

# An Agrofood waste physicochemical characterization for its valorization

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# ABSTRACT/RESUME

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Key Words:

Byproduct; Coconut oil; Distilled fatty acids; Liquid soap; Sunflower oil; Abstract: This study was focused on biorefinery byproducts mainly, based on distilled fatty acids from sunflower oil and coconut oil; for their valorization. They were characterized oleochemically, for sunflower oil, the Iodine Index: 120 g  $I_2$  / 100g fat noted the unsaturation of its fatty acids and the Refractive Index (RI) n 20: 1417 revealed its siccativity. As for the coconut oil, the saponification index: 250 mg KOH / g fat was on the rise, insured its ability to promote saponification and the IR  $n_{20}$ : 1. 448 attested its unsiccativity; polyunsaturated fatty acids of these both oils were revealed via gas chromatography. These tests results were promoted for the development of a liquid soap, that was characterized physicochemically via the residual base: 0.02%, total fat content: 25.52%, residual glycerol content: 14.68%, pH: 07.90, lyophilic hydrophilic balance: 20. 02, surface tension: 34.10 dynes / cm, concentration micelle critic: 05. 103 mol /l and spectral methods such FTIR, XRD and SEM corroborated these analyses; which were confirmed the presence of carboxylic fatty acid salts. This work was conclusive, resulted in a liquid soap loaded with 14.68% of vegetable glycerin with moisturizing, emollient and softening properties, in conformity with hygiene standards.

#### I. Introduction

Biotechnology was developed the innovations necessary for the new chemistry [1] which, was Involved the development of new processes in particular byproducts biorefineries, which were poorly valued [2]. Biomolecules of "green chemistry" was diversified sectors of traditional chemistry, but could only develop fully with intense innovation effort [3]. Now, the main by-product recycled methods concerned oleochemical applications [4] in fact, a trend towards agro-based surfactant was evident [5-6]. New features sparked great interest in still underutilized for scientific research sources [7-8]. Indeed, from these oilseeds,

it was possible to confer new phytosanitary properties to the formulation [9-12].

Currently, users were more sought about the impact of their purchases on the environment [3]. In response to the registration of the Regulation and Authorization of Chemicals (REACH), a new surfactants were developed from renewable sources natural [13]. Vegetable Oils (VO) was interested properties related to unsaponifiable (excellent antioxidants) to improve and restore skin structure [14,15], because they were excellent lubricating agents capable of protecting skin [5, 8, 16]. Nevertheless, interest in biosurfactants developed from Fatty Acide (FA) remains limited [17], fortunately currently, a return to soap in favor was obvious, for biosurfactants based only on natural products [18].

In this context, our study was focused on oilseed biorefinery by-products mainly, based on Distilled Fatty Acids (DFA), from on Sunflower Oils (SO) and Coconut Oil (CO) as our Lipid Matter (ML) for valorization. They were characterized their oleochemically AFNOR standarded [19]; and their polyunsaturated FA (PUFA) were revealed via Gas Chromatography (GC). These tests results were promoted for the development of a liquid soap, (LS), loaded with vegetable glycerin. LS was subjected to physicochemical analyzes inherent surfactant AFNOR standardized [20], resulted on Carboxylic FA Salts (CFAS) and was corroborated via spectral methods essentially based on FTIR, XRD and SEM. Noted that the effect of these disinfecting agents was not studied in detail [21].

#### **II. Materials and Methods**

The LM was a mixture of DFA from on SO and CO provided via the Algerian biorefinery called CEVITAL located in Bejaia (East of Algeria).

#### **II.1. LM characterization**

The LM was characterized oleochemically AFNOR standardized, essentially based on Refractive Index (RI)  $n_{20}$ , Saponification Index (SI), Iodine Index (II) and Density (D<sub>20</sub>). Oleochemical constants of DFA from on SO and CO. The results were recorded in Table1.

#### **II.2. LM Structural Identification**

#### Gas Chromatography (CG)

Wolff most Accorded to often the DFA composition was determined after transesterification of triglycerides into Methyl Esters (ME) followed by their separation by on a Carbowax column of length L = 50 m and diameter D = 0.25 nm GC [22], component a chromatograph, type Chrompack CP9001. Equipped with an ionization flame injector with the following conditions: the oven temperatures of the injector and the detector were respectively from 150 to 200°C with a gradient of 5 ° C / min; the carrier gas was helium (He) at 2ml /min the volume injected into the column, with about 0.5 ul and that at the instant of the injection of the ME, with an analysis time of thirty (30, 00) minutes. Chromatogram was recorded in Figure 1.

