bar affected the rheological properties (viscosity, phase separation), carotenoids and sensory qualities of mango nectar. Changes in related factors, including particle size distribution, were also investigated.

2 | MATERIAL AND METHODS

2.1 | Raw material

Fifty-two kg of "Kent, cat. 1" mango (*M. indica* L.), harvested in Peru, were purchased from a local market (Carrefour, Saint Clément de Rivière, France). Before further processing, the mangoes were peeled, pitted, sliced and pureed using a Santos classic 28 extractor (Vaulx-en-Velin, France). The purees were then immediately frozen at −20°C. The mango nectar was produced without sugar, to reach a fruit content of 40%. It was then filtered through a 200 μm snap-ring filter bag (EATON®, Dublin, Ireland). High-pressure homogenization (HPH) was carried out on a bench-scale SPX high-pressure homogenizer, model APV 1000 Lab (SPX FLOW, North Carolina, USA) at pressures ranging from 300 to 1000 bar, with a flow rate of 375 ml/min and a number of cycles ranging from 3 to 8. The fluid was maintained at a temperature of 30°C.

2.2 | Chemicals

All the solvents, reagents and chemical species used for extractions and analyses were ordered from Sigma-Aldrich® (Saint Louis, Missouri, USA).

2.3 | Physico-chemical analysis

The pH and titratable acidity (TA) of mango nectars were measured using an automatic TitroLine® easy titrator (SI Analytics, Mainz, Germany) and expressed as a % of citric acid according to Tharanathan et al. (2006). Pulp mass was determined by the methodology proposed by Cheng et al. (2011). Turbidity was determined after centrifugation of the samples at 10000*g* for 10 min at 20°C

using a HI 98703 turbidimeter (HANNA instruments, Rhode Island, USA) (Servent et al., 2020). Dry matter was measured after drying for 16 h at 50°C and then for 48 h in a vacuum oven at 70°C. Color was evaluated using a Minolta CR-410 chromameter (Konica Minolta, Tokyo, Japon) according to the CIELAB scale. All the analyses described were carried out in triplicate.

2.4 | Particle size distribution (PSD), rheological measurement and phase separation speed

PSD was determined by a Mastersizer 3000 (Malvern Instruments, Malvern, U.K.). Laser light diffraction was used to measure particles from 0.02 to 2000 µm. The area-based mean particle diameter $D_{[3,2]}$ (µm), volume-based mean diameter $D_{[4,3]}$ (µm), D_{90} (µm) and span (PSD extent) were calculated using MasterSizer Software V3.62. The values of the particle refractive index, particle absorption index and dispersant refractive index were 1.73, 0.1 and 1.33, respectively. Each of the 3 replicates was measured 3 times (Zhou et al., 2017).

Viscosity measurements were carried out with a B-one plus viscometer (Lamy Rheology instruments, France) at 25°C in a rotational test with a shear rate of 1, 10 and 100 rpm for 45 s using the Spindle L-1 mobile. The viscosity in mPa·s as a function of shear rate was plotted and the Herschel-Bulkley model (Equation 1) was applied to determine the initial viscosity, consistency and flow indices.

$$y = y_0 + Kx^n \quad (1)$$

where y_0 is the yield stress of the material, factor K is the consistency factor, and factor n is the flow behavior index.

The sedimentation kinetics of the samples were determined using a Turbiscan (Formulaction, Toulouse, France). Particle sedimentation was measured each hour for 6 days at 25°C with a laser (near-infrared light) measuring transmission and backscattering of the light over the entire height of the sample. The light backscattering values after 30 h were compared for all the samples. The lag phase before phase separation, the sedimentation rate (Equation 2), and the sedimentation velocity after the lag phase were then compared.

$$\text{Sedimentation rate} = \frac{V_f}{V_0} \times 100 \quad (2)$$

where V_f is the final volume of sedimentation and V_0 is the initial volume of nectar before decantation.

2.5 | Carotenoid analysis

Carotenoids were extracted by the method of Soto et al. (2021) before HPLC analysis. An HPLC 1100 Series (Agilent Technologies, Santa Clara, USA) equipped with a YMC C_{30} 250 × 4.6 mm × 5 µm column (YMC, Kyoto, Japan) and a refrigerated (4°C) autosampler was used. Twenty µl was injected through the column, which was maintained at a temperature of 30°C. A DAD detector was set at 450 nm. The elution gradient consisted of 3 solvents: H_2O (A), methanol (B) and methyl tert-butyl ether (MTBE, C) at a flow rate of 1 ml/min. The elution gradient was set as follows: 0–2 min, isocratic 40% A – 60% B (initial conditions); 2–5 min, 20% A – 80% B; 5–10 min, 4% A – 81% B – 15% C; 10–60 min, 4% A – 11% B – 85% C; 60–70 min, isocratic 4% A – 11% B – 85% C; 70–71 min, 100% B; 71–72 min, with a return to initial conditions for re-equilibration.

