

ORIGINAL ARTICLE

Setting up a diagram process for the elaboration of a new plant-based beverage from *Pinus halepensis* seeds: Selection of unit operations and their conditions

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Abstract

Aleppo pine seeds are a potential candidate for the production of nondairy milk due to their high protein (26%) and lipid (37%) content and their availability. For the first time, this study presents the implementation of a manufacturing scheme from seeds to final product, including pre-extraction, extraction, and post-extraction (physical and microbiological stabilization), using a $2^{(7-4)}$ fractional factorial screening design. The seven factors were seed soaking time, grinding time, temperature and seed/water ratio during extraction, xanthan gum concentration, homogenization pressure, and pasteurization temperature. Beverage quality analysis consisted of measurements of chemical composition, color, stability, and rheological behavior. The results of experimental design showed that the dry matter as well as the lipid and protein contents increased significantly by increasing the ratio (grain/water) and the grinding time. On the other hand, the extraction temperature led to a better extraction of lipids but a lower extraction of proteins. Regarding stability, xanthan gum had a negative effect, while homogenization pressure and pasteurization temperature showed their effectiveness in decreasing the particle size and increasing the physical stability of the product.

Practical applications

Plant-based drinks, also called plant milks, are interesting alternatives to animal milk. They have similar organoleptic properties but are more sustainable and offer very different nutritional composition depending on the plant used. As the consumers demand is constantly increasing, setting up fabrication processes from new plant sources is relevant. Aleppo pine seeds are a potential matrix for the production of vegetable beverages because they are very nutritious and available in the entire Mediterranean area. This study presents the setting up of a fabrication diagram of a plant-based beverage made from Aleppo pine seeds. Seven production parameters were tested, including pre-extraction (seed conditioning), extraction, and post-extraction (physical and microbiological stabilization), thanks to a fractional factorial $2^{(7-4)}$ screening design. The best conditions enabled the production of a final beverage comparable to other commercialized plant-based beverages in terms of nutritional and organoleptic quality.

1 | INTRODUCTION

Animal milk has always been described as an essential and mandatory food for its high protein, fat, and mineral content (Mesfine, Feyera, & Mohammed, 2015). However, plant-based beverages commonly known as nondairy milk or plant milk are increasingly finding their way onto the market. Sales of these products have grown by approximately 61% since 2012 (Lopes, Duarte, Nunes, Raymundo, & Sousa, 2019). This growth is due to the various nutritional benefits these products can offer. Indeed, aside their diverse tastes, plant-based beverages can have interesting contents in carbohydrates and proteins associated to essential fatty acids and can contribute to diabetes control, cholesterol reduction and cardiovascular disease prevention. That is why they are more and more consumed as an alternative to animal milk by people with lactose intolerance, vegetarians, and vegans and also, by health and environmentally conscious consumers (Kurajdova & Tábořecká-Petrovicova, 2015; Lopes et al., 2019).

The main sources of these beverages are almond, soy, and coconut with market shares of 64, 13, and 12%, respectively. But given the enthusiasm of consumers to change and diversify beverage consumption, new types of vegetable drinks have appeared and have experienced a rapid growth in popularity, for example, pecan, quinoa, hazelnut, and flax based beverages (Kurajdova & Tábořecká-Petrovicova, 2015).

For this reason, researchers and industries are constantly looking for new plant-based sources that could be suitable for plant milk production (Kurajdova & Tábořecká-Petrovicova, 2015). Aleppo pine (*Pinus halepensis* Mill.) seeds could be a new vegetable drink that does not exist on the market and no research work has been performed in this sense. Indeed, seeds are highly nutritious and are a good source of sugars (5.6%), proteins (26.6%), and lipids (36.7%) with essential fatty acids, mainly unsaturated, with linoleic acid and oleic acid being the major ones (Kadri et al., 2015). Moreover, Aleppo pine is largely widespread throughout the Mediterranean basin found in both natural and urban environments (Dhibi et al., 2012). In total, the forests of Aleppo pine occupy more than 3.5 million hectares (Gazol, Ribas, Gutiérrez, & Camarero, 2017). Given the nutritional quality of the seeds and the availability of the trees, their valorization in the food industry is interesting from a nutritional and economic point of view. The study of the conception of a plant-based beverage using these seeds is therefore relevant. The process of plant milk production consists in extracting the nutritive compounds of the seed in water. However, the design of this process is a real challenge, since seeds and plant milk are complex in their structure. Seeds have many coats that may impair extraction efficiency and the resulting plant-based beverage is a complex colloidal system formed essentially by an oil-in-water emulsion and dispersed particles that is unstable. The most common challenge about setting up such production is to maximize the yield of extraction for the economic viability while ensuring the beverage quality, that is, physical and microbiological stability. Creaming and sedimentation are the two migratory phenomena that are a consequence of physical destabilization of the beverages assumed complex colloidal systems. Creaming and sedimentation are unacceptable for the consumers (Briviba, Gräf, Walz, Guamis, & Butz, 2016). To tackle this problem, several processes have

been studied and adapted to the different plant-based sources. Various pre-extraction steps were conducted to prepare the seed for extraction, namely soaking, roasting, and cooking (Ahmadian-Kouchaksaraei, Varidi, Varidi, & Pourazarang, 2014; Ukwuru & Ogbodo, 2011). For the extraction steps, different parameters were tested such as the ratio of seeds to water, the time (Kizzie-Hayford, Jaros, Schneider, & Rohm, 2015) and temperature of extraction (Duarte et al., 2009). Different strategies were adopted for enhancing physical stability after extraction by first choosing an appropriate separation step, either filtration or centrifugation. This step was followed by the formulation step that consists of the addition of stabilizing food additives, among others (Maghsoudlou, Alami, Mashkour, & Shahraki, 2016; Taherian, Fustier, & Ramaswamy, 2007) and/or more importantly, by ultra-high-pressure homogenization (Bernat, Chafer, Rodríguez-García, Chiralt, & González-Martínez, 2015). It is very important to carry out this stabilization step before the final microbiological stabilization by thermal treatment (Bernat et al., 2015; Polisel-Scopel, Hernández-Herrero, Guamis, & Ferragut, 2012).

The main objective of this study is first to ascertain the potential of Aleppo pine seeds for the production of a plant-based milk alternative and to present a structured approach to creating a process diagram for this new product. In addition, this study aims to better understand the effect of each parameter (alone or coupled) of this complex process from extraction (pre-treatment, ratio, grinding time, and temperature) to stabilization (formulation, homogenization, and pasteurization) through a multicriteria assessment of the quality of the beverage by means of a screening design testing seven different parameters.