# II. 3. LS synthesis

The LS synthesis process was thermochemical at 65  $^{\circ}$  C, under atmospheric pressure, with a few grains of pumice stone; to promote reaction upon heating and to regulate boiling; carried out under alkaline

conditions, called saponification; which consumed two (02,00) hours [23].

The absence of a authentic cauldron was replaced by artisan cauldron, consisted of a pitcher (stainless steel) reserved for the LM placed in another large pot which served bath. Everything was installed in a jacket with a thermostatic hotplate; this traditional installation was topped with a dosing burette The artisan cauldron for saponification was recorded in Figure 2.

#### **II. 4. LS protocol experimental**

The LS was saponified from 25% LM (mixture of DFA from on SO and CO), generated using 1.75 M ethanoic KOH solution: with percentages dependent on those of the LM and of variable concentration for saponification [24-25]. Noted that, the SI was used to calculate the quantity of potash to be added was an average and can vary empirically. LM and KOH were at the same temperature, when they were in contact. Therefore, KOH was recommended to reduce it by at least 5% (safety margin); usually, for reasons of efficiency potash was used after 24 hours, since its preparation and that, for complete consumption of potash by saponification; however, some oils remained in the unsaponified LS. The quantity of potash was gradually introduced and stirred with a necessary circular motion to trigger the saponification which favored the hydration of the LS [26-27]. The adjustment pH was checked with pH-meter, throughout the operation until the KOH was absorbed. Figure 3 showed LS saponified.

# II. 5. LS characterization

LS was subjected to physicochemical analyzes inherent surfactant AFNOR standardized, essentially based on Residual Base (RB), Total Fat Content (TFC), Residual Glycerol Content (RGC), Hydrogen potential (pH), Hydrophilic Lyophilic Balance (HLB) and Critical Micellar Concentration (CMC):

#### **Residual Base (RB)**

The RB LS was assessed un-contributory basis for saponification.

#### Total Fat Content (TFC)

The TFC was obtained via the LS decomposition, with the mineral acid.

#### Residual Glycerol Content (RGC)

The RGC in LS, collected in a graduated tube.

# Hydrogen potential (pH)

Evaluated the LS acidity or basicity, using digital 212 HANNA pH meter, evolution pH of LS in function of KOH; was showed in Figure 4.

#### Hydrophilic Lyophilic Balance (HLB)

The HBL was derived by the incremental method of (Davies),

**Superficial Tension (ST):** Determined via the ring method of Nouy [5].



#### **Critical Micellar Concentration (CMC)**

The CMC was deduced from curve of TS, represented by ST= f (CMC); the result was showed in Figure 5.

The Association Vegetable Chemistry (AVC) currently worked on the byproducts qualification, via LS structural identification [28].

#### **II. 6. LS structural identification**

LS structural identification was supported by spectral methods essentially based on FTIR, XRD and SEM.

#### Infrared Spectroscopy (FTIR)

The FTIR measurements were recorded in a KBr phase by using "GAN 1000 PC Perkin Elmer 4100 FTIR" type coupled to digital computer with the spectra between 4000 and 400 cm<sup>-1</sup>; recorded in Figure 6.

#### X-ray diffraction (XRD)

The diffractometer was the PAN-alytical type: XPERT-PRO, copper anticathode ceramic tube, generator power at RX: 40 mA, 45 KV, equipped with data acquisition software; recorded in Figure 7.

#### Scanning Electron Microscopy (SEM)

LM was dried in the open air, and then introduced into a Quanta 650 SEM-type, high voltage FEI. The observation were made under a magnification (MAG): 300 x equipped with an ETD detector, secondary electron, with a distance WD: 10.6 mm, a spot of 5.0 and a horizontal scale of 400  $\mu$ m; imaged on Figure (8a). The perception of lipid pores was carried out, sub-enlargement (Mag): 1000 x, WD: 10.8 mm and a horizontal scale of 100  $\mu$ m; imaged on Figure 8 (b).

#### **III. Results and discussion**

#### **III. 1. LM characterization**

Lipid treatment required knowledge of its oleochemical properties [29], was resulted with in perfect agreement in the Codex Alimentarius with the appreciable length PUFA of the SO approved, via the II: 120 g I2 / 100g fat, attested it a frank fluidity and the IR n 20: 1.417, was confirmed its siccativity. These specificity was maked an excellent lubricant [26,30-31].

