Valorizing Olive Oil Mill Wastewater: Transforming Waste into Natural Soaps

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Abstract. In this research, the main objective is to find a solution to the problem of olive mill wastewater (OMWW). This solution involves the recovery of liquid waste produced by the crushing units, which contain a significant amount of oily residues despite their initial treatment. The concept is based on separating these discharges into aqueous and oil phases and using the latter to manufacture natural soaps.

The liquid waste from the extraction of olive oil has a significant content of oily residues, characterized by a very high acidity (2.73%), exceeding the value of edible olive oil, as well as a high saponification index (186.2 mg KOH/g), making it an ideal source of fat for saponification.

The results of the valorization of the OMWW in soap manufacturing reveal several significant elements. First, following the characterization of cold and hot products, a clear preference emerges in favor of soaps made using the cold process. In addition, the production yield is notable, with a rate of 94% for cold saponification and 89.9% for hot saponification, highlighting the efficiency of the process. Finally, the study highlights the importance of the quantities of fatty acids used in the formulation of soaps. An optimal formula is identified, comprising 40% oils recovered from the OMWW, 30% olive oil, 20% cocoa oil, and 10% castor oil, demonstrating the need for a precise balance to obtain quality soaps.

This study solves the problem of the OMWW by reusing them to make natural soaps, thus reducing industrial waste. It also opens up new economic opportunities by creating a profitable and environmentally responsible production chain, promoting the transition to a circular economy.

Keywords: Olive mill wastewater, Valorization, Cold soap, Oleic acid, Sodium hydroxide, Saponification

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I. Introduction

The olive oil mill wastewaters represent a significant environmental challenge due to their high acidity and high content of oily residues. According to the Codex Alimentarius [1], these oily residues are harmful to human health and are usually released into the environment without any use. However, this study aims to make the most of these OMWW by transforming them into two types of natural soaps: a cold process soap made by a batch process and a hot soap made by a continuous process.

The technique used to separate the oily residue from the aqueous residue is the natural settling of the samples. This simple and ecological method makes it possible to recover the oil fraction, which will then be transformed into soaps by well-controlled saponification processes. Tags: The aim is to demonstrate the potential for the recovery of the OMWW, a waste product often considered an environmental burden, by converting it into a value-added product, namely natural soaps.

This innovative approach will help reduce OMWW's environmental impact and develop a production chain for ecological soaps from renewable resources. The results of this study could pave the way for new opportunities to add value to the by-products of the olive industry, thus promoting a circular economy in this sector.

II. Materials and Methods

2.1 The OMWW Sample Source

The OMWW samples were collected primarily at the semi-modern crushing unit (MSU)) located in Imzouren (northern Morocco [2,3,4]. It should be noted that these crushing units do not use any chemical additives during the production process.

Samples were taken from a well-homogenized storage basin. In order to keep their initial characteristics, they are quickly transported to our laboratory at the National School of Applied Sciences in Al-Hoceima and then stored at a temperature of 4°C [5,6]. The sample was stored in closed plastic containers at room temperature.

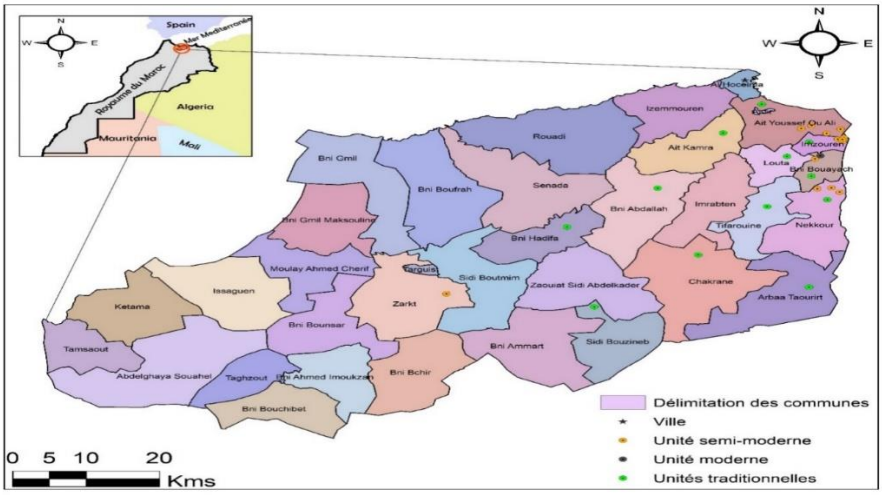


Fig.*1*: Map showing the location of industrial units in the province of Al Hoceima.