2 | MATERIAL AND METHODS

2.1 | Raw material

Aleppo pine cones, harvested from the Collo forest in Skikda, Algeria, were heat treated at 180°C to melt the resin that seals the cone scales and cause them to open and release the seeds trapped inside. These seeds were recovered, washed with tap water, and then dried at 37°C for 24 hr.

2.2 | Chemical reagent

All reagents used in this study were purchased from Sigma-Aldrich (St. Louis, MO): chloroform, methanol, soybean lecithin, guar gum, xanthan gum, starch, amylopectin, and Bradford reagent.

2.3 | Preparation and processing of *Pinus halepensis* seed beverages

To set up the general process of *Pinus halepensis* seed beverage presented in Figure 1, several pretests were conducted to identify the appropriate unit operations and the right range of parameter variation.

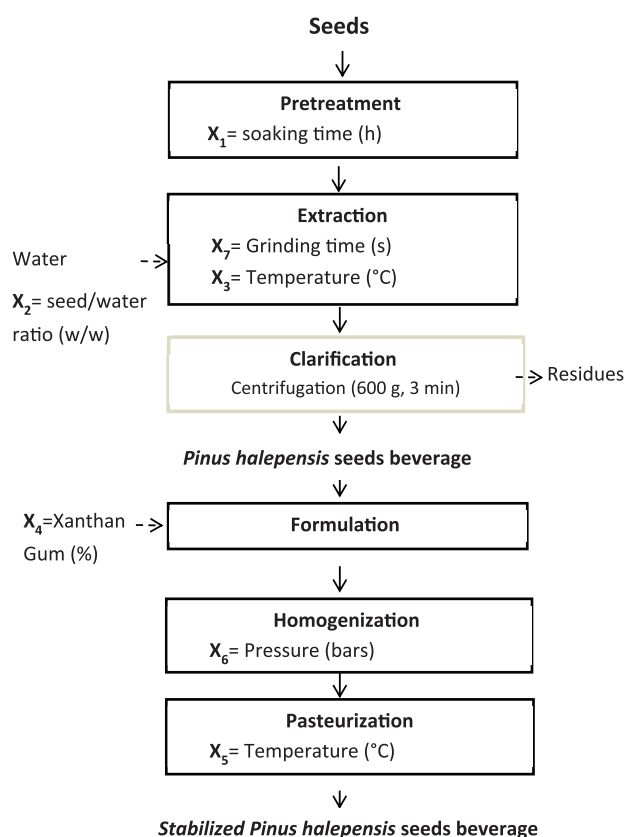


FIGURE 1 Manufacturing process of *Pinus halepensis*. Mill beverage and list of factor X_{1-7} of the screening design

2.3.1 | Selection of unit operation and material

Before extraction, a pretreatment by soaking was tested on the raw seeds at ambient temperature in beakers at a seed/water ratio of 1/10. After draining, the seeds were incorporated in water for extraction. Extraction was carried out in a Thermomix TM6 Vorwerk (Wuppertal, Germany) at the highest speed on the whole seed without dehulling. To start the extraction at the set temperature, the Thermomix was first preheated with the appropriate volume of water before the seeds were incorporated and ground. After extraction, different clarification options were tested. Filtration was first used with filters of different pore diameters, but this operation failed in achieving good yield and purity (remaining black particles, results not shown). Therefore, centrifugation was tested for different combinations (acceleration: time) and was finally carried out at 600 g for 3 min using a Aventi JE centrifuge (Beckman Coulter, CA). After extraction, in order to stabilize the beverage by formulation, several food additives, soybean lecithin, guar gum, xanthan gum, starch, and amylopectin, were tested at different concentrations. Xanthan gum was selected visually because it presented the best solubility and stabilizing property. Finally, physical and microbiological stabilization

TABLE 1 Independent variables of milk manufacturing process and their levels in the fractional screening design

Independent variables	Symbols	Level	
		−1	+1
Soaking time (hr)	X_1	0	4
Seed/water ratio of extraction (/10)	X_2	1	3
Temperature of extraction (°C)	X_3	20	100
Grinding time (s)	X_7	20	120
Xanthan gum (%)	X_4	0	0.4
Pressure of homogenization (bar)	X_6	0	700
Temperature of pasteurization (°C)	X_5	65	85

were also considered by applying high pressure homogenization with a homogenizer from A/S APV 1000/2000 (SPX FLOW Technology, Silkeborg, Denmark) followed by pasteurization in a water bath using 25 mL Pyrex tubes.

2.3.2 | Range of processing conditions for each step

Based on the operations selected and given the large number of potential factors affecting the beverage quality, we opted for the fractional factorial $2^{(7-4)}$ screening design to optimize production. Three central points were added to evaluate the repeatability of experiments. The range of processing conditions are presented in Table 1 and were used to define the factor X_{1-7} variation of the screening design of experiments presented in Table 2. Soaking was carried out with a duration range of 0–4 hr (X_1). Extraction was carried out from 20 to 100°C (X_3) at different seed/water ratios from 1/10 to 3/10 (wt/wt) (X_2) and ground from 20 to 120 s (X_7). Then, xanthan gum was added at room temperature from 0 to 0.4% under stirring (X_4). Finally, homogenization was performed from 0 to 700 bars (X_6) and pasteurization in a water bath at 65 to 85°C (X_5). Duration of pasteurization treatment at 65 and 85°C was set to achieve a pasteurization value (P) of 100 min at $T_{ref} = 70^\circ\text{C}$. P was calculated from the time–temperature curve of the beverage in the tubes according to Equation (1),

$$P = \int_{t_0}^{t_{end}} 10^{\left(\frac{T(t)-T_{ref}}{z}\right)} \cdot dt, \quad (1)$$

where $T(t)$ is the heat treatment time (time of exposure to heat) and z is the thermal activation value (the temperature increase leading to a 90% reduction of the treatment time).

A total of 11 Aleppo pine beverages were produced corresponding to the conditions of the 11 essays of the screening design (Table 2).