2.6 | Aroma compound analysis

The DHS method was used to extract aroma compounds using a Gerstel autosampler (Gerstel, Mülheim an der Ruhr, Germany). Desorption and analysis were carried out on a GC–MS using an Agilent 7890B GC (Agilent Technologies, Santa Clara, USA). Two grams of nectar was placed in a 10 ml glass vial with 3-heptanol as the internal standard. The sample was equilibrated to 30°C, then the headspace was swept with a nitrogen flow at 1 ml/min stirred at 500 rpm. The volatile compounds were collected on a Tenax TA trap and dried with an additional purge flow at 100 ml/min, at 50°C, for 2.5 min to remove residual water. The collected volatile compounds were then desorbed using an automatic thermal desorption unit (TDU) maintained at 30°C (0.4 min) and then increased to 300°C at 120°C/min. The desorbed compounds were transferred to a CIS4 cooled injection system, in which the compounds were cryofocused. The CIS4 temperature was raised from −10°C to 300°C at 12°C/s and held for 5 min. Volatiles were analyzed on a DB-Wax column (60 m × 250 µm × 0.25 µm). Mass spectra were recorded in EI^+ mode at 70 eV within a range of 40 to 350 Da with a solvent delay time of 2 min and a scan speed of 4.52/s. The analyzer and source temperatures were 150°C and 250°C, respectively. Mass spectrometry data were analyzed using MassHunter software version B.08.00. Volatile compounds were identified by comparing their mass spectra to the NIST 08 library (Wiley, New Jersey, USA). Semi-quantification of the volatile compounds was expressed as the peak area ratio (PAR), which was calculated by dividing the GC peak area by the internal standard peak area.

2.7 | Sensory analysis

A panel trained in descriptive sensory analysis established the sensory profile of four mango nectars. The panel was composed of four women and eight men between 23 and 50 years old, each of them a member of CIRAD's internal jury, trained and validated throughout the year. Before the evaluation, two calibration sessions were carried out and the sensory lexicon was discussed, re-evaluated based on another mango-based product recently tasted by the panel members, and modified with the help of sensory references. During the evaluation, the samples were presented in random order in translucent cups, bearing a three-digit code. The tastings took place in

individual booths under white light. The sensory profiles of the nectars were established by each panelist ($n = 12$) in duplicate. During the evaluation, a 15 min rest was imposed between the two sets of 4 samples, and the panelists were instructed to cleanse their palate with water between tastings. All samples were kept at 4°C until the day of the evaluation. Panel scores on a scale of 0–10 were then reviewed to assess the repeatability of each judge and agreement within the panel.

The sensory lexicon used in this study included 13 sensory descriptors: overall smell, mango smell, color, flavors (sweet and sour), persistence in the mouth, thickness, overall aroma and mango aroma, fruity, spicy and green flavors, and finally the overall quality of the product.

2.8 | Statistical analyses

All assays were carried out in triplicate and the statistical analysis was done using XLSTAT v.2019 (Addinsoft, Paris, France). An analysis of variance followed by Tukey's range test was conducted at $p < .05$, ($n = 3$). The results are presented as means ± standard deviations (SDs). A principal component analysis (PCA) was carried out to determine the position of samples on the map of sensory profiles and volatile aroma compounds.

3 | RESULTS AND DISCUSSION

HPH did not have a significant impact on pH and TA values. Overall, the average pH was 4.75 (0.14) and TA was 0.09 (0.01) g $eq_{citric\ acid}$/100 g. Similar results have been found in mango or apple juices (Kaushik et al., 2014; Suárez-Jacobo et al., 2010).

The nectar color analysis showed that the HPH process led to significant changes, as shown in Table 1. Compared to the control, the *L* and *b* parameters increased, while the *a* values decreased in the HPH sample when the pressure and number of cycles increased. Considering the increase in the *L* parameter, the HPH treatment resulted in a brighter mango nectar. On the other hand, the *b* increase was correlated to a yellower nectar than the control. This quality improvement was attributed to the reduced activity of browning enzymes caused by HPH (Roobab, Abida, et al., 2022; Saldo et al., 2009). However, the higher the pressure was (from 600 to 1000 bar), the higher was the Δ*E* value after HPH treatment (from 1.35 to 2.55). This rise in Δ*E*, that is, browning with also a decrease in *a* hue, might mainly be attributed to the Maillard reaction induced by the shear stress at the HPH restriction zone, causing a local increase in temperature (Suárez-Jacobo et al., 2012). On the other hand, the number of cycles also affected color. This complex behavior in color, a combination of enzymatic and non-enzymatic changes, has already been studied. Zhou et al. (2017) reported that it can be more specifically due to oxidation throughout the process. The influence of the number of cycles can be explained by a larger amount of dissolved oxygen, with a longer treatment time inducing Maillard browning reactions (Zhou et al., 2017). Similar results were reported by Arroyo et al. (1997), who found that browning occurred after HP treatment of some vegetable products, sometimes making them unacceptable to consumers. Consumer trends today show strong demand for safe and high-quality products, with a taste, texture and color similar to a fresh product (Tribst et al., 2009). Using HPH on an industrial scale raises the issue of preserving the fresh product color to satisfy consumers.