As for CO, showed an increase in SI: 250 mg KOH / g of fat, confirmed its capacity for saponification, supported with excellent chemical stability and the increase in RI n 20: 1.448 testified to its non-siccativity; hence its combination with the LM recommended for this purpose to promote the LS gel form, by saponification [32]. Oleochemical

constants of DFA from on SO and CO were recorded in Table1.

*Table 1.* Oleochemical constants of DFA from SO and CO

Oleochemical constants	DFA SO	Standards	DFA CO	Standards
<b>R I</b> (n <sub>20</sub> )	1.417	1.470	1.448	1.448
SI (mg KOH/g fat)	198	188-198	250	250-264
$\begin{array}{c} II \; (gI_2  /  100g \\ fat) \end{array}$	120	120-134	8.00	6-8
<b>D</b> <sub>20</sub> eau 20°C)	0.922	0.918-0.923	0.845	0.845-0.846

#### **III.2. LM structural identification**

#### Gas Chromatography (GC)

For SO, the chromatogram detected a high content of essential PUFA linoleic acid C<sub>18: 2</sub> (omega-6), supplemented with a mono-unsaturated FA, C<sub>18:1</sub> oleic acid (omega-9), was totalized more than 85% of its composition, distinguished by wetting agents  $(C_{8-10})$ , detergents  $(C_{12}-C_{16})$ , emulsifiers or softeners  $(C_{18-22})$ , [26]. The literature affirmed that in plants: linear  $C_{18}$  chains were the most widespread ensured good biodegradability [33]; this distinction offered appreciable spectrums of them industrial applications, where surfactants were in great demand and used either as an excipient or as a source of active substance [30, 33].

CO contained a short chain rich in Caproic acid C  $_6$ : 0, Caprylic acid C $_8$ : 0, and medium chain Capric acid C $_{10}$ : 0, Lauric acid C $_{12}$ : 0, Myristic acid C $_{14}$ : 0, Palmitic acid C $_{16}$ : 0. conferred it an excellent chemical stability [33], as well, two long chains, C $_{16}$  and C $_{18}$ , conferred it a lipophilic character. Moreover, CO included higher glycerol content than all VO and an exceptional moisturizer for skin [34].

Today, the needs of the "green revolution" lean in favor of these liquid oils (SO) and semi-solid (CO), as they argued for the development of hydrophobic surfactant with variable chain length, mainly for the cosmetics [33]. The chromatogram was recorded in Figure 1.

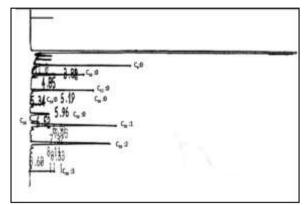


Figure 1. Chromatogram of DFA from SO and CO

#### **III.3. LS** synthesis

The LS synthesis was occurred under alkaline conditions called saponification, was accomplished with the artisan cauldron, recorded in Figure 2. The contents of the reaction thermochemical at  $65^{\circ}$  were stirred by manual agitator (spatula) so that the exchange surface between the reactants was high.

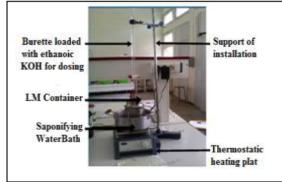


Figure 2. Artisan cauldron for saponification

VO interested properties for applications in the fields of cosmetics, detergents or lipid chemistry [29], which justified their choice studied in this work by their byproduct oleochemical to develop biosurfactants, very high added value [32-33].

SÓ recognized as an excellent lubricant favorable to the development of LS, with softening properties and good biodegradability [33].

CO contained Lauric acid, recognized as antimicrobial and antiviral, its use was to confer antiseptic properties to LS [32, 5]. The increase in Lauric acid resulted in a highest SI, promoted a foaming and washing; which resulted in a surfactant (LS) loaded with vegetable glycerin, vitamin E and antioxidant [34-35]; reason for their mixture suggested by the literature. Noted that the assets of most surfactants were used in the applications were mixtures, hence their attraction to pharmaceutical applications [17]; as a renewable source of supply [36].

In the past, soap was one of the most used hygiene products [5]; which was developed in solid form, constituted a reservoir of microbial germs [36],

hence its abandonment [37]. The reason why LS was formulated in order to develop a "chemistry and biomaterials" activity through traditional industries [34-35] 100% ecological; contained the natural unsaponifiable with unique properties for the skin; which was against the industrial process reducing the quality of LS [38].