2.2. Analysis of the Separate Oil Phase

To ensure that our fatty acid used in the soap manufacturing process complies with the International Organization for Standardization (ISO) method, a chemical characterization of the oil recovered from the OMWW is necessary. The characterization parameters adopted are saponification number, acid number, peroxide value, and iodine value.

2.2.1. The Saponification Index

The saponification index (Is) is the mass in mg of potash (KOH or NaOH) sufficient to convert an ester to carboxylate ions and alcohol [7]. The determination of this index goes through three steps:

- ***Preparation of our Olive Acid:***

The oil recovered from the OMWW is insoluble in water, which requires it to be dissolved in a solvent. We put 4 g of our olive acid in a beaker, then add 100 ml of solvent (ethanol + ethyl oxide) and shake until our fat dissolves.

- ***Determination of the Saponification Index:***

In a beaker, we pour 10 ml of our fat solution, add 25 ml of alcoholic potash (c = 0.5 mol/l), and place the beaker in a boiling water bath at 100 °C for 30 to 45 minutes. After adding 2 to 3 drops of phenolphthalein, the excess potash is dosed with hydrochloric acid (c= 0.5 mol/l) until the phenolphthalein is discolored. The test is repeated two to three times.

- ***Realization of the Witnesses:***

It is important to conduct control tests to determine the precise concentration of alcoholic potash. Add 25 mL of alcoholic potash and 10 mL of solvent to a beaker, milk it under the same operating conditions as the test, and dose until the phenolphthalein is discolored.

The saponification index is calculated using the following equation:

$$I_s = \frac{(V_t - V_e) \times C_{HCL} \times M_{KOH}}{m}$$

With:
$\boldsymbol{I_s}$: Saponification index.
$\boldsymbol{V_t}$: Volume paid to the control (ml).
$\boldsymbol{V_e}$: Test volume (ml).
$\boldsymbol{C_{HCL}}$: Concentration of hydrochloric acid (mol/l).
$\boldsymbol{M_{KOH}}$: Molar mass of KOH (56.1 g/mol).
$\boldsymbol{m}$: the mass of oil weighed (g).

2.2.2. Acid Number

This index represents the amount of potash (mg) needed to neutralize the free acidity of fat, and it increases over time [1]. To determine this index, follow these steps:

- ***Preparation of the Fat Solution*:**

We followed the same step to determine the saponification index.

- ***Determination of the Acid Number:***

We put 100 ml of our sample into a beaker, adding 10 ml of alcoholic potash and 2 to 3 drops of phenolphthalein. Then, titration with hydrochloric acid until the solution is discolored. The test is repeated two to three times.

- ***Realization of the Witnesses:***

Add 10 ml of alcoholic potash and solvent to a beaker with 2 to 3 drops of phenolphthalein and dose with hydrochloric acid until the solution is discolored.

The following equation determines the acid index:

$$I_a = \frac{(V_t - V_e) \times C_{HCL} \times M_{KOH}}{m}$$

With:

$\boldsymbol{I_a}$ **:** Acid index.

Vt: Volume of HCL solution poured into the control (ml).

V: Volume of HCL solution assay(ml).

CHCL: Concentration of hydrochloric acid (mol/l).

MKOH : Molar mass of N (56.1g/mol).

m: the mass of oil weighed (mg).

2.2.3. Peroxide Value

The peroxide value allows us to determine the freshness of the oil. It is expressed as the milliequivalents of active oxygen in 1 kg of product [8]. It is determined by the following volumetric method:

- Dissolve 1 g of our recovered oil in a solvent (a mixture of acetic acid and chloroform).
- Add 15 ml of the potassium iodide solution.
- Place in the dark for 5 minutes.
- Add 60ml of distilled water.
- 1ml of a starch solution.
- Titration with sodium thiosulfate solution (Na2S2 O3) until the color becomes transparent.

The test is repeated two to three times, and a control test is required. The following equation gives the peroxide value:

$$\text{Peroxide value } (méq\ d'O_2/kg) = \frac{(V_e - V_t) \times 1000 \times N}{m}$$

With:

Vt: Volume of Na2S2 O3 solution poured into the control (ml).
V: Volume of Na2S2 O3 solution assay(ml).
N: Normality of the Na2S2 O3 solution.
m: the mass of oil weighed (g).