Dry matter was constant whatever the HPH treatment, with an average value of 6.2 (0.3) g/100 g. As presented in Figure 1, nectar turbidity and mass pulp showed significant differences when

TABLE 1 Effect of HPH on the color parameters of mango nectar

Pressure (Bar)	Number of cycles (c)	*L**	*a**	*b**	Δ*E*
0	0	34.5 (0.3)^{d}	0.1 (0.3)^{a}	20.5 (0.7)^{cd}	0.0
300	3	34.6 (0.2)^{d}	0.1 (0.2)^{a}	20.1 (0.4)^{cd}	0.6 (0.3)^{de}
	6	34.7 (0.3)^{cd}	0.1 (0.4) ^{a}	20.2 (0.5)^{cd}	0.8 (0.2)^{de}
	8	34.1 (0.4)^{d}	−0.1 (0.2)^{a}	18.9 (0.9)^{d}	1.7 (1)^{bcd}
600	3	35.7 (0.2)^{b}	0.3 (0)^{a}	21.2 (0.2)^{c}	1.4 (0.3)^{cd}
	6	35.4 (0.3)^{bc}	0.4 (0.2)^{a}	20.5 (0.5)^{cd}	1.1 (0.4)^{de}
	8	35.8 (0.1)^{b}	0.1 (0.1)^{a}	20.8 (0.1)^{c}	1.3 (0.1)^{cd}
800	3	39.6 (0.6)^{a}	−1.5 (0.4)^{b}	27.5 (1.1)^{a}	2.5 (0.8)^{abc}
	6	39.3 (0.1)^{a}	−2.2 (0.1)^{c}	26.7 (0.3)^{ab}	2.4 (0.1)^{abc}
	8	39.0 (0.1)^{a}	−2.2 (0.1)^{c}	25.5 (0.7)^{b}	2.5 (0.4)^{abc}
1000	3	39.2 (0.3)^{a}	−2.3 (0.2)^{c}	26.7 (0.3)^{ab}	2.4 (0.1)^{abc}
	6	39.5 (0.2)^{a}	−2.4 (0.2)^{c}	26.6 (0.4)^{ab}	2.7 (0.1)^{ab}
	8	39.5 (0.1)^{a}	−2.8 (0.1)^{c}	26.1 (0.1)^{ab}	2.9 (0.0)^{a}

Note: Different letters in the same column represent a significant difference (ANOVA, Tukey, $p < .05$).

the pressure increased. For the treatment at 300 bar turbidity was similar to the untreated nectar with 471 (39) NTU, while it showed a significant increase after 800 bar, with more than 569 (15) NTU. Nectar pulp mass values were more impacted by pressure than by the number of cycles. Pulp mass was at least two-fold higher than the control after all HPH treatments, with values of 3.7 (0.3), 8.9 (0.4), 8.1 (0.2) and 7.9 (0.5) g/100 g for the control, 300, 800 and 1000 bar samples, respectively. This behavior associated with a constant dry matter could be explained by the development of a hydrocolloid network. Indeed, Van Buggenhout et al. (2015) highlighted that HPH converted suspended particles into colloidal particles by enhancing interaction between pectin functional groups and water molecules, thereby increasing the water-holding and swelling capacity of pectin. Moreover, homogenization caused gradual changes in the structure of pectic polymers, which became less tightly bound to cell walls. Consequently, suspended pectic polymers increase in the nectar and the turbidity increases (Van Buggenhout et al., 2015).

The amount of total carotenoids in the mango nectar control sample was 4.5 $\times 10^{-3}$ (0.4 $\times 10^{-3}$) mg/g. β-carotene accounted for 25% of the total carotenoids with 1.1 $\times 10^{-3}$ (0.1 $\times 10^{-3}$) mg/g and violaxanthin dibutyrate was the second most abundant carotenoid with 6.8 $\times 10^{-4}$ (1.7 $\times 10^{-4}$) mg/g. Chen et al. (2004), found similar results with mango pulp, which contained a range of 0.9–9.2 $\times 10^{-3}$ mg/g of total carotenoids. In their case, all-*trans*-β-carotene was the main compound (29.3 $\times 10^{-3}$ mg/g), followed by *cis* isomers of β-carotene (9.9 $\times 10^{-3}$ mg/g), violaxanthin and its *cis* isomers (6.4 $\times 10^{-3}$ mg/g). In most cases, cis-isomer carotenoids are generated after prolonged exposure to heat.

The total carotenoid content decreased with the number of cycles, with a 15–20% loss between 3 and 8 cycles at pressures up to 800 bar and a 45% loss at 1000 bar (Figure 2). This carotenoid