Unstable fatty acids (constituted the ML), on which a base was reacted to cause the formation of watersoluble alkaline salts of general formula: (R-COOK) and glycerol [5] and it was noted that soap detergency was due to RCOO [39]. the unsaponified and glycerin remaining will constitute the "superfat" an added value to the finished product [17]. Glycerin helped restore the skin's natural protective layer that soaps can alter, they was deposed a film on the skin; thus the skin will be hydrated for longer [40]. Recognized for their high content in alpha-tocopherol or vitamin E, and phytosterols, antioxidant properties, those who made "multi-purpose" oil in the cosmetic and pharmaceutical industry [26, 30, 41].

The Figure 3 clearly showed the deposit of LS translucent characterized by the absence of coalescence in the dispersed phase of the formulation [36, 42]; an appropriate amount of water was added to the LS, greatly reducing skin dehydration so, the LS formula was related to mild shampoos, classified biosurfactants [43-44].



Figure 3. LS saponified

# **III.4. LS physicochemical constants**

The physicochemical constants demonstrated the presence of LS AFNOR standarded essentially based on Residual Base (RB), Total Fat Content (TFC), Residual Glycerol Content (RGC), Hydrogen potential (pH) Hydrophilic Lyophilic Balance (HLB) and Critical Micellar Concentration (CMC):

#### **Residual Base (RB)**

Well designed LS was without any base in its original condition [26], the tolerance was recommended to 0.02%; ensured its conformity [44].

Moisture Content (MC)



The MC value of 51% water was a skin hydration; noted the refinements that the LS possessed [32].

#### **Total Fat Content (TFC)**

From a preparative point of view, the carboxylic function provided a lipophilic biosurfactants explained the remaining 25.52% of FA, corresponded to unsaponified so, justified free alkaline deficiency in the LS [32]. This excess was only beneficial for the skin. For this reason, the 5% reduction in alkalinity was called "lipid ratio" [45]; agreed that superfat agents protect the epidermis from irritations [5, 20, 46 -47].

#### **Residual Glycerol Content (RGC)**

The RGC value of 14.68% was vegetable glycerin generated by saponification resulting via LS [6, 44]; ensured an occlusive film (barrier) to the epidermis and hydration that protected the skin from irritation from external aggressions [48-49].

#### Hydrogen potential (pH)

The pH value of 7.9 suggested the partial transformation of the LS in soap [39], certified its alkalinity [50]. Cosmetic treatments were alkaline, to fight against excess acidity and stimulated the sebaceous glands to promote compatibility with the skin lining [51-52].

pH = f (soap content) indicated that hydrolysis was complete and the result was similar to literature [44], was recorded in Figure 4.

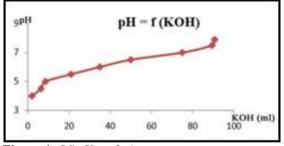


Figure 4. LS pH evolution

#### Hydrophilic Lyophilic Balance (HLB)

The HLB value of 20.02 indicated that LS was an emulsifier [44].

#### Superficial Tension (ST)

For the formulation of a cleaner to be effective, it was necessary to control the ST between the solvent and the stain. The ST value of 34.10 dyne / cm

indicated that the longer the carbon chain, the more hydrophobic it was; [2, 53], it was for helped to remove dirt.

#### **Critical Micellar Concentration (CMC)**

The 510-3 mol / L value was translated into log c = 2.3 at 25 ° C, confirmed the presence of CFAS, was resulted electrical repulsions, which prevented the formation of micelles in the LS [5, 4, 53]; thus corroborated the steps of removing organic dirt and microorganisms [54]. Noted, that these properties had a direct impact to evaluate potential applications of LS [17], The CMC was deduced from curve of TS, represented via ST= f (CMC); the result was showed in Figure 5.

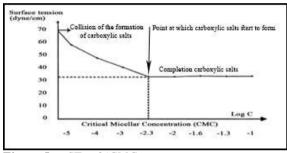


Figure 5. ST = f(CMC)

#### **III.5. LS structural identification**

# Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR showed absorption bands with different vibrations of the CFAS. A prominent absorption band at 3000 cm<sup>-1</sup>, characteristic of the vibration  $\gamma$  OH corresponding to the LS liquefaction of water and the endogenous of the LM [55-56].The valence vibration  $\gamma$  CH, CH2, CH3 was found to 2960 cm<sup>-1</sup> and 2356 cm<sup>-1</sup> characteristic of the CFAS, was corresponded to the LS [5, 57]. Accorded to the bond vibrations C = C observed in the area 1560 cm<sup>-1</sup>, was revealed a predominance of the hydrocarbon chain of a monounsaturated (oleate) or di-unsaturated (linoleate) ester, was confirmed that the LS resulted from saponification of VO [5, 22]. Result was imaged in Figure 6.

