

Formulation of a dessert cream enriched with the fruit *Zizyphus lotus* L. based edible gelatin obtained from leather rejects

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ABSTRACT/RESUME

Abstract: Our study is part of sustainable development and environmental protection, consists in recovering leather rejects in the agri-food sectors in the form of gelatin and focuses on the possibility of assessing the role of using fruit of *Zizyphus lotus* L. in dessert cream to improve its sensory and nutritional qualities. The results of the physical-chemical tests of *Zizyphus lotus* L. are satisfactory, and namely: the water content 15.65 %; ashes 3.52 mg; the rate of soluble solids 8 °Brix; pH 6.66; electrical conductivity 2.17 μ s/cm; titratable acidity 1.12g; lipids 0.97%; carotenoids 0.213 mg; polyphenols 0.471mg; antioxidants 0.206 %; total sugars 17.19 g/l; cellulose 1.95 %; protein 1.87 % and pectin 2.78 mg. The *Zizyphus lotus* L. appears to be richer in mineral elements potassium 57.21 mg; calcium 85 mg; sodium 130 mg; magnesium 66 mg; zinc 2.52 mg; iron 2.75 mg; copper 0.68 mg; manganese 2.35 mg. The organoleptic and rheological characteristics of the dessert cream show that the dessert cream made with 90 % of the aqueous extract of *Zizyphus lotus* L. and 2 g of the gelatin got from leather rejects is the most appreciated compared to other gelatin compositions, this dessert cream gives very interesting results.

I. Introduction

The leather industry is one of the most important economic sectors in Algeria. Although we export a large part of our semi-finished leather products, our country has many difficulties sourcing high-quality hides in sufficient quantities on the domestic market. The market for hides has great potential, but local hides are often of poor quality due to the carelessness with which they are maintained. In fact, an awareness of the stakeholders, from breeders to manufacturers, should allow a better valorization of this animal wealth [1].

Algeria has many units of tanneries, but the transformation into finished leather represents only 70 to 80% of the raw weight. Moreover, nearly a quarter of the skins are wasted, at various stages, as non-exploitable materials, namely: useless borders, wool, flesh, skin tissue, etc.....

Taking into consideration this situation in order to be able to value these wastes and to reduce environmental pollution, we propose to make the recovery and the recycling of the raw rejects of the not yet tanned skins and to see all the potentialities of their valorization in terms of products being able to have an interest and even to replace certain components used in the various Algerian dies like chemistry, the cellulose, the polymer, the textile, the cosmetology and more particularly the agro-alimentary one. Therefore, our objective is in the context of recovery of these rejects in the form of gelatin in order to produce a cream dessert enriched in the wild fruit of *Zizyphus lotus* L. while considering the possibility of improving the composition of the creamy dessert.

Zizyphus lotus L., which belongs to the family Rhamnaceae, is very popular in tropical regions; it is known by the vernacular name "sedra". *Zizyphus*

lotus L. is a fruit with a starchy consistency, it contains a high carbohydrate content of 10% of dry matter, and a moisture content of 13%, however, it is low in protein [2]. Zizyphus lotus L is one of the fruits that contain almost all the mineral elements (iron, zinc, calcium, magnesium, etc.), it is notably composed of vitamins such as vitamin A, vitamin C, biotin, etc. It also has a high content of cellulose and pectin fiber [2]. Due to its chemical composition, Zizyphus lotus L is considered as a functional food beneficial to human health and widely used in traditional medicine [2].

From this perspective, the first objective is to recover and valorize the raw waste of the tannery in the form of gelatin. Then, five formulations of dessert creams based on this gelatin and aqueous extracts of Zizyphus lotus L. were developed. Physicochemical and microbiological characterizations were carried out. A study of the impact of the elaborated gelatin and of the aqueous extracts of Zizyphus lotus L. on the texture and consistency of the formulated dessert creams was deepened by sensory and rheological analyses in comparison with a standard industrial dessert cream.