2.2.4. Iodine Value

The iodine value represents the weight of iodine (g) fixed on 100g of fat. The following protocol determines it [9]:

- Put 0.5 g of our recovered oil in Erlenmeyer.
- Add 20 ml of cyclohexane to solubilize the oil.
- Add 25 mL of Wijs reagent and shake.
- Leave the solution in the dark for 60 minutes, shaking every 15 minutes.
- Add 20 ml of potassium iodide (to 100 g/L).
- Add 100 ml of distilled water.
- Cap and shake vigorously for 5 min.
- Dose with sodium thiosulphate until color changes.

The test is repeated twice, and then a control is performed under the same conditions.

$$I_i = \frac{(V_t - V_e) \times N \times 100}{m}$$

With:

I_i : Iodine value.

Vt: Volume of sodium thiosulfate solution poured into the control (ml).

V: Volume of sodium thiosulfate solution test (ml).

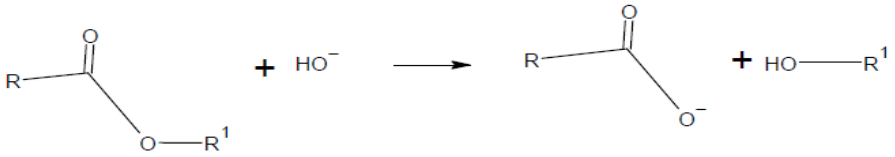
N: the normality of sodium thiosulfate (mol/l).

m: the mass of oil weighed (mg).

2.3. Saponification

2.3.1. Principle and Techniques of Saponification

Saponification is a slow and complete chemical reaction that converts an ester into carboxylate ions and alcohol. It is the hydrolysis of the ester in an alkaline medium by a base (KOH or NaOH) between 80 and 100 °C. It is the opposite of esterification. This reaction allows the synthesis of soap. Initially, this reaction converted a mixture of glyceride and strong base into a mixture of soap (or fatty acid salt) and glycerin, hence the name. It was discovered in 1823 by the French chemist Michel-Eugène Chevreul [10], which showed that triglycerides can be considered a chemical bond between glycerol and fatty acids. The primary saponification reaction is Ester + hydroxide ion carboxylate + Alcohol.



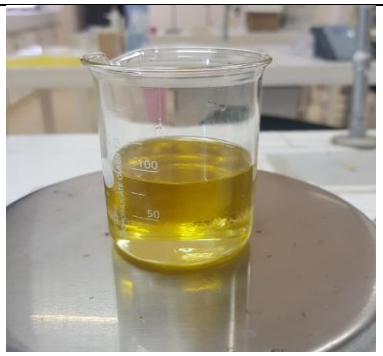
The first step in soap making for the two different processes is the dissolution of caustic soda in distilled water. The amount of sodium hydroxide used is determined by the soap calculator or by multiplying the saponification number by the fatty acid mass. After the sodium hydroxide has dissolved, the solution is allowed to sit and relax.

2.3.2. Cold Saponification

Cold saponification is the most widely used technique in natural saponification because of its simplicity, economic efficiency, and product quality.

The manufacturing steps followed during our work are those published by Martial Gervais Oden Bella, subtitled: "Soaps and detergents" [11].

Table 1: *The stages of soap making***.**

Steps	Description	Images shown
Sodium Hydroxide Preparation	The weighed sodium hydroxide is poured into the water (not the other way around). This reaction will increase the temperature of the solution, stirring gently until the sodium hydroxide is dissolved. Let the temperature drop to around 40°C.	 **Fig.2 :** The NaOH solution.
Weighing the recovered oil	In a beaker, the recovered oil is weighed and heated to a temperature of 40°C.	 **Fig.3:** Weighing the recovered oil.
Blend through blender	In a container, 200ml of recovered oil is placed, and the sodium hydroxide solution is carefully added drop by drop, mixing regularly and in the same direction until a uniform semi-heavy paste is formed (the saponification trace).	 **Fig.4**: The saponification trace.
Release	Pour the batter into a silicone mold and cover with plastic wrap. Let it dry for 24 hours before removing from the pan.	 **Fig.5**: Pouring the paste into the silicone mold.