TABLE 2 Matrix of the fractional screening design $2^{(7-4)}$ with coded value of the different parameters of manufacturing process of the Aleppo pine seed beverage

Assay	Soaking time (h) X_1	Seed/water ratio X_2	Temperature of extraction ($^{\circ}\text{C}$) X_3	Xanthan gum (%) X_4	Temperature of pasteurization ($^{\circ}\text{C}$) X_5	Pressure of homogenization (Bar) X_6	Grinding time X_7 (s)
1	-1	-1	-1	1	1	1	-1
2	-1	-1	1	1	-1	-1	1
3	1	1	-1	1	-1	-1	-1
4	1	1	1	1	1	1	1
5	1	-1	1	-1	1	-1	-
6	1	-1	-1	-1	-1	1	1
7	-1	1	-1	-1	1	-1	1
8	-1	1	1	-1	-1	1	-1
9	0	0	0	0	0	0	0
10	0	0	0	0	0	0	0
11	0	0	0	0	0	0	0

2.4 | Analysis of Aleppo pine beverage

2.4.1 | Chemical properties

Dry matter (DM) was determined by the AOAC method (AOAC, 2005). The extraction yields were calculated as the ratio of the weight of DM of the final beverage over the initial DM of the seeds used. The pH was measured with a pH meter (Titroline 96, Schott-Geräte GmbH, Mainz, Germany). The lipids were quantified with the Folch method using chloroform/methanol (3:1) for extraction (Corrales Hernández et al., 2017). Proteins were measured with the Bradford method (Kruger, 2009). Finally, aroma compounds were extracted with SPME (Solid Phase Micro-Extraction) and analyzed with GC-MS Agilent 6890/5973 series (Agilent Technologies, Palo Alto, CA) equipped with a DB-WAX polar column (60 m, 0.25 mm, 0.25 μm phase film thickness, Agilent J&W GC column) using butanol as an internal standard.

2.4.2 | Physical properties

Rheological behavior

Rheological measurements of *P. halepensis* seed beverage were performed using a Physica MCR301 rheometer (Anton Paar GmbH, Graz, Austria) equipped with a Couette flow measuring cell (Ref. DG27/T2000/SS), according to the procedure of Dahdouh et al. (2015). All measurements were conducted at 25°C . Consistency index, flow index, and viscosity at a shear rate of 100 s^{-1} were deduced from the following formula (Equation (2)):

$$\eta = K\dot{\gamma}^{n-1}, \quad (2)$$

where η is the apparent dynamic viscosity (Pa-s), K is the consistency index, $\dot{\gamma}$ is the shear rate (s^{-1}), and n is the flow index.

Particle size distribution

Particle size distribution was determined by laser light scattering using a Mastersizer 3000 (Malvern Instruments, Ltd., Worcestershire, UK) and log-spaced array detector in a range between 0.01 and $3,500 \mu\text{m}$. This method involved loading the sample into a dispersant unit HydroMV filled with water as suspension medium set for obscuration at 20% and stirring of 1,500 rpm (Corredig, Kerr, & Wicker, 2001). Particle size distribution was expressed as $D_{(4:3)}$ (particle diameter means), DV_{90} (particle diameter of 90% of particles), and span (particle dispersion) indices.

Colloidal stability

The stability of the suspension was evaluated after storage of the sample for 48 hr at 4°C . A volume of 10 mL of *P. halepensis* beverage was placed in a 15 mL graduated tube and three phenomena were observed and measured for stability: creaming, dephasing, and sedimentation. The ratio of the volume of creaming (c), phase shift (p), or sediment (s) to the total liquid volume were measured. Creaming, phase shift, or sediment are considered equally decreasing visual stability as can be seen in Figure 2. Therefore, we proposed the following equation to gather these three phenomena as an index of visual stability (Equation (3)):

$$VS = 100 - \sqrt{a^2 + p^2 + c^2}. \quad (3)$$

Optical properties

The color of the beverage was expressed as whiteness index (WI), Chroma (C^*), and hue (h^*_{ab}), by measuring it with the $L^*a^*b^*$ system using a CR400 colorimeter (Konica Minolta, Tokyo, Japan) with the illuminant of D65 and an angle of 0° and using following equations (Equations (4)–(6)) (Bernat et al., 2015),

$$WI = 100 - \sqrt{(100 - L^*)^2 + a^{*2} + b^{*2}}, \quad (4)$$



FIGURE 2 Illustration of creaming, phase shift, and sediment to calculate the visual stability index (VS) according to Equation (3)

$$C^* = \sqrt{a^{*2} + b^{*2}}, \quad (5)$$

$$h_{ab}^* = \arctan(b^* / a^*), \quad (6)$$

where L^* represents the lightness with value from black (0) to white (100), a^* represents that positive values are red and negative values are green. b^* represents that positive values are yellow and negative ones are blue.

2.5 | Statistical analysis

The statistical analysis of the experimental design was carried out using the Statistica 7.1 software and the factors were considered significant at $p < .1$. The hypothesis of linearity underlined by the experimental design chosen was validated by the analysis of the significance of curvature. The linearity is validated if the curvature, which is the difference between the theoretical mean proposed by the model and the experimental mean, does not exceed 5%.

3 | RESULTS AND DISCUSSION

3.1 | Composition of *P. halepensis* seeds

Aleppo pine seed composition was experimentally measured in wet basis and contained $27.48 \pm 0.17\%$ proteins, $31.55 \pm 0.11\%$ lipids,

and $4.97 \pm 0.17\%$ carbohydrates. These values were close to results presented by Kadri et al. (2015). The dry matter (DM) of the raw seeds was 91.97%. DM of the seed soaked for 2 hr was 82.01 and 77.96% for the seeds soaked for 4 hr. The composition of the remaining 27% of matter was attributed to fibers and minerals (Zhong et al., 2018). The decrease of DM is due to the hydration kinetic. Hydration reached 9.96% in 2 hr and 14.03% in 4 hr which are significant, similar to almond (Khazaei, 2008), but quite low in comparison with rich starchy seeds like soybean (de Lima, Kurozawa, & Ida, 2014).

3.2 | Results of the experimental design: Effect of processing factors on the multicriteria quality of the Aleppo pine seed beverage

The seeds were used to produce a plant-based beverage according to the chart presented in Figure 1 and conditions of the experimental design presented in Tables 1 and 2. The quality analyses, for which the chemical and physical characteristics were measured on the nine different beverages obtained, are given in Table 3.