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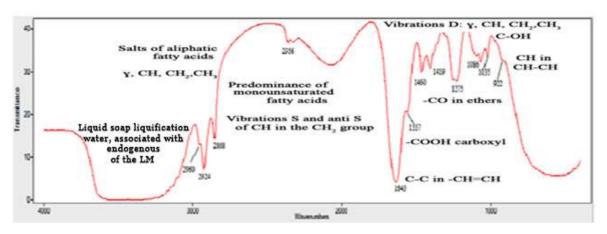


Figure 6. Fourier Transform Infrared Spectroscopy (FTIR) of LS

#### Diffractometry (XRD)

XRD showed varying forms The crystal polymorphism. This observation was resulted from the spatial organization of the hydrocarbon chains (lateral arrangement) and from the stacking of the triglyceride molecules in strata (longitudinal arrangement). There were two main categories of crystalline varieties: those were corresponded to a compact and compact arrangement of the chains were due to specific interactions ( $\beta$  and  $\beta$  'forms); was defined the soap phase and that associated with a looser arrangement ( $\alpha$  form) by loss of these interactions; the most compact forms were generally more stable, defined by the amorphous phase reflecting the lipid phase of LS [58-59]. Result was showed in Figure7.

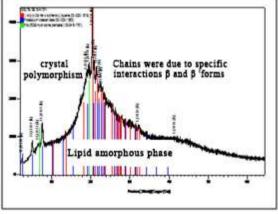
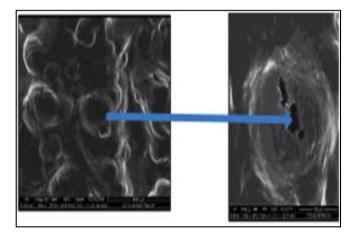


Figure 7. Diffractometry XRD of LS

#### Scanning Electron Microscopy (SEM)

SEM was one of the most appropriate tools to clearly confirm the morphology of LS, mainly glycerophospholipids amphiphilic bilayers form and consisted of biological membranes or spherical vesicles called liposomes. The cosmetics industry used them to encapsulate the active substance suspended in an aqueous medium [60]. This system was presented some advantages, such as the protection of the trapped substances and the ability to incorporate the active ingredients in different vesicles. [36]. Figure 8 (a) was developed to illustrate the overall structure of the lamellar isotropic aqueous phase for most soaps and detergents formed of spherical or cylindrical micelles swollen normal or reversed, constituting a model soap biosurfactants [44].

Figure 8 (b), was developed to highlight the porous structure of a soap intended for the release of an active principle due to the diffusion of fluids (by phagocytes), which was created by erosion, either by lipolysis, or by enzymatic hydrolysis [44]. Thereby cause, a structuring of the interface which was made the resilient surface, so much more stable [4].



*Figure 8. (a)Overall structure of LS* SEM view via SEM (b)Porous structure of LS view via

#### **IV.** Conclusion

The oleochemical study of DFA resulted in values in accordance with the AFNOR standard; the GC support revealed the PUFA profile of the LM, composed of SO and CO, having an important role in many physiological functions, including those of the skin. For SO, the chromatogram was detected the detergent properties of its the long, medium and short chains. As to CO was distinguished via Lauric acid C12: 0, recognized for its antiseptic properties and with two long chains,  $C_{16}$  and  $C_{18}$ , ensured it lipophilic induced softening properties, with an excellent chemical stability; noted that CO contained higher glycerol content than any VO, which made it an exceptional moisturizer for the skin. This distinction offered them significant spectrum of industrial applications of surfactants.

The purpose of this study was part of a context marked by regulation (REACH); which led us to the synthesis on a 100% natural LS by saponification via KOH; manufactured with a minimum of chemical reagents, to remain in a concept of green chemistry, based on biodegradable Algerian DFA, loaded with vitamins, and antioxidants, which have retained their therapeutic properties to complete the REACH requirements. LS defined surfactant, was specified via 14.68% glycerol, as well a superfat justified via the remaining 25.52% of FA, comforted by pH: 07.90, a Lyophilic Hydrophilic Balance: 20. 02, surface tension: 34.10 dynes / cm. Support for methods such as FITR, XRD and SEM revealed a porous lipid structure capable of phagocitating antiseptics, hence its ability to develop antiseptic formulations.

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