2.3.3. Hot Saponification

This method is based on cooking the fat and caustic soda mixture at 80° to 100°C during the entire process. The steps to follow in this method are:

- Pour 22ml of oil recovered from the OMWW into a 250ml flask.
- Add 20 ml of caustic soda solution with a concentration of 7.5 mol/l.
- Add 15 ml of ethanol with a concentration of 16.94 mol/l to promote contact between the reagents.
- Add 2 to 3 grains of pumice stone to regulate the boiling.
- Place in a reflux heater for 1 hour at 80°C.
- Pour the mixture into a beaker containing a cold solution saturated with sodium chloride.
- Filter the vacuum mixture using the Büchner method.
- Rinse the soap with distilled water to remove soda, salt, and glycerin.
- Remove as much moisture as possible by drying soap in a double boiler on a hot plate at a temperature of 80°C.
- Pour the soap into the silicon molds and let it air dry.

Fig.6: Photograph of reflux heating.

2.4. Analysis of Soap Products

2.4.1. Determination of Hydrogenated Potential

pH is a significant indicator, so soap pH must be basic. Dissolve 0.5g of soap in 150ml of distilled water and then measure the pH with a pH meter.

2.4.2. Determination of Moisture Content

The percentage of humidity is a critical indicator of the quality of the soap produced. It is determined by calculating the difference between the mass of soap dried in the oven at a temperature of 105°C for 3 hours and the mass of soap dried at the same temperature but only for one hour [12].

2.4.3. Determination of the Foaming Power of Soap

The determination of the foaming power of soap is made by measuring the foam content formed for the complete dissolution of a test sample of soap (0.5g) in a volume of distilled water (50ml) compared to control according to the following formula:

$$Tm(\%) = \frac{Sample\ foam\ height\ (cm)}{Foam\ height\ control\ (cm)} \times 100$$

2.4.4. Determination of the Melting Point

The melting point is the temperature at which hard soap turns into a paste. It is determined by placing a test sample of the soap on the hot plate and recording the melting temperature.

2.4.5. Evaluation of the Antiseptic Efficacy of the Soap Produced

The experimental protocol used to evaluate the effectiveness of the soap produced in eliminating bacteria is as follows:

- The preparation of PCA (Nutrient Agar) Culture Medium, a non-selective, nutrient-rich medium, is recommended for the standardized enumeration of aerobic bacteria in fluids.
- Test for bacteria before washing your hands by placing each hand on the surface of your culture medium for one minute.
- Place thoroughly washed and disinfected hands on the culture medium for one minute.
- Incubate the agar plates containing the samples for 24 h at 30°C.
- Count the bacterial colonies presented in each dish.

Fig.7: Bacteriological sampling before hand washing.

2.5. The Effect of the Fat

As part of studying the type effect and the amount of fatty acid used, we made 4 types of soaps with different portions of the 4 oils. The following table shows the tests performed.

Table 2: The percentages of oils used to manufacture handmade soaps.

Oils used	**Soap 1**	**Soap 2**	**Soap 3**	**Soap 4**
Oils recovered from the OMWW	50%	50%	40%	40%
Olive oil	50%	40%	40%	30%
Cocoa oil	-	10%	20%	20%
Castor oil	-	-	-	10%

III. Results

3.1. Characterization of Oil Recovered from the OMWW

The OMWW used in this study was taken from a traditional crushing unit in Imzouren, characterized by their reddish-brown to black coloration. The recovery of the oily residues is done by the natural settling technique of the OMWW of our sample with a yield of 12.3%, so the crude OMWW gives us 1.23 l% of oil.

The following table shows that the recovered oil has a very high acidity equal to 2.73%, which exceeds the value of the acidity of edible olive oil published by the international food standard (0.8%) [13]. This makes it unfit for consumption and can be used to manufacture soaps according to the internal standard of the COGB La Belle unit 5 to 10% [14]. The saponification index value obtained is 186.2 mg KOH/g, which explains the richness of the OMWW recovered in fatty acids. This value is in the range mentioned by the Codex Alimentarius 182-196 mg KOH/g.

Table 3: Characterization of recovered oily residues.