II. Raw materials

II.1. Animal raw material

We note that throughout the process of storage and processing of leather hides, more than 19% of the latter is discarded as solid waste. In Algeria, the large volume of these discharges is about 3000 tons/year, which poses a real problem, concrete and serious [3]. Indeed, because of their richness in collagen and fat [4], they represent a reserve of valuable products that can be integrated into the manufacture of many items.

These commodities of great economic value, which we are trying to account for through certain tests, can perfectly replace or at least complement certain products imported by the sector concerned.

II.2. Vegetable raw material

The wild fruit of Zizyphus lotus L. comes from the wilaya/province of Tlemcen located in western Algeria between 30 °C and 35 °C. The harvest of these fruits was made during the year 2018 during the month of September. The fruits are stored in the freezer at (-5 °C) until further use.

III. Experimental section

III.1. Treatment of waste from raw sheep skins

In this section, we have separately collected the main components of the waste, namely: wool and collagen. To this end, we carried out tests on samples of raw waste from dry sheep skins recovered during sampling.

III.2. Preparation of gelatin powder

In order to prepare gelatin powder we took a certain amount of waste from raw skins. This stage requires two distinct operations namely:

- Washing by using warm water at 25 °C. The pieces of skin are washed well for a while, with renewing the bath and rinsing each time, then cutting the skin into small pieces to increase the contact surface, and facilitate hydrolysis.
- Hydrolysing of the skin by, cooking carried in distilled water at 80°C for 8 hours. The gelatin racks we obtained under went the following operations: filtration, purification, lyophilization, grinding and sieving. Until obtaining a powdered gelatin, which we intend for later use as agelling agent in the dessert cream.

III.3. Preparation of vegetal material

After thawing, the edible parts, seeds and almonds of the fruits of Zizyphus lotus L. were manually isolated, washed, cut and stored until the powder was prepared.

III.4. Preparation of Zizyphus lotus L. powder

The fruits of Zizyphus lotus L. were dried in the open air for 15 days, crushed several times in a knife grinder M 20 - IKA at 20000 rpm, and sieved through a RETSCH AS 200 sieve of 90 µm, which made it possible to obtain a powder of orange-to-brown color that will be used later to prepare the aqueous extract that will be later used in the formulation of the dessert cream.

III.5. Chemical and microbiological tests on gelatin

The pH measurement was carried out by a pH meter [5]. Microbiological analyses have been carried out, according to the standards of the Algerian interministerial decree of 24/01/1998, related to the microbiological specifications of some foods tuffs [6]: the standard requires the total absence of faecal coliforms and staphylococcus aureus.

III.6. Physical characterization of the fruit of Zizyphus lotus L.

The physical characteristics are carried out on 30 fruits taken at random, for which each fruit is determined by: The weight of the whole fruit and pulp (pulp/fruit), the dimensions of the whole fruit (length, volume and density).

III.7. Physical-chemical analyses of the fruit

The moisture content was determined by drying the fruit, of Zizyphus lotus L. in an oven at a temperature of 103 ± 2 °C, up to constant weight [5, 7]. Ash dosing is based on the destruction of any

organic matter put, under the effect of high temperature, in a muffle furnace for 3-5 h at 550°C [8, 9]. The level of soluble solids (TSS) is determined using a refractometer [10]. The pH measurement was carried out by a pH meter [5]. The electrical conductivity that is in correlation with the soluble alt content [9], is determined by a conductivity meter [11, 12]. Titratable acidity was measured, as in [13, 9], by neutralizing the total free acidity contained in the aqueous extract of *Zizyphus lotus* L, and expressed in relation to the malic acid content [14]. The lipid content was determined using a Soxhlet device [15] and, according to the procedure described by AFNOR [16]. Carotenoids are extracted by the method of Moumouni & al. [17]. Polyphenols are estimated by the method of Folin Ciocalteu [18], when they, polyphenols, are oxidized they reduce the reagent of Folin Cioclateu into a complex having a blue color [19, 20]. The measurement of anti-radical activity is based on the fact that the DPPH radical [21] (purple coloring) is very reactive and seeks to stabilize to form colorless derivative in the presence of *Zizyphus lotus* L. The total sugar content is determined by the sulfuric phenol method described by Dubois et al. [22], using a spectrophotometer (UV-VIS, JASCO V-530). Reducing sugars are determined by the method of Miller et al. [23], the reducing function gives an orange color with the DNSA reagent, and the results are expressed relative to a glucose standard range at an optical density of 540 nm. The content of a non-reducing sugar is obtained by the difference between the total sugar content and the reducing sugars present in the sample. The determination of the fiber content is carried out by a self-analyzer called "Fibretec" according to the Weende method [24]. The protein content, that is calculated using the total nitrogen content, is dosed by volumetry after incineration according to the Kjeldahl method [25]. The pectin content has been achieved by the method described by Kalapathy and Proctor [26, 27]. The mineral