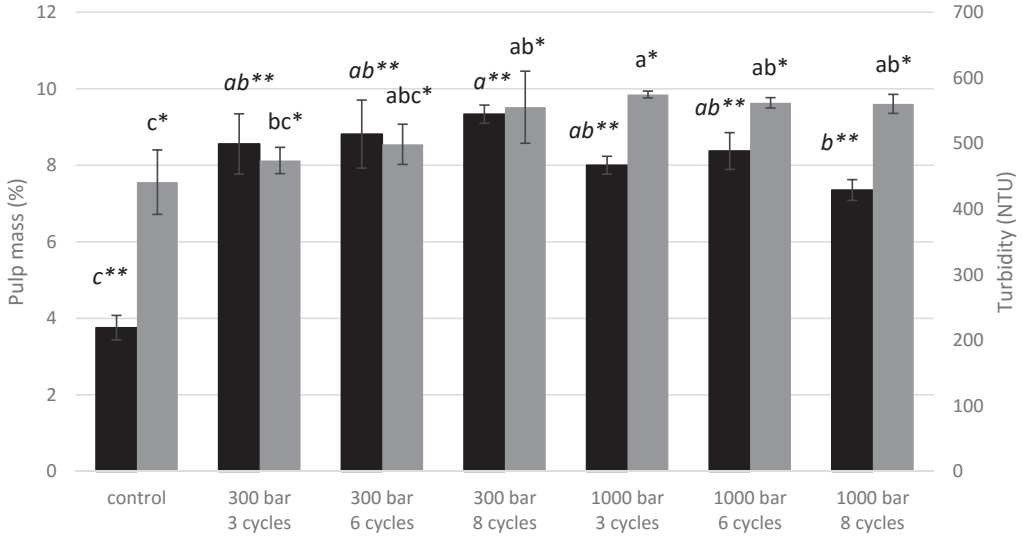

FIGURE 1 Nectar pulp mass (■ on the right) and turbidity (■ on the left) of mango puree treated at 300 and 1000 bar by HPH with 3, 6 or 8 cycles. Different letters represent a significant difference (ANOVA, Tukey, $p < .05$)

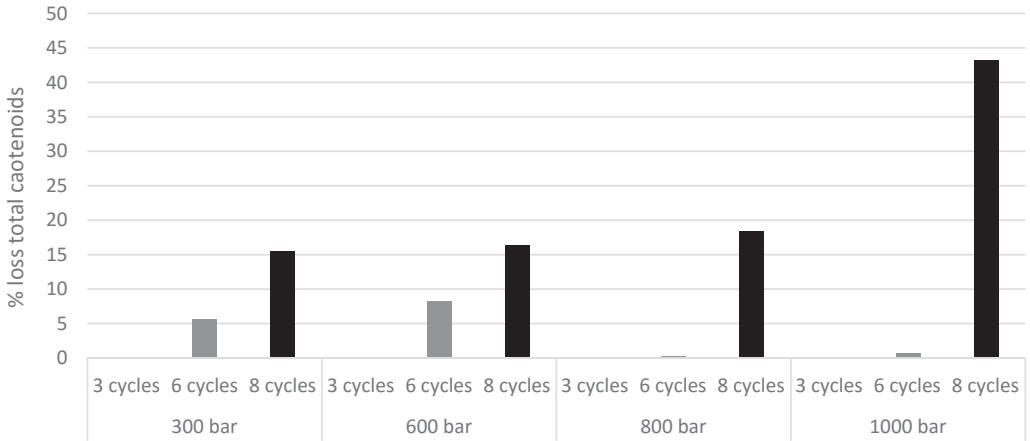

FIGURE 2 Percentage loss of total carotenoids for HPH samples treated at 300, 600, 800 and 1000 bar depending on the number of cycles

shedding can be linked to the color parameter, which decreased, and the clarity of the nectar, which increased with the pressure and number of cycles. On the other hand, HPH did not influence the carotenoid profile. The violaxanthin/β-carotene ratio was constant. These compounds may undergo oxidation processes, which can be stimulated by many factors, such as lighting, heating, or the presence of metals, enzymes, peroxides, etc., the last being the main causes of carotenoid degradation described in the literature (Velázquez-Estrada et al., 2013). In this case, oxygenation due to the process time and nectar recycling might be the main causes of carotenoid degradation. Although the primary requirement for consumers remains taste, they are increasingly focusing on the health benefits of the products they consume. Another specific study should therefore be conducted to determine nutrient bioavailability via in vitro digestion or in vivo assays (Soto et al., 2021).

As expected, HPH significantly decreased the diameter of mango nectar particles, with the volume peak of PSD shifting to 10 μm after 3 HPH cycles at 800 and 1000 bar (Figure 3). The D_{90} value was at least 6 times higher for the untreated sample compared to a sample treated at 300 bar for 8 cycles, indicating that a significant reduction in the larger particles could be achieved by HPH. In addition, a distribution peak around 100 μm was often observed, mostly depending on the number of cycles, with a maximum volume density at 6 cycles for all samples. This phenomenon, related to the influence of the number of cycles on the particle status of nectar, was already observed by Salehi (2020). A small number of cycles can reduce particle size, while an increase in processing time could cause the re-aggregation of these new small particles. For more than 6 or 8 treatment cycles, these aggregates seemed to be broken down again into 10 μm particles. The D_{90} and Span (i.e., dispersion of the particle size distribution) values shown in Table 2 confirmed this phenomenon of aggregation, with significantly higher values for 6-cycle treatments. The D_{90} value varied between 160 and 195 μm for the 6-cycle treatments, depending on the pressure applied, and decreased to values between 20 and 30 μm for the 8-cycle treatments. Similarly, Span values were divided by 4 between samples treated for 6 cycles and 8 cycles.

The viscosity of all the treated nectars decreased with the shear rate increase, from [2500–3000] mPa.s at 1 rpm to approximately 10 mPa.s at 100 rpm (data not shown). Nectars exhibited a non-Newtonian behavior, characteristic of a pseudo-plastic fluid. Similar findings have already been reported for mango products (Ahmed et al., 2005; Liu et al., 2013). This behavior is typical of the formation of a specific layer arrangement that decreases viscosity by lowering dissipation due to collisions between particles (Brader, 2010; Cheng et al., 2011). However, the initial viscosity at 1 rpm (Table 2)