3.2.1 | Chemical characteristics

Dry matter, fats, and proteins

The statistical study showed that the ratio (seeds/water, wt/vol) (X_2), grinding time (X_7), and temperature of extraction (X_3) were the only significant factors affecting dry matter, protein, and fat contents. The ratio (seeds/water, wt/vol) and temperature of extraction had a positive effect on all three responses with respective p values of 0.0007, 0.045, and 0.023 for DM, 0.0007, 0.020, and 0.038 for fats, and 0.001, 0.005, and 0.018 for proteins. The temperature of extraction had a positive effect on lipids but a negative effect on DM and proteins. Based on the assumptions of this experimental design, no interaction effect was identified for these three responses. Since the curvature, which is the difference between the theoretical mean proposed by the model and the experimental mean, was insignificant, the DM, lipid, and protein models can be assumed to have a linear response. The DM of the final beverage varied significantly between 2.2 and 9.4% with a mean of 5.8%. These values were close to those found in almond beverages (6.2%) (Maria & Victoria, 2018), in melon seed beverages (9.9%) (Akubor & Ogbadu, 2003), and in almond and hazelnut beverages (6.6 and 5.9%, respectively) (Bernat, Cháfer, Chiralt, & González-Martínez, 2014). Lipid results varied between 1.2 and 7.2% with an average of 4.0%, which is comparable to the levels found in soy milk (3.2%), almond milk (3.4%), sesame milk (about 7%), and peanut milk (about 4%) (Lee & Beuchat, 1992). The level of proteins found in the beverage was between 0.5 and 2.3% with an average of 1.3%. These results are close to those found in melon seed milk which is around 2% and in soy bean and almond milks 1.2 and 0.7%, respectively (Akubor & Ogbadu, 2003; Sharma, Kumari, Wongputtisris, Nout, & Sarkar, 2015).

TABLE 3 Results of chemical and physical properties of Aleppo pine seed beverage samples obtained from the fractional screening design

Assay	Nutritional quality				Aromatic compounds				Stability	Droplet size distribution			Rheological behavior				Color	
	Dry matter (%)	Yields (%)	pH	Fats (%)	Proteins (%)	α -pinene (%)	β -myrcene (%)	D-limonene (%)		D _(4:3)	DV ₉₀	Span	Viscosity (Pa/s) ^a	Consistency index (K)	Flow index (n)	Whiteness index (%)	Chroma	Hue angle
1	2.5	21.5	6.7	1.2	0.9	0.531	0.057	0.019	100.0	12.0	19.0	2.0	16.6	0.033	0.86	78.6	3.3	65.7
2	3.0	21.3	7.2	2.5	0.5	0.041	0.003	0.021	94.3	216.4	461.7	2.5	19.5	0.213	0.47	74.7	7.1	80.7
3	9.0	28.1	6.3	5.5	2.0	1.004	0.189	0.047	33.4	172.9	402.7	2.9	13.6	0.398	0.24	85.0	7.7	89.0
4	9.0	28.6	6.5	6.9	1.9	0.6	0.153	0.052	100.0	35.5	74.3	2.5	26.4	0.079	0.78	79.2	8.7	75.6
5	2.2	21.5	7.2	1.6	0.5	0.507	0.164	0.032	94.3	21.5	43.3	2.2	1.1	0.001	1.00	83.0	3.5	74.5
6	3.9	21.6	6.8	2.0	1.3	0.832	0.117	0.04	100.0	50.8	102.5	2.3	1.2	0.001	1.01	83.0	3.5	88.7
7	9.4	28.4	6.5	5.6	2.3	1.215	0.466	0.065	91.6	11.6	23.4	2.9	2.0	0.002	0.96	94.0	5.9	88.8
8	7.6	28.3	7.1	6.5	1.3	0.823	0.215	0.062	81.2	43.6	86.4	2.1	1.4	0.001	0.92	77.4	11.6	47.6
9	6.2	32.8	6.5	3.5	1.2	0.489	0.057	0.024	97.5	41.1	82.1	2.1	11.8	0.038	0.74	80.4	8.2	78.6
10	6.0	32.7	6.7	3.8	1.3	0.475	0.081	0.024	98.3	24.7	47.7	2.0	10.6	0.023	0.84	78.6	8.3	76.3
11	6.5	32.9	6.5	3.6	1.2	0.531	0.057	0.019	100.0	12.0	19.0	2.0	16.6	0.033	0.86	78.6	7.8	76.4

^aAt a shear rate of 100 s⁻¹.

Yields

The yield depended only on the ratio X_2 ($p = .000004$) and there were no interaction effects with other factors. In this case, the curvature was significant. Therefore, the yield was assumed a nonlinear response of the ratio. The yield varied between 21.3 and 32.8% from ratio 1/10 to 3/10 with an average of 24.9%. These values were higher than the yields obtained for tiger nut beverage, which were between 11 and 19% (depending on seed size, grinding time, and soaking temperature) but lower than that of 56% obtained during almond beverage production (Maria & Victoria, 2018).

pH

The results showed that the pH was influenced negatively by the ratio (X_2) ($p = .033$) and positively by the temperature of extraction ($p = .034$). The interaction effect and the curvature were insignificant for this response. For all products, the pH was still quite neutral. The pH values obtained (with an average of 6.8) were comparable to those of cowpea milk (Nnam, 2003), melon seed milk (Akubor & Ogbadu, 2003), soymilk and almond milk (Kundu, Dhankhar, & Sharma, 2018).

Aromatic compounds

The aroma compounds most present in the beverage of Aleppo pine were α -pinene, β -myrcene, and D-limonene. Significant factors with a positive effect were the soaking time (X_1) ($p = .0004$) for α -pinene, the ratio (X_2) for α -pinene, β -myrcene, and D-limonene (with p -values of 0.0025, 0.014, and 0.0087, respectively) and with a negative effect of the temperature of extraction (X_3) for α -pinene and β -myrcene (with p -values of 0.035 and 0.0007, respectively), and of xanthan gum (X_4) for α -pinene, β -myrcene, and D-limonene (with p -values of 0.0025, 0.0254, and 0.0078, respectively). Furthermore, since factor X_6 was significant for the β -myrcene response, and factors X_2 and X_3 were also significant, there could be an interaction effect between these two factors: ratio and temperature of extraction (X_2X_3). Finally, curvature was significant for α -pinene and β -myrcene, which means that only the D-limonene model could be linear. Aroma compound values ranged from 0.04 to 1.21% for α -pinene, 0.01 to 0.46% for β -myrcene, and 0.02 to 0.06% for D-limonene.

3.2.2 | Physical characteristics

Stability

The factors influencing stability were (in descending order): the ratio (factor X_2) with a negative effect ($p = .0001$), pasteurization temperature (factor X_5) ($p = .0001$), and grinding time (factor X_3) ($p = .0001$) (the same positive effect) and then, the homogenization pressure (factor X_6) ($p = .0002$) which also had a positive effect, temperature of extraction (factor X_3) ($p = .0005$) and finally, the soaking time (X_1) ($p = .0007$) and xanthan gum (X_4) ($p = .0007$) with the same negative effect. It makes sense that many factors affect stability. This is the most delicate point and the manufacturing challenge. Both the formulation factors (ratio and additive that should

not be increased too much) and process temperature and homogenization are important.