constituents [28] (Na, Ca, Mg, K, Zn, Fe, Cu and Mn) were analysed separately using a HACH DR 6000 UV-VIS spectrophotometer. The infrared analysis was performed on a, JASCOFT/IR-4100 Fourier transformer, spectrometer where the frequency is ranged between 400 and 4000cm⁻¹. The antibacterial activity is evaluated by the diffusion technique in a cultivating medium (method known as disc method described by Falleh et al., [29]).

III.8. Method of preparing the aqueous extract of *Zizyphus lotus* L. (intended for the formulation of the dessert cream)

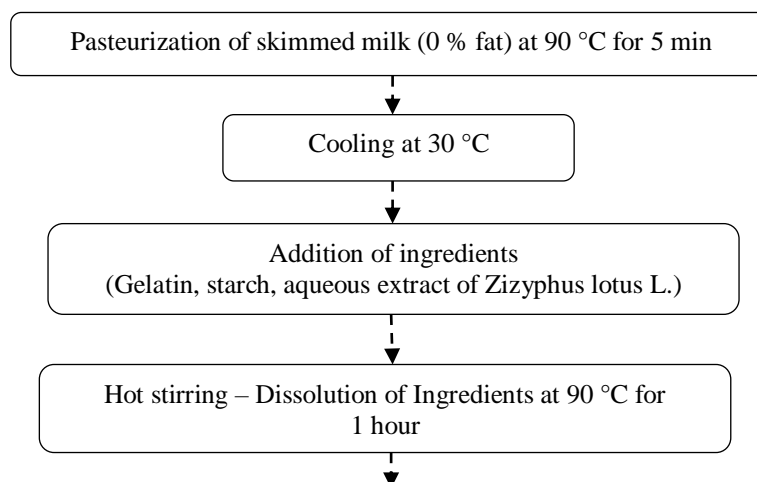
The decoction was also carried out on 50 g of powder with 500 ml of distilled water, this preparation was brought to a boil for 15 min at 100 °C, the extract is then cooled to room temperature, filtered, and then concentrated using a rotary evaporator, stored at (-5°C) in the appropriate buffers.

III.9. Tests to elaborate dessert cream enriched with the aqueous extract of *Zizyphus lotus* L.

We shall respect ingredients and manufacturing diagram of the commercial dessert cream as in table 1and fig 1:

Table1: The formulation of the dessert cream

Dessert Creams	Skimmed milk (0% fat) in ml	Starch in g	Gelatin in g	Aqueous extract in ml
Blank test	50	1	2	0
C1	50	1	2	50
C2	50	1	2	60
C3	50	1	2	70
C4	50	1	2	80
C5	50	1	2	90
C6	50	1	2	100