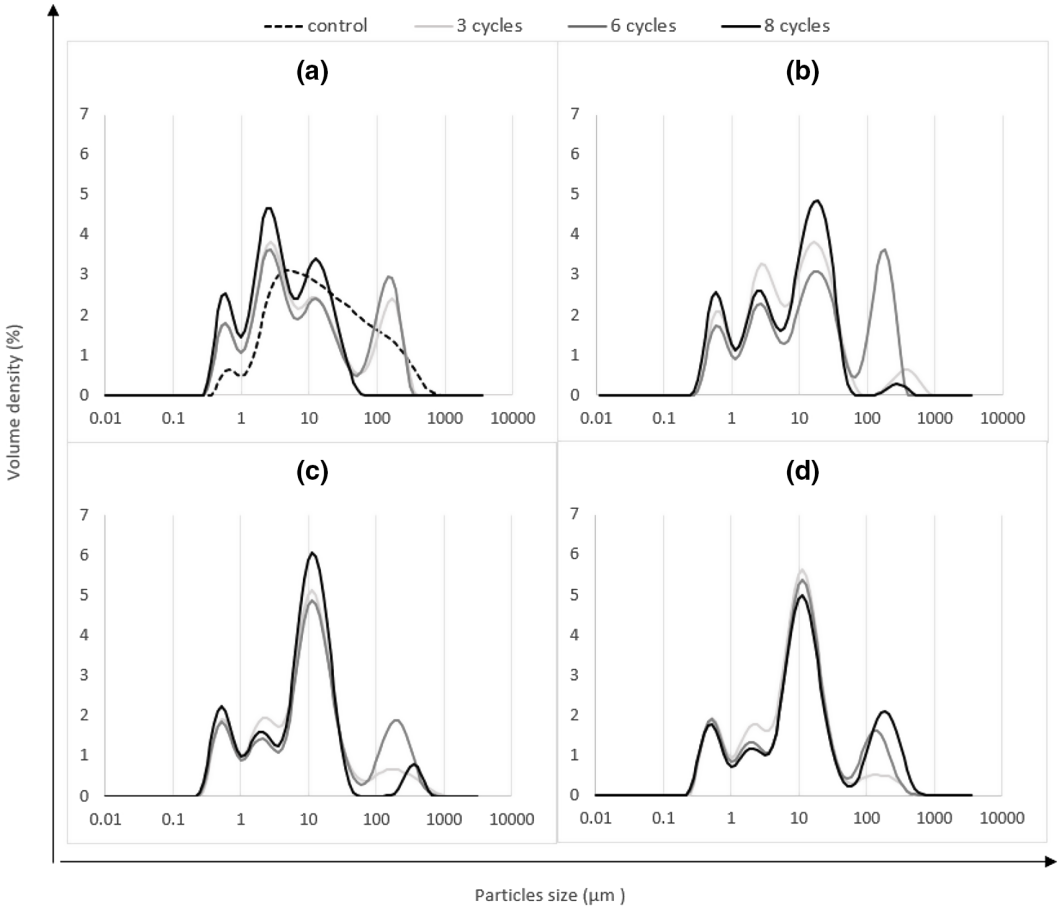

FIGURE 3 Particle size distribution (PSD) of mango nectar treated with HPH, volume density versus particle size (log scale) in μm after 3, 6 and 8 cycles at: (a) 300 bar, (b) 600 bar, (c) 800 bar, (d) 1000 bar

TABLE 2 Viscosity, span, D_{90} and sedimentation parameters of mango nectar depending on the HPH treatment

Pressure (Bar)	Cycles	Viscosity (mPa·s)	D_{90} (µm)	Span	Lag phase (h)	Sedimentation velocity (ml·h^{-1})	Sedimentation rate (%)
0	0	2217 (21)^{abc}	146.3 (19.6)^{bc}	10.7 (1.0)^{bcd}	2	0.32	14.7
300	3	2520 (218)^{bc}	162.5 (11.9)^{abc}	24.8 (1.2)^{a}	32	0.01	30.7
	6	2857 (197)^{bc}	165.3 (11.6)^{ab}	21.6 (2.3)^{a}	30	0.01	35.3
	8	2820 (330)^{bc}	20.4 (0.3)^{d}	5.3 (0.1)^{cd}	28	0.01	21.2
600	3	1303 (74)^{bc}	43.8 (19.9)^{d}	6.1 (1.9)^{cd}	24	0.06	22.0
	6	2547 (442)^{cd}	181.2 (14.3)^{ab}	12.1 (0.9)^{bc}	27	0.06	23.3
	8	915 (124)^{de}	27.9 (4.1)^{d}	3.3 (0.2)^{d}	48	0.06	30.7
800	3	2043 (497)^{abc}	33.4 (2.3)^{d}	3.4 (0.2)^{bcd}	24	0.04	38.2
	6	3407 (389)^{a}	192.7 (36.6)^{a}	16.3 (3.8)^{ab}	23	0.07	40.0
	8	2207 (403)^{e}	25.6 (2.7)^{d}	2.7 (0.2)^{cd}	22	0.12	38.7
1000	3	2404 (162)^{abc}	31.5 (3.6)^{d}	3.3 (0.3)^{d}	31	0.03	38.7
	6	2937 (172)^{bc}	118 (25.6)^{c}	10.7 (1.9)^{bcd}	22	0.10	39.3
	8	2707 (145)^{ab}	197.2 (62.6)^{a}	16.5 (3.8)^{ab}	32	0.31	39.3

Note: Different letters in the same column represent a significant difference (ANOVA, Tukey, $p < .05$).

of HPH-treated mango nectars was constantly higher after 6 cycles (between 1.2 and 1.9-fold) than for the other treatments. This difference could be related to the results observed for the PSD, where the volume density of particles at 100 µm increased between 3 and 6 cycles and decreased after 6 cycles. Aggregation of the particles, then destruction of the aggregates depending on the number of cycles, thus explained viscosity behavior. Samples with the largest populations of 100 µm displayed higher viscosity, which confirmed that particle aggregation started from a treatment time equivalent to 6 cycles (Figure 3). Furthermore, all viscosities tended towards a maximum value of 10 mPa.s with the increase in shear stress, suggesting that aggregates can deform in the direction of stress. This result adds value to the effect of HPH on mango nectar rheological properties.