All factors were significant, therefore there may be interaction effects between soaking time and ratio (X_1X_2) because X_5 (pasteurization temperature) was significant, between X_2X_3 and X_1X_2 , ratio and temperature of extraction (X_2X_3) because X_6 (homogenization pressure) was significant and finally, between soaking time and temperature of extraction (X_1X_3) because X_7 (grinding time) was significant. However, the curvature was also significant which may be due to the nonlinearity of this model. The following indicators can explain further the stability observations.

Droplet size distribution

Figure 3 illustrates the difference in the particle size distribution of the eleven assays. Most samples had about the same maximum diameter volume distribution (around 50 μm) except four samples, of which two samples (assay 3 and 2) had most of their maximum particle volume density around 500 μm . This result was influenced by the presence of xanthan gum at the highest concentration (0.4%), the minimum pasteurization temperature (65°C) and the minimum homogenization pressure (without homogenization). Moreover, two samples presented the maximum of the particle volume density of the lowest size, around 10 μm . These samples were influenced mainly by the conditions of maximum pasteurization and homogenization.

Particle size $D_{(4;3)}$. Pasteurization temperature (X_5) ($p = .003$), xanthan gum (X_4) ($p = .005$), and homogenization pressure (X_6) ($p = .007$) were the factors that influenced the mean particle diameter in this order, X_5 and X_6 with a negative effect, and X_4 with a positive effect, but no significant interaction effect was observed. However, the curvature was significant, so the model can be nonlinear. The diameter of the particles varied between 11.6 and 216.4 μm , the maximum diameter obtained was higher than the $D_{(4;3)}$ obtained by most vegetable

beverages even without any treatment, for example hazelnut milk had a $D_{(4;3)}$ of 172.1. This difference was mainly influenced by the presence of xanthan gum (Gul, Saricaoglu, Mortas, Atalar, & Yazici, 2017).

Particle size $DV_{(90)}$. The 90% particle size was influenced the most negatively by pasteurization temperature ($p = .0002$), then, positively by xanthan gum ($p = .004$), and then, negatively by homogenization pressure ($p = .0078$). No interaction effect has been revealed. However, the model could be nonlinear because the curvature was significant.

Span. The particle dispersion (span) was influenced by ratio with a positive effect ($p = .0094$) and by the homogenization pressure with a positive effect ($p = .0046$). The model seemed to be non-linear (significant curvature). The span values varied between 1.9 and 2.9. A study of hazelnut milk (Gul et al., 2017) showed that these values varied between 1.9 and 3.2 depending on the concentration and pressure of homogenization.

Rheological behavior

Figure 4 describes the difference in rheological behavior of the eleven samples as a function of shear rates. Assays were grouped into two large groups: one group containing four samples (assay 5, 6, 7, 8) with a viscosity close to 1 mp/s with Newtonian behavior (which are samples that do not contain xanthan gum); and another group with a higher viscosity. Samples 1–4 contained gum xanthan and their dynamic viscosity decreased as the shear rate increased. Therefore, gum xanthan gave shear-thinning properties to the samples. The slight difference observed between samples 2, 3 and 1, 4 may be caused by the difference in homogenization pressure that was maximal for samples 2 and 3. Therefore, the homogenization pressure had a fluidizing effect on the product (Tanguy & Ascanio, 2005).

Viscosity. The viscosity was influenced first by xanthan gum (X_4) ($p = .0006$), then in the following order by grinding time (X_7)

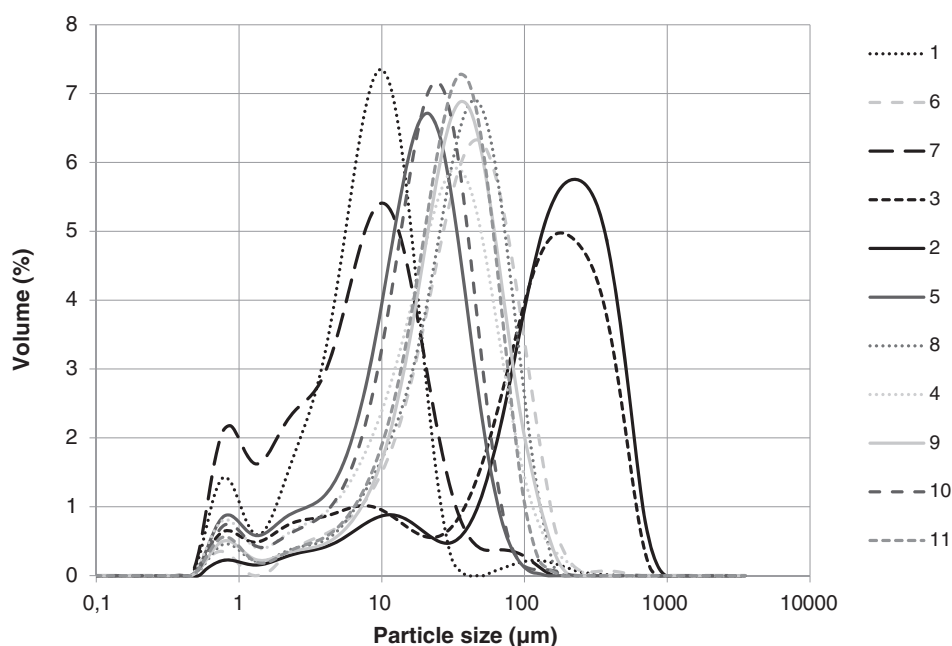


FIGURE 3 Particle size distribution curves of the different Aleppo pine beverages obtained according to the screening design experiments