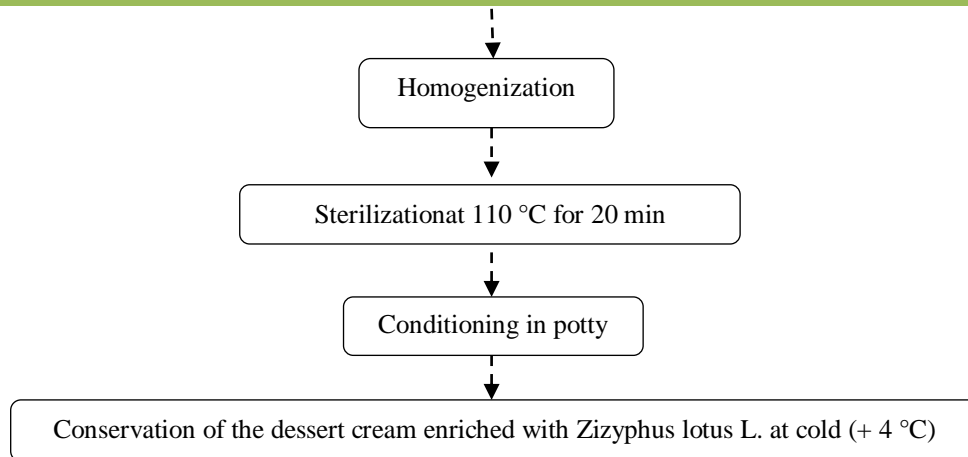


Fig 1: Diagram of the elaboration of the dessert cream enriched with Zizyphus lotus L

III.10. Physical-chemical and microbiological analyses of the formulated dessert cream

The moisture (water) content was determined by drying the dessert cream in an oven at a temperature of 103 ± 2 °C up to a constant weight [5, 7]. The ash was measured according to standard procedures [8, 9]. While the total dry matter content is the result obtained after evaporation of the water from the dessert cream in the oven at 103 ± 1 °C for 24 h [30]. The pH measurement was carried out by a pH meter [5]. The microbiological analyses of the formulated dessert cream were determined according to guirand's method [31]. A sensory

evaluation of the dessert cream was performed by the Friedman test [32].

Rheological analyses of the dessert cream were carried out by measuring the flow properties (viscosity and evolution of the stress in function of the imposed shear speed) and the verification of thixotropic behavior with a rheometer the AR2000 with cone-plane.

IV. Results and discussions

IV.1. Moisture content and dry matter

The moisture and dry matter contents of the raw samples are shown in Table 2.

Table 2: Moisture and dry matter content of solid sheep hide waste at national scale.

	Treated hides and skins	Untanned wastes	Tanned wastes	Hides without values	Mass wastes
Gross weight (tons)	3000	330	218	30	578
Humidity (%)	10	10	13	10	-
% of loss (gross weight)	-	10	8	1	19
Dry matter weight (tons)	2670	294	188	27	509
% of loss(dry matter)	-	10	8	1	19

The analysis of this table 2 shows, at first sight, that only 81% of the dry mass is transformed into finished leather (about 1/4 of the hide is rejected at different stages).

IV.2. Chemical and microbiological tests of gelatin

▪ pH

The pH obtained is equal to 5.5: it is in the standard (identical to the pH of commercial gelatin).

▪ Microbiological analysis

Table 3: Results of microbiological analyses of gelatin

analyzes	Samples	Standards
Fecal coliforms	Abs	NF T 90-413
Staphylococcus aureus	Abs	ISO 6888-3

Overall, the resulting gelatin is considered a healthy product.

IV.3. Physical-chemical characterization of the fruit of Zizyphus lotus L

Table 4: Summary of the results of physical-chemical analyses of Zizyphus lotus L.

Parameters	Average values
Pulp/fruit (%)	39,77 ± 1,06
Moisture content (%)	15,65 ± 0,30
Ash (mg)	3,52 ± 0,02
Level of soluble solids (°Brix)	8 ± 0,23
pH	5,10 ± 0,05
Electrical conductivity (µS/cm)	2,17 ± 0,08
Titrate acidity (g of citric acid for 100 g of product)	1,12 ± 0,03
Lipids (%)	0,97 ± 0,02
Carotenoids (en mg d'EQ QE β-carotène /100 g d'extrait)	0,213 ± 0,02
Polyphenols (en mg d'EAG GAE/100 g d'extrait)	0,471 ± 0,01
Anti-radical activity (%)	0,206 ± 0,03
Total sugars (g/L)	17,19 ± 0,59
Reducing sugars(g/L)	0,89 ± 0,07
Non-reducing sugars(g/L)	16,3 ± 0,55
Cellulosic fibers (%)	1,95 ± 0,50
Proteins (%)	1,87 ± 0,35
Pectin (mg)	2,78 ± 0,53
Sodium (mg/100g)	130 ± 2,30
Calcium (mg/100g MS)	85 ± 1,23
Magnesium (mg/100g MS)	66 ± 1,08
Potassium (mg/100g MS)	57,21 ± 1,20
Iron (mg/100g MS)	2,75 ± 0,02
Zinc (mg/100g MS)	2,52 ± 0,02
Manganese (mg/100g MS)	2,35 ± 0,02
Copper (mg/100g MS)	0,68 ± 0,03