As shown in Table 2, phase separation was observed for the control sample from the first couple of hours, while the HPH-treated samples did not show any phase separation before 22 h. The most delayed phase shift (48 h) was observed for the sample treated at 600 bar for 8 cycles. HPH was responsible for an average delay of 28.5 h, preventing phase separation of mango nectar. Industrially, it would be advantageous to optimize this process to delay the phase shift as much as possible, to make nectars homogeneous and attractive at the time of purchase. The sedimentation velocity of spheres, as a function of particle properties in a dispersed medium, is governed by Stokes' law. According to this principle, serum viscosity is inversely proportional to sedimentation (Saricaoglu et al., 2019). All nectar samples should have a similar serum viscosity and smaller particle sizes caused by HPH could have led to greater stability. Moreover, increasing water retention by pectic hydrocolloids could have delayed particle sedimentation by increasing nectar viscosity. Similar results were reported by Kubo et al. (2013) for tomato juice and by Betoret et al. (2009) for citrus juices. Silva et al. (2010), for their part, found that HPH-treated pineapple pulp showed sedimentation after 10 days of storage. On the one hand, van der Waals's attraction between smaller particles affected the narrowest population, while larger particles were only affected by hydrodynamic forces. Sedimentation velocity after the lag phase was measured by the slope of the curve of the percentage of retrodiffusion over time at 90% of the final sedimentation volume height (Table 2). It was found that sedimentation velocity after the lag phase for treated samples was lower than that of the control, but increased with the pressure and number of cycles. The kinetic speed was multiplied by 30 between a treatment at 300 bar for 8 cycles and a treatment at 1000 bar for 8 cycles. Moreover, the number of cycles also impacted sedimentation velocity, being 10 times higher after HPH at 1000 bar for 8 cycles compared to a treatment at 1000 bar for 3 cycles. This observation could be explained by the quantity of small and homogenous particles that would simultaneously sediment in HPH samples. The sedimentation rate represented the percentage of the total volume sample that had settled. As shown in Table 2, the sedimentation rates of the treated samples were at least twice as high as that of the control, except for the 600 bar samples. These variations, observed for an identical dry matter, were closely related to the volume of pulp present in the sample (Figure 4). Indeed, as the mass of pulp increased, the sedimentation rate increased with pressure and stabilized for pressures higher than 800 bar. This could also be correlated to the pectic network operating with the HPH treatment.

Considering previous quality observations, it was expected that sensory analysis differences might exist, but be slight. Consequently, only four samples were analyzed by sensory analysis: the control and HPH samples after 6 cycles at 300, 600 and 1000 bar, in order to maintain the panelists' capacity to distinguish small variations. The analyses are described in Table 3. These profiles highlighted that the sensory qualities of the nectar were not impacted by the HPH process. The only exception was the case of color, for which the judges perceived a significant difference. Indeed, according to the panel scores, nectars treated at a pressure higher than 600 bar displayed a browner tone (score: 6.6) than that of the other two nectars (score 4.4). Forty volatile compounds from mango nectar were identified by comparing

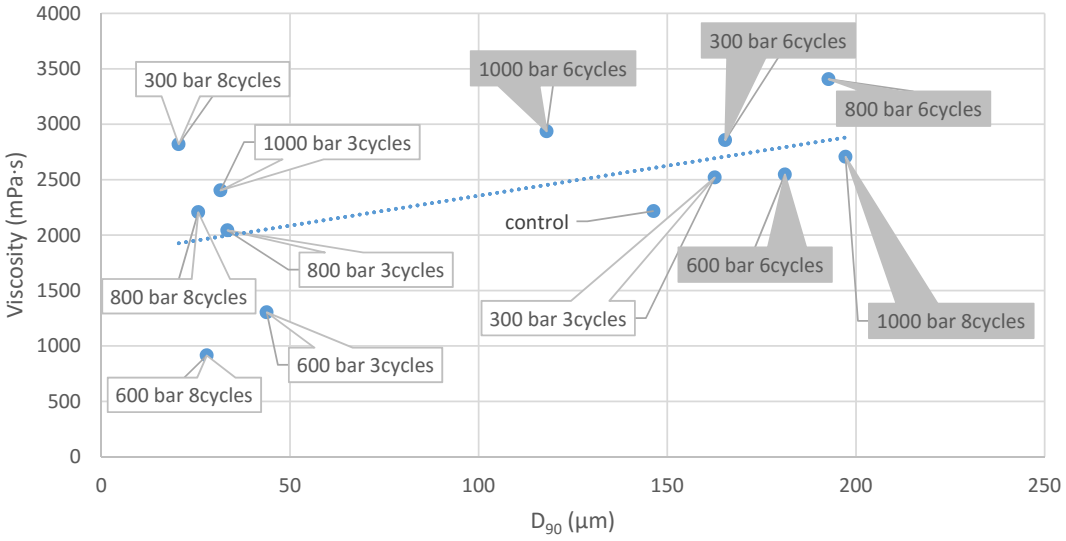

FIGURE 4 Correlation between viscosity and D_{90} of mango nectar depending on the HPH treatment