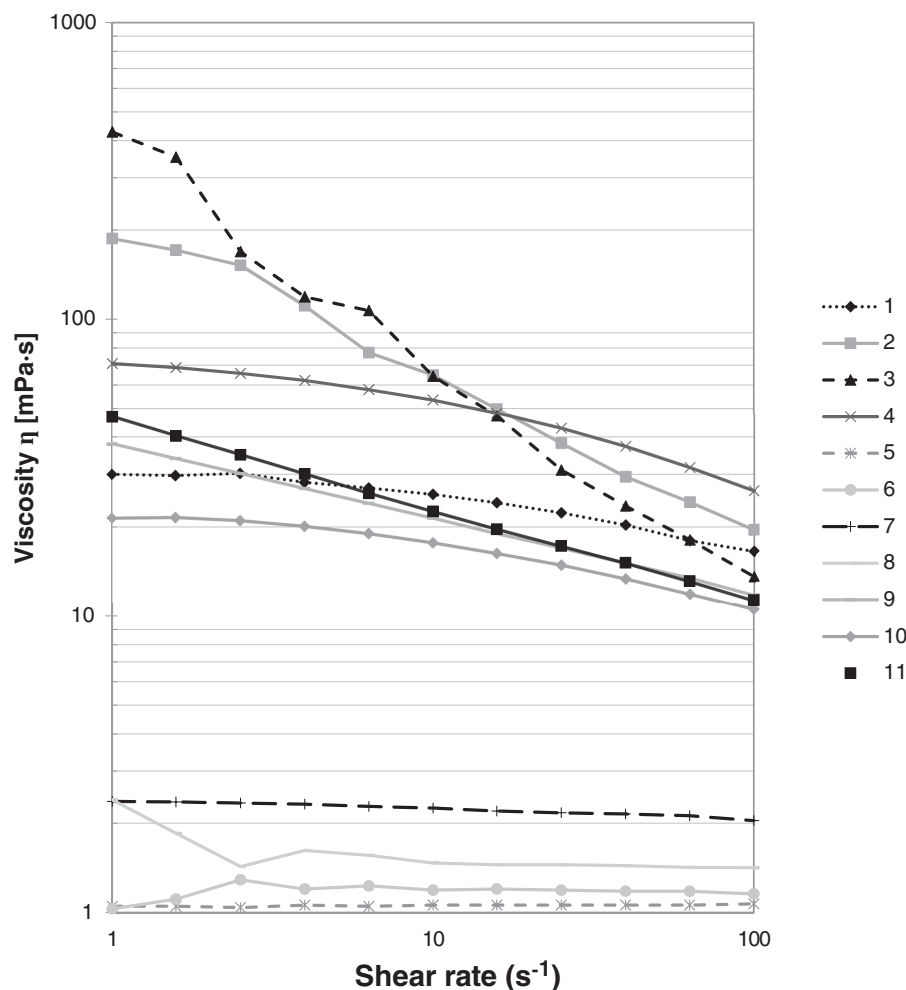


FIGURE 4 Rheological behavior of different Aleppo pine beverage samples obtained according to the screening design experiments

($p = .011$), temperature of extraction (X_3) ($p = .013$), pasteurization temperature (X_5) ($p = .028$) and finally, homogenization pressure (X_6) ($p = .035$). All these factors affected the viscosity with a positive effect. No interaction effects were observed. Indeed, the model could be assumed as being linear because the curvature was nonsignificant.

Consistency index. All factors were significant with the same degree: soaking time, ratio, temperature of extraction, and grinding time had a negative effect and the rest of the factors had a positive effect with the same p -values $<.0001$. The model could be not linear because curvature was significant.

Flow index. The flow index was influenced by xanthan gum (X_4) (negative effect) ($p = .003$), then pasteurization temperature (X_5) (positive effect) ($p = .013$) and then homogenization pressure (positive effect) ($p = .017$), no interaction effect has been revealed, and the model could be assumed as being linear since curvature was nonsignificant.

Color

Whiteness index, hue, and chrome values were calculated for every sample (Table 3) but only whiteness index (WI) was used as a response of the experimental design since this parameter is more relevant for milk-like products and it changed significantly as a function of the processing factors as observed by Bernat et al. (2015).

The factors that were found to be significant regarding WI are (in decreasing order of effect), temperature of extraction (negative effect) ($p = .016$), xanthan gum (%) (negative effect) ($p = .028$), homogenization pressure (negative effect) ($p = .033$), ratio (positive effect) ($p = .041$), and finally pasteurization temperatures (positive effect) ($p = .049$). The factor X_6 (homogenization pressure) was significant and the factors X_2 (ratio) and X_3 (temperature of extraction) were also significant. In this case, the result may reveal a possible interaction between the ratio (X_2) and the temperature of extraction (X_3), which must be confirmed by further experiments. However, the curvature was not significant, which means that the model could be linear. The WI of the beverage varied between 74.6 and 94.0% with an average of 81.8% which is close to the values of WI obtained on almond and hazelnut milks in the study of Bernat et al. (2015).

3.3 | Discussion about the main effects of each processing factor on the quality of Aleppo pine seed beverage

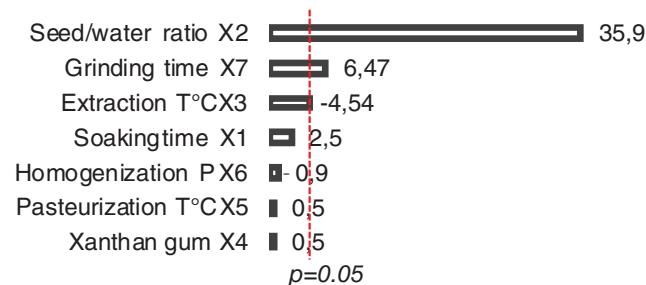
The effect of key factors in the Aleppo pine seed plant-based beverage manufacturing process and their analysis of variance on the chemical and physical quality are presented in Table 4 and the Pareto charts of the most important responses (dry matter, stability, and whiteness index) are shown in Figure 5.

TABLE 4 Analysis of variance for the experimental results obtained for the different responses of the screening design (regression coefficient and statistical significance)