It turns out from these results that the pulp/fruit ratio of *Zizyphus lotus* L. (39.77%) is high. which makes it possible to express parameters of great interest from a food point of view so it makes it possible to estimate the exploitable quantity in relation to the whole fruit [2]. Water content is an index of quality. It reflects a good maturity of the *Zizyphus lotus* L. itself. It also has an important influence on the physical-chemical stability of the product during preservation. The fruit of *Zizyphus lotus* L. has a low water content (15.65%) which facilitates their preservation. On the other hand, the ash content is high in the pulp is 3.52%. These results are similar to those reported by Saadoudi et al. [2] and Boudraa et al. [33] which are: 3.32% and 3.21% respectively for the same species. Soluble solids represent all solids dissolved in water, including sugars, salts, proteins and carboxylic acids [34]. The value obtained by this study is 8 °brix close to those reported by Müller et

al. [35] and Serçe et al. [36], which are 8.1 % and 11.9 % respectively for the *arbutusunedo* L. fruit. According to Wang et al. [37], the brix is 27.9 %; this result differs from the percentage obtained in the present study, this content tells us about the nutritional quality of the sample to be analyzed. The pH is a parameter determining the ability of food to preserve; it is one of the main obstacles, which the microbial flora must cross to ensure its proliferation. The pH obtained from *Zizyphus lotus* L. (5.10) is slightly acidic, which blocks the proliferation of this flora. Electrical conductivity correlates with soluble salt content [34]. According to the analysis performed; the value of the electrical conductivity is 2.17 µS.cm⁻¹. Titrate acidity of the fruit of *Zizyphus lotus* L. is 1.12 g; provides information on the quantity of organic acids present in the sample [35]. In general, they are intermediaries of metabolic processes; they influence the growth of microorganisms and affect the quality of preservation of products [35]. The fruit of *Zizyphus lotus* L. has a low fat content (0.97%). However, our results are much closer to those obtained by Saadoudi et al. [2] (0.84%), for the same fruit. According to the results carotenoids, polyphenols and antioxidants of *Zizyphus lotus* fruit varied greatly between 0.213 mg EQ β-carotene/100 g extract; 0.471 mg of EQ AG /100 g of extract and 0.206%; It is obvious that the high activity of the aqueous extracts of *Zizyphus lotus* L. is attributed to their richness in phenolic compounds, flavonoids and tannins [2]. A study by Kang et al. [38], suggested that polar molecules present in plant extracts contribute to increased anti-free radical activity. *Zizyphus lotus* L. showed a total sugar content of 17.19% which is slightly lower than those found by Saadoudi et al. [2], which is around 24.29 % for the same species, this sugar content is responsible for the sweetness of the food. The reducing sugars of *Zizyphus lotus* L. fruits (0.89%) is included in the range of 0.57 to 2.79% found in the *Zizyphus jujuba* variety by LI et al. [39]. On the other hand, the non-reducing sugar content of the aqueous extract of *Zizyphus lotus* L. fruits is 16.3 g/L which is lower than those found by Benahmed Djilali et al. [40], with averages that varied from 19.18 g/L to 26.90 g/L for the same species. Dietary cellulosic fibers are carbohydrate polymers of vegetable origin [35]. The cellulose content of *Zizyphus lotus* L. fruits is 1.95%, this content is lower than that found by Saadoudi et al. [2], that is 5.41% for the same fruit. It appears from our results that the fruits of *Zizyphus lotus* L. are low in protein with a value of 1.87%. These results are similar to those reported by the work of Saadoudi et al. [2], for the same fruit and which is around 1.43%. It appears from our results that the