TABLE 3 Sensory profiles of 4 mango nectar samples: Control and HPH-processed nectars for 6 cycles at 300, 600 and 1000 bar

Pressure (Bar)	0	300	600	1000
Overall odor	5.7	5.4	5.7	5.6
Mango odor	4.1	3.7	4.3	4.2
Color	4.4	5.4	6.5	6.6
Sweet taste	4.4	4.5	4.9	4.1
Sour taste	4.4	4.0	4.1	3.6
Persistence	4.2	4.1	4.5	4.1
Thickness	3.3	3.7	3.9	3.3
Overall aroma	6.1	5.7	5.8	5.4
Mango aroma	4.8	4.7	4.4	4.5
Fruity aroma	3.3	3.2	3.1	3.0
Spicy aroma	0.7	0.6	0.4	0.5
Green aroma	2.0	1.6	1.8	1.6
Overall quality	5.5	5.5	4.9	5.1

their mass fragmentation patterns with those stored in the NIST database. Seventeen of the most discriminating compounds were listed and semi-quantified by relative peak areas of total ion chromatography (TIC) from the MS signals divided by the internal standard peak area (supplementary file). In Figure 5, the PCA based on the main two principal components (F1 & F2) accounting for more than 69% of the variation, shows that 3 groups of volatile compounds discriminated the HPH process: alcohols, terpenes and furanoids. Alcohols, mainly 1-Octanol and 1-Decanol, were present in larger quantities in mango nectars having undergone HPH treatments at pressures lower than 600 bar and fewer than 6 cycles. HPH might enhance the evaporation or the degradation of these compounds, but no change in aromatic perception was detected by sensorial analysis. Terpenic compounds discriminated samples with treatments at 800 bar, especially Δ-3-carene, which is one of the main aromatic compounds of mango odor (Bonneau et al., 2017). Lastly, compounds characteristic of degradation reactions, such as the Maillard reaction (enzymatic oxidative reactions from fatty acids), such as furan-2-pentyl and pentadecanal, distinguished samples which underwent HPH treatment. The presence of furans indicated that the local heat increase during the process can cause a Maillard reaction to develop. The measurement of furanoids can be an indicator of the Maillard reaction, linked to brown pigment formation. Figure 5 shows that the abundance of these reaction indicators increased proportionately with the pressure and was maximum for treatments at 1000 bar. Color changes can be associated with friction force in the homogenizer during high-pressure treatment. These results were confirmed by the panel who found browning of the nectar for the samples treated at 600 and 1000 bar for 6 cycles. This was also in agreement with the study of volatile compounds since a 10-fold increase in the furanoic compound content was observed with the increase in pressure. It can be assumed that nectar browning results from Maillard reactions caused by heating during homogenization at pressures above 800 bar. The appearance of aromatic degradation compounds derived from hydroxymethylfurfural, and arising from the Amadori and Heyns rearrangements during Maillard reactions (i.e., pyrrazine, furfural etc.), remains a problem on an industrial scale, since it leads to a decrease in the sensorial qualities of nectar (Tessier & Birlouez-Aragon, 2012). Differentiating between browning from an enzymatic or non-enzymatic (Maillard, caramelization) reaction is hard during process transformations, since both reactions can occur. In our case, HPH on mango nectar, most of the process conditions tested allowed sample browning to be avoided and when it did occur, it was correlated to furan appearance. Therefore, at the higher pressure studied in this work, the local temperature might be sufficient to generate non-enzymatic browning, even when cooled and maintained at 30°C. It will therefore be necessary to find a compromise to limit degradation reactions due to the process, and to guarantee greater acceptance by consumers.