Parameter	**Unit**	**Value**
ph	-	4.9
Acidity	%	2.56
Peroxide Value	meq O2/kg	9.42
Saponification index	mg KOH /g	186.2
Iodine value	g/100g	81.12

3.2. Preparation of pasty soaps based on recovered oils

3.2.1. Cold Saponification

This technique does not consume a lot of time and energy, it is highly recommended thanks to its simplicity as well as the cold produced soap has a beneficial effect on the skin since it contains glycerin and the vegetable oils used have retained their properties since they are not subjected to any temperature**.**

Fig.8: Photograph of cold soap after drying.

3.2.2. Hot Saponification

This technique minimizes soap manufacturing time by accelerating the saponification reaction by temperature and ethanol, promoting contact between soda and fatty acid. The soap produced differs from that produced in the cold process since the glycerin is extracted during the leaching step after saponification, which consists of gradually adding a saturated salt solution. This type of soap is intended for use in laundry and toiletry since it is of high quality.

Fig.9: Photograph of cold soap after drying.

3.3 . Characterization of the Soaps Produced

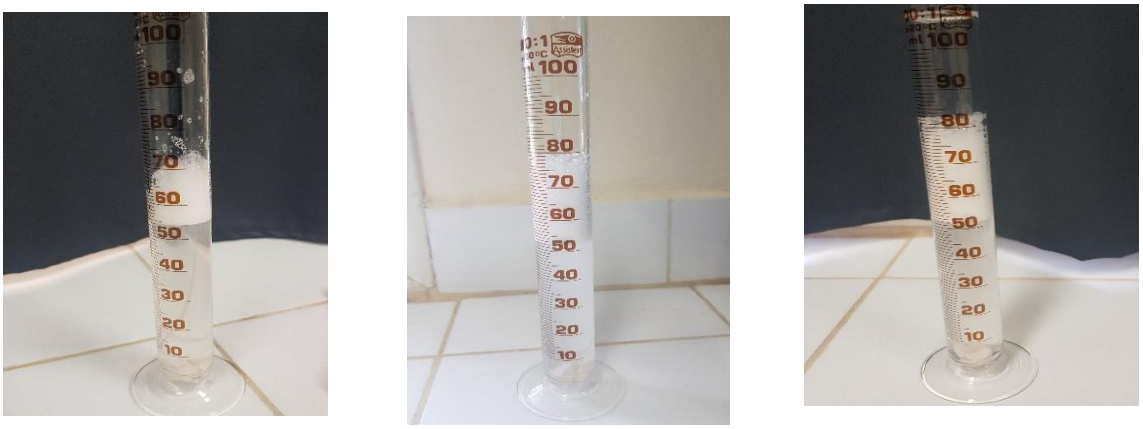
The resulting soaps have a beige color with a clean appearance **(**Figures 9 and 10**),** and the following table shows the results of the analyses carried out.

Table 4: Summary table of the analyses carried out on the soaps produced.

Soap	Hydrogenated Potential ph	Humidity (%)	Foaming power (%)	Melting point °C	Peroxide value (O2 meq/kg)
Cold Process Soap	7.8	11.5	18	150	4
Hot Soap	9.3	13	14	200	5.6

The most important parameter to check is the pH. The measured pH is 9.3 for reflux soap and 7.8 for cold process soap. These values are within the pH values set there between 7 and 9. The foaming power is around 18 for cold process soap and 14 for hot soap. Regarding the melting point for cold process soap, it is between 150°c and 200°c for reflux soap. These values are consistent with those found by several researchers [14].

According to ISO standard 672 [13], the humidity threshold for solid soaps is between 11% and 13%. The results obtained show humidity values of around 12.7% for cold process soap and 11.5% for hot soap. Both of these values are within the threshold.



a) Cold Process Soap b) Hot soap c) Sample soap

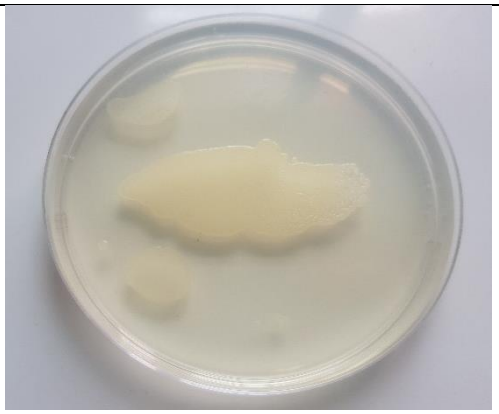