fruits of *Zizyphus lotus* L. are low in protein with a value of 1.87%. These results are similar to those reported by the work of Saadoudi et al. [2], for the same fruit and which is 1.43%. The values found for the different mineral elements in our test that sodium is the most abundant with a value of 130 mg/100g, followed by calcium, magnesium and potassium with values of 85 mg/100g, 66 mg/100g and 57.21 mg/100g respectively. The *Zizyphus lotus* L. itself. Also constitutes a source of trace elements, such as: iron 2.75 mg/100g, zinc 2.52 mg/100g, manganese 2.35 mg/100g, copper 0.68 mg/100g. Under Doukani et al. [34], pectin is a component of soluble fiber with interesting technological applications in the gelation of a mixture of fruit and sugar. The pectin content of the fruits of *Zizyphus lotus* L. is around 2.78 %, it is close to that found by Saadoudi et al. [2] is 2.31 %, is included in the interval from 0.57 to 2.79 %

found by Li et al. [39]. According to Doukani et al. [34], these variations in the physical-chemical results of the fruits of *Zizyphus lotus* L. can be attributed to several factors such as the age of the plant, the stage of maturation and the physiological state of the fruit during analysis, geographical location, genetic makeup, soil condition and agronomic and climatic conditions of the crop.

IV.4. Analysis of the infrared spectrum of the fruit *Zizyphus lotus* L

As for the IR treatment, the analysis was carried out on a wavelength range ranging from 4000-400cm⁻¹, which revealed the appearance of characteristic peaks of the adsorption bands are attributed to the presence of the alcohols, the phenols, the alkynes, the amides, the amines, the alkenes and of nitrile's.

IV.5. Antibacterial activity



Fig 2: Photograph of the growth inhibition zones of *Staphylococcus aureus*, *Escherichia coli* and *Pseudomonas aeruginosa* induced by the aqueous extract of *Zizyphus lotus* L.

After 24 hours of incubation at 37 °C, we notice from the figure, the absence of colonies in a circle around the wells indicates that our aqueous extract of *Zizyphus lotus* L. has a bacteriostatic effect compared to the strains used.

IV.6. Physical-chemical analyses of formulated dessert creams

Table 5: The results relating to the physical-chemical analyses of the dessert creams formulated

Pasteurized dessert cream	Samples	Standards [41]
Pasteurization temperature (°C)	100 °C	110 °C
Moisture content (%)	60,21 ± 0,52	-
Ash (%)	2,81 ± 0,02	-
pH	6,4	6,3 - 6,5
Total dry extract, en (g/l)	251,47 ± 0,03	250 - 260
Weight (g)		100
Expiration date		D + 15

We note that these results comply with AFNOR,

1995 standards and this tells us that the dessert cream is of good physical-chemical quality.

IV.7. Microbiological analyses of the formulated dessert cream

Table 6: Results related to microbiological analyses of manufactured dessert cream

Analyzes	Samples	O.J.A.R [42]	Standards
Enterobacteriaceae	Abs	10 ²	ISO 21528-2:2004
Staphylococcus	Abs	10 ²	NF V 08-057
Salmonella	Abs	Abs in 25 g	ISO 6579

The microbiological quality of the dessert cream during the analysis is generally excellent and no pathogenic dose for humans has been detected, it appears that the dessert cream analyzed is of good quality and complies with the standards of the Official Journal of the Algerian Republic [42].

IV.8. Sensory results (Friedman test)

- F1 for taste: $F1 = 2.79$
- F2 for texture: $F2 = 10.28$
- F3 for color: $F3 = 7.07$
- Criterion L: $L = 11.07$ (theoretical value)

So $F1, F2, F3 < 11.07$, it can be said that there is not a significant difference between the 6 products.

From the results of the rankings and the sum R of the taste test scores (taste, texture, color), it was found that the dessert cream formulated C5 with 90% aqueous extracts is the most appreciated by tasters.