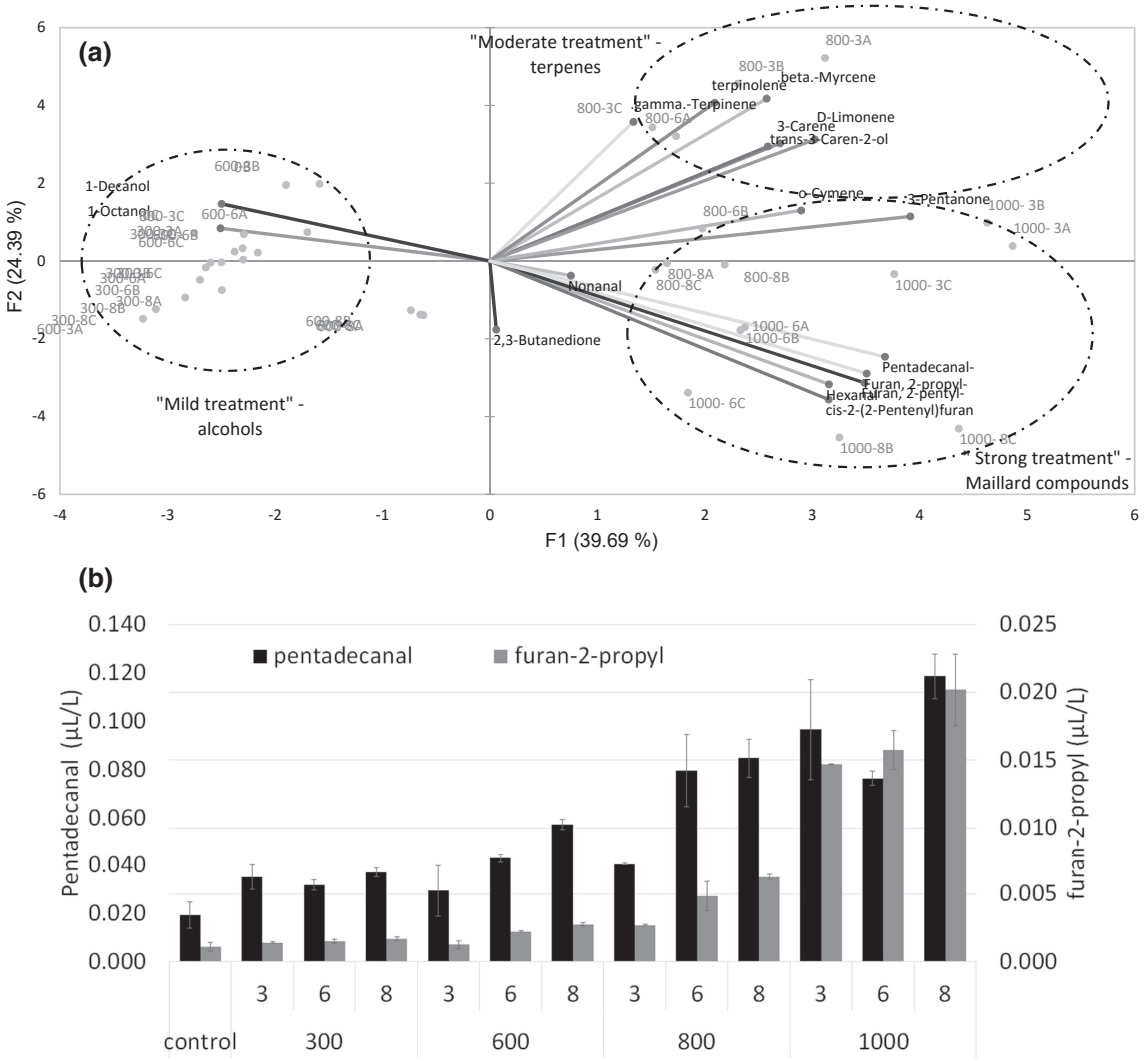

FIGURE 5 Principal component analysis of volatile aroma compounds identified in mango nectar (a), along with furan, 2-pentyl (right scale) and pentadecanal (left scale) contents (b) depending on the HPH process

4 | CONCLUSIONS

This work studied how the HPH process affects mango nectar in a pressure range rarely studied in the literature. The process helps delay mango nectar phase separation by at least 22 h. Treatment at 600 bar for 8 cycles delayed phase separation by 48 h, without any perceptible impact on the sensory qualities for consumers. The aggregation of particles, then the destruction of the aggregates depending on the number of cycles, explained the evolution of rheological measurements and helped in understanding that the best compromise might be in 3–6 cycles, also reducing the process time. Despite maintaining a temperature of 30°C during higher pressure treatments (800 and 1000 bar), browning of the nectar (formation of undesired compounds resulting from Maillard reactions) was observed. At 1000 bar, there was a 10-fold increase in furanoids associated with a 45% loss of bioactive compounds. This work, which is useful for industrial application, calls for a further in-depth investigation to find an optimum treatment to delay phase separation without added products, while preserving the nutritional and sensory qualities of mango nectar. Moreover, the bioaccessibility and the bioavailability of carotenoids with optimized HPH conditions should also be investigated in a specific study.

AUTHOR CONTRIBUTIONS

Victoria Joly: Writing - original draft; Methodology; Formal analysis. **Pierre Brat**: Funding acquisition; Writing - review & editing; Supervision; Methodology; Project administration. **Michael Nigen**: Methodology; Formal analysis; Investigation; Writing - review & editing. **Marc Lebrun**: Formal analysis; Methodology; Validation; Writing - review & editing. **Isabelle Maraval**: Formal analysis; Methodology; Investigation; Supervision; Writing - review & editing. **Julien Ricci**: Methodology; Investigation; Writing - review & editing; Formal analysis. **Nelly Forestier-Chiron**: Formal analysis; Investigation; Methodology. **Adrien Servent**: Methodology; Writing - review & editing; Writing - original draft; Formal analysis.

ACKNOWLEDGMENT

The authors are thankful to the QualiSud joint research unit for providing the necessary funds for this study and to Mr. Jean-Paul Danflous for his technical assistance.

CONFLICT OF INTEREST

The authors have declared no conflicts of interest for this article.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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How to cite this article: Joly, V., Brat, P., Nigen, M., Lebrun, M., Maraval, I., Ricci, J., Forestier-Chiron, N., & Servent, A. (2022). Effect of high-pressure homogenization on the sensory, nutritional and physical characteristics of mango nectar (*Mangifera indica* L.). *Journal of Food Processing and Preservation*, *00*, e17049. https://doi.org/10.1111/jfpp.17049