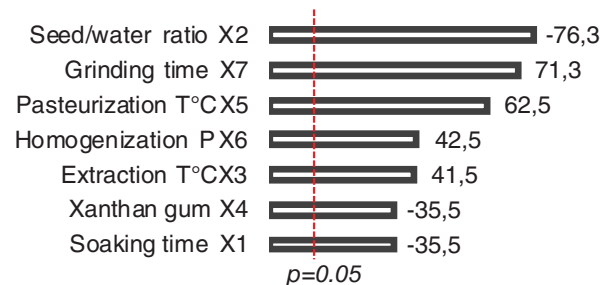
	Chemical properties							Physical properties								
	Nutritional quality				Aromatic compounds			Stability	Droplet size distribution			Rheological behavior		Color		
	DM	Yields	pH	Fats	Proteins	α -pinene	β -myrcene		D-limonene	Stability index	Particle size $D_{(4,3)}$	Particle size DV_{90}	Span		Viscosity at a shear rate of 100 ⁻¹	Consistency index (K)
Mean/inter	5.82***	24.90***	6.79***	3.97***	1.35***	0.69***	0.17	0.04	86.85***	70.52***	151.67***	2.41	10.227***	12.837***	0.77***	81.85***
R^2	0.99	0.99	0.97***	0.99	0.99	0.99	0.99	0.98	0.99	0.99	0.98	0.98	0.99	1	0.97	0.98
Curvatr.	0.41****	7.88***	6.79***	-0.32***	-0.11****	-0.22***	-0.09*	-0.01	10.98***	-36.61*	-85.20*	-0.37***	0.98***	-12.80***	-0.02***	-1.89****
Soaking time (X_1)	0.20****	0.03****	-0.10****	0.04****	0.07	0.04*	-0.01****	5.10 ⁻⁴ ****	-4.92***	-0.37****	4.02****	0.07****	0.33****	-12.71***	-0.02***	0.69****
Seed/water ratio (X_2)	2.92***	3.417***	-0.21*	2.14***	0.53***	0.21***	0.08**	0.01*	-10.29***	-4.65****	-4.95****	0.18**	0.64****	-12.71***	-0.05****	2.04*
Temperature of extraction (X_3)	-0.37**	0.02****	0.20**	0.39**	-0.29***	-0.20***	-0.03*	-5.10 ⁻⁴ ****	5.60***	8.70****	14.75****	-0.09	1.87**	-12.76***	0.01****	-3.28*
Xanthan gum (X_4)	0.04***	-0.03****	-0.11****	0.04****	-0.02****	-0.15***	-0.07*	-7.10 ⁻³ *	-4.92***	38.66***	87.78**	0.06*	8.80***	12.83***	-0.19***	-2.49*
Temperature of pasteurization (X_5)	-0.04****	0.07****	-0.06****	-0.14****	0.05****	0.01****	0.03*	-2.10 ⁻⁴ ****	9.62***	-50.40***	-111.66***	-0.02****	1.29**	12.68***	0.12**	1.86*
Pressure of homogenization (X_6)	-0.07****	0.09****	-0.01****	0.17****	-2.10 ⁻³ ****	-2.10 ⁻³ ****	-0.03*	1.10 ⁻³ ****	8.45***	-35.05***	-81.11***	-0.21**	1.15**	12.68***	0.11**	-2.31*
Milling time (X_7)	0.52**	0.06****	-0.03****	0.28**	0.15**	-0.02****	0.01****	2.10 ⁻⁴ ****	9.62***	8.05****	13.81****	0.12****	2.05**	12.76***	-0.08****	0.85****

* $p < .1$; ** $p < .05$; *** $p < .01$; **** $p > .1$.

Dry matter



Stability



Whiteness Index

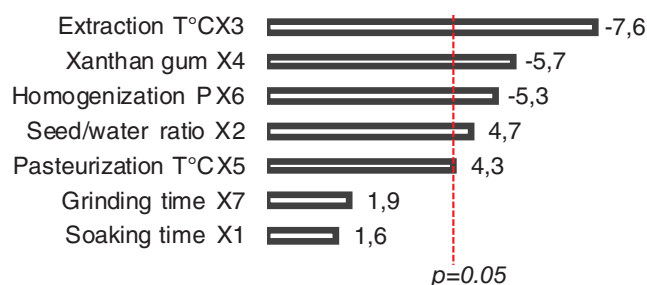


FIGURE 5 Pareto charts representing the effect of all investigated factors on dry matter, stability, and whiteness index

3.3.1 | Soaking time X_1

The soaking time did not exhibit any effect on the composition quality of the beverage (dry matter, yield, pH, fats, and proteins) but it allowed more aromatic compounds (α -pinene and D-limonene) to be extracted. The same positive effect of soaking on aromatic compounds was found in tiger nut milk (Djomdi, Ejoh, & Ndjouenkeu, 2007). This may be due to the fact that soaking markedly hydrated the seeds which softened the plant matrix and enables a better release of aroma compounds in the beverage during grinding (Djomdi et al., 2007). However, the longer the soaking time, the less the milk was stable and consistent. The soaking time negatively affected the stability and consistency because stability of plant-based milk depends on the size of dispersed phase particles.

3.3.2 | Ratio X_2

The ratio was positively proportional to the yield and the final DM as well as the protein, fat levels, and aroma compounds (α -pinene,

β -myrcene, and D-limonene). As explained above, the DM was essentially influenced by the ratio. An increase in the ratio lowered the pH. This small but significant decrease may be due to the release of organic acids, a phenomenon which is not favorable for stability. The ratio decreased the consistency and increased the span and therefore increased the instability of the product. This can be explained by the fact that the ratio increased the amount of matter and that at a critical concentration, a destabilization can take place. However, the ratio had no significant effect on particle size, viscosity, and flow index. On the other hand, the higher the ratio, the higher the whiteness index, which leads to the conclusion that the whiteness index was also proportional to the amount of DM in the beverage.

3.3.3 | Temperature of extraction X_3

The temperature of extraction had a positive effect on lipid extraction, but a negative effect on dry matter and this is explained by the fact that this parameter also has a negative effect on protein content. The study, carried out by Ejoh, Djomdi, and Ndjouenkeu (2006), revealed that when the temperature was raised, the cell walls swelled and ruptured thus increasing the level of solids in the water. This observation is consistent with the positive effect of temperature extraction on all nutrients except on the proteins. Indeed, when beverages are heated to temperatures above 65°C, the deployed proteins expose their hydrophobic groups to the outside and aggregate together to form very high molecular weight molecules (Donato, Guyomarc'h, Amiot, & Dalgleish, 2007). This phenomenon can explain a reduced concentration of protein in the beverage because of a decrease in solubility. In addition, this change may have an impact on their emulsifying properties (Claeys et al., 2013). The aggregation of proteins and the reduction of their solubility mean that when the mixture is centrifuged, the proteins settle and are eliminated with the solid residue, which may be the reason why grinding at high temperatures reduces the level of proteins in the beverage. The temperature of extraction was also inversely proportional to the concentration of aroma compounds (α -pinene and β -myrcene). This may be due to the action of temperature on the destruction of aroma compounds. Also, the positive effect of the temperature of extraction on pH may be due to the destruction of acids. The temperature of extraction caused the whiteness to decrease, which may be due to the higher extraction of some particles from the seed coat. The higher was the temperature of extraction, the more stable was the beverage regarding its positive effect on viscosity and negative effect on consistency.