IV.9. Rheological analysis of dessert creams formulated

IV.9.1. Measurement of flow properties

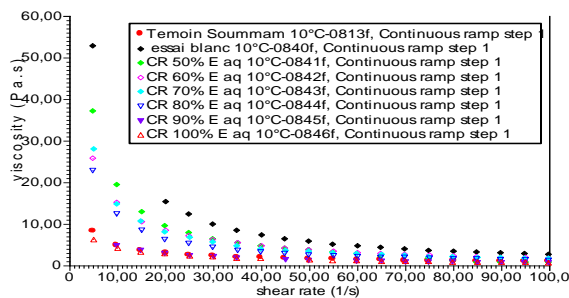


Fig 3: Reactivity of the dessert cream at different concentrations of aqueous extract of jujube tree at 10 °C.

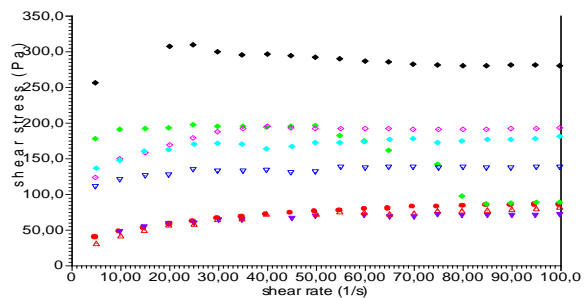


Fig 4: Flow curve of the dessert cream at different concentrations of aqueous extract of jujube tree at 10 °C.

The measurement of viscosity is part of the parameters of the dessert cream; this result is often of prime importance. It depends on three parameters: the gelling agent concentration, the treatment temperature and the pH of the medium. The two curves present the rheological parameters of the dessert cream at different dosages of aqueous extracts at 10 °C.

certain difference that can inform us about the quality of the different dosages of aqueous extracts of jujube.

According to the results obtained, the more the shear speed is noticed, the more the viscosity of the decreased samples with high stress.

Compared between the six samples, we observe that the more the concentration of aqueous extract increases the more the dessert creams become less viscous which is justified by the effect of the aqueous extract but this does not prevent that we have always kept the texture of a dessert cream.

On the other hand, we notice that the samples have the same curve appearance which is translated by the same behavior vis-à-vis the shear speed with a

Based on the results obtained, it is concluded that the two samples with 90 and 100% aqueous extract have the same behavior with the Soummam control (dessert cream marketed).

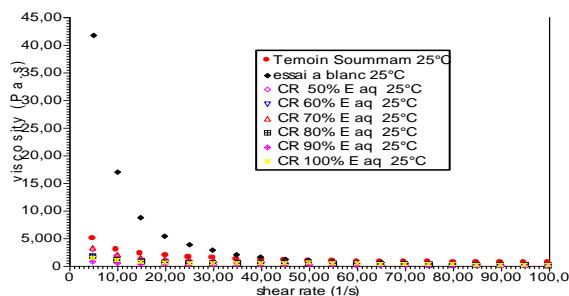


Fig 5: Reactivity of the dessert cream at different concentrations of aqueous jujube extract at 25 °C.

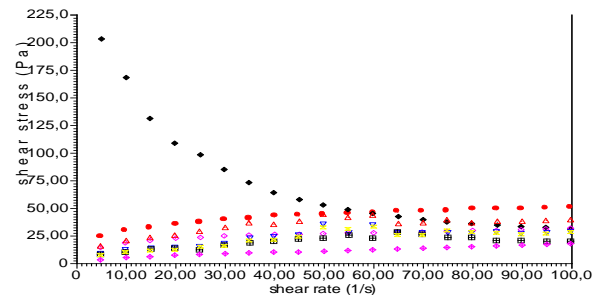


Fig 6: Flow curve of the dessert cream at different concentrations of aqueous jujube extract at 25 °C.

Compared to the behavior of dessert creams at 10 °C and those found at 25 °C, it is observed that the more the processing temperature increases the viscosity and the decreased stress.

On the other hand, it is observed that all samples at 25 °C have the same behavior with the Soummam control (dessert cream marketed).

IV.9.2. Verification of thixotropic behavior

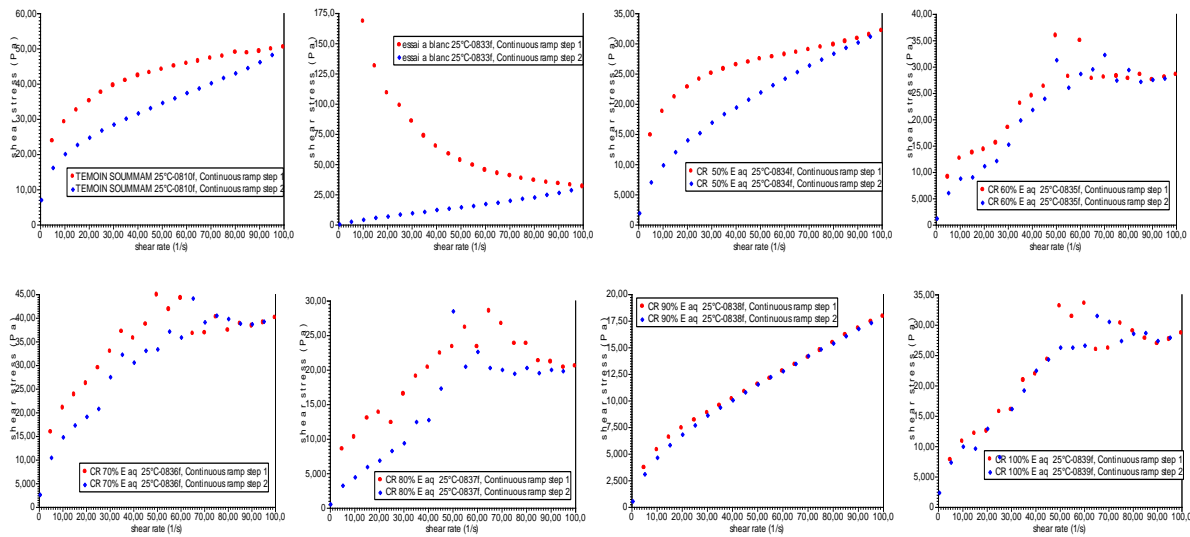


Fig 7: Verification of the thixotropy behavior of the dessert cream at 25 °C.

In our case, the property sought is the texture and consistency of the dessert creams.

According to the graphs in Fig 7, it can be seen that the only cream that has the thixotropy property is the dessert cream with 50 and 90 % aqueous extracts of *Zizyphus lotus L.*, with a predominance of 90 %. The product returns to its original state after mechanical work (shear speed).

We conclude from this analysis that the cream serves 90% aqueous extracts is the most suitable for industrial scale and meets the quality requirements (viscosity, texture and consistency).

V. Conclusion

The leather industry did not wait for the appearance of directive or circular decrees to recycle all or part of its waste in its own manufacture, but the recovery of waste from the tannery presents a pleasure in the triple aspect: Quality control; pollution prevention (environmental protection); valuation of solid co-products.

This study allowed the development of a food formulation of the natural type, and to highlight the possibility of the use of animal skin of the aqueous extract of *Zizyphus lotus L.* as an aroma in the preparation of dessert cream.

Very interesting results at the end of this work from the point of view of:

- Economic: by recovering the fruits of *Zizyphus lotus L.* into high value-added products and raw skin waste.
- Nutritional: by the elaboration of natural food products based on the extract of the fruits of *Zizyphus lotus L.* and gelatin made by raw skin waste.
- Organoleptic: by the possibility of developing a

dessert cream with a good texture and a pleasant flavor.

- Hygienic: by highlighting its antibacterial activity vis-à-vis the strains previously used.

It appears possible through this study, to produce a dessert cream by a natural aroma (the extract of the fruit of *Zizyphus lotus L.*). This extract enriches the food products elaborated with mineral elements, and vitamins. This makes it possible to consider the formulated product (dessert cream) as a functional food.

VI. References

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