3.3.4 | Xanthan gum X_4

The amount of xanthan gum added had no effect on the chemical properties in general (dry matter, yield, lipids, proteins, pH) except that it lowered the level of aroma compounds (α -pinene, β -myrcene, D-limonene). It is known that the use of polysaccharides as thickeners often leads to the retention of aroma compounds and decreases their liberation in the headspace, which results from two main possible

mechanisms: the slowing down of their diffusion caused by the increase of the viscosity of the solution and the inter and intramolecular interactions, absorption and encapsulation (Secouard, Malhiac, Grisel, & Decroix, 2003). The study by Secouard, Malhiac, and Grisel (2003) highlighted the same effect of xanthan gum on limonene. Contrary to most studies that prove that xanthan gum is an excellent stabilizer (Taherian et al., 2007), our study showed that stability was inversely proportional to the concentration of xanthan gum. According to Taherian et al. (2007) xanthan gum exerts its best stabilizing effect at low concentrations. In our results, the non-linearity of the stability toward xanthan gum may reveal that the beverage containing low concentration is much more stable than the beverage containing no xanthan gum or at high concentrations. Moreover, we also noticed in the preliminary study that the stability in the presence of xanthan gum also depended on the ratio (seeds/water). On the other hand, the more concentrated xanthan gum was, the more the particle size increased ($D_{(4:3)}$ and $DV_{(90)}$). The same particle magnifying effect was exhibited in the study of Mirhosseini, Tan, Hamid, Yusof, and Chern (2009). This may be due to the aggregating effect that can occur between the xanthan gum and other compounds of the beverage (Taherian et al., 2007) that can cause the observed increase of the viscosity and consistency (Kumar, Roy, Devra, Dhiman, & Prabhakar, 2021). Xanthan gum has the ability to form a network by entering lipidic droplets with a film, which somehow immobilizes the particles of the hydrocolloid system, so that the higher the concentration of xanthan gum, the more viscous and consistent the beverage was (Taherian et al., 2007). Finally, xanthan decreased the whiteness index and this is explained by the yellowish color of the xanthan gum that changed the color of the beverage at high concentrations (Taherian et al., 2007).

3.3.5 | Pasteurization temperature X_5

The pasteurization temperature had almost no effect on the chemical properties of the beverage (dry matter, yield, proteins, lipids, pH) but decreased the particle size ($D_{(4:3)}$ and DV_{90}). This is the opposite found in most studies where pasteurization increased particle size, caused by the aggregation of particles, especially proteins (Bernat et al., 2015). This inverse result can be explained by the fact that there were many more lipids than proteins in the samples and that the temperature can cause the lipid droplets to break and therefore decrease in size (Cavazos-Garduño et al., 2016). Pasteurization temperature also increased the parameters of rheological behavior (viscosity, consistency index, and flow index). This can be explained by the fact that the high temperatures induce a partial denaturation of the proteins and their aggregation. This denaturation increases the hydrodynamic volume resulting in a slight gelation. On the other hand, the rheological behavior can be the result of the hydration of the biopolymer chains of the soluble fibers (Bernat et al., 2015). These phenomena may explain its positive effect on stability. The slight positive effect of pasteurization temperature on whiteness index may also be due to the decrease of particle sizes contributing to light reflection. However,

this effect could be counterbalanced by a possible Maillard reaction activation that can be noticed by the decrease of the Chrome value and Hue angle (Bernat et al., 2015).

3.3.6 | Homogenization pressure X_6

The homogenization pressure had no effect on the chemical properties of the beverage. However, it has a very significant positive effect on the stability and this may be due to its negative effect on the particle size behavior ($D_{(4:3)}$, DV_{90} , and span) and its positive effect on the rheological behavior (viscosity, consistency index and flow index). Regarding color, the higher the homogenizing pressure, the lower the whiteness index. The homogenizing pressure also reduced whiteness. This could be due to the higher refraction of light due to the formation of smaller molecules (small sizes) and to some degree to oil separation (Kumar et al., 2021). In addition, the homogenization pressure could change the conformation of the proteins, causing them to unfold but also cut the lipidic droplets of the beverage. Both phenomena can cause the observed increase of viscosity and consistency (Kumar et al., 2021) and make the product more stable. Indeed, the homogenizing process has proven to be successful in reducing particle size and to bring rheological changes. This is due to the fact that in high shear mixers, the product is agitated at high speed, which disrupts the cohesion of the particles. High pressure cause turbulence, cavitation and shear action produces collisions between the particles and the wall from HPH. Milk treated by HP showed higher gel strength, higher gel firmness, and lower synergy firmer and lower synergy (Dhiman & Prabhakar, 2020).

The homogenization pressure and the pasteurization temperature are the main factors improving the stability by reducing particle size which favors their dispersion in the aqueous phase, increasing the viscosity and preventing flocculation and coalescence.

3.3.7 | Grinding time

The grinding time has shown a positive effect on the dry matter as well as on proteins and lipids. The more the mixture was crushed, the more compounds were extracted. It also had a positive effect on stability and viscosity and a negative effect on the consistency index. Similarly, a positive effect of grinding time on the dry matter of the tiger nut beverage was found in the study of Ellis, Oduro, Saalia, and Asante (2014). As in many studies (Kundu et al., 2018), when the grinding time increases, the diffusion of the particles consisting essentially of lipids and fibers also increases by destroying more cell walls and releasing their contents.

4 | CONCLUSION

The design of the experiments allowed the impact of various key operations and key factors of the production of Aleppo pine seed

beverage on its quality to be studied. It was shown that all the factors had a significant impact on the quality parameters. The soaking time had a negative effect on the stability. During extraction, the ratio was a very important factor for the nutritional quality. However, the higher the ratio, the more unstable the product was. Grinding time and extraction temperature were positive factors for nutritional quality and stability, except that extraction temperature had a negative effect on protein extraction. Regarding the formulation step, the xanthan gum that was added to thicken and stabilize the beverage had a positive effect on viscosity but increased the particle size. Finally, high homogenization pressure and pasteurization temperature had almost the same positive effect on stability and this was due to their reducing effect on particle size and increasing effect on viscosity. Aleppo pine seeds are indeed an interesting source for the design of a plant-based beverage, because we could still have a beverage comparable to other commercialized plant-based beverages in terms of nutritional and physical quality. In order to determine the optimal conditions for the design of the beverage, a complete experimental design must be applied to confirm these effects and sensory analysis and a study of acceptability of this new product must be studied.

CONFLICT OF INTEREST

The authors declare no potential conflict of interest.

AUTHOR CONTRIBUTIONS

Amina Abbou: Formal analysis; methodology; writing – original draft.

Nabil Kadri: Project administration; supervision; writing – review and editing. **Adrien Servent:** Formal analysis; methodology. **Madani Khodir:** Funding acquisition. **Manuel Dornier:** Methodology; supervision; writing – review and editing. **Antoine Collignan:** Data curation; methodology; writing – review and editing. **Nawel Achir:** Methodology; project administration; supervision; writing – review and editing.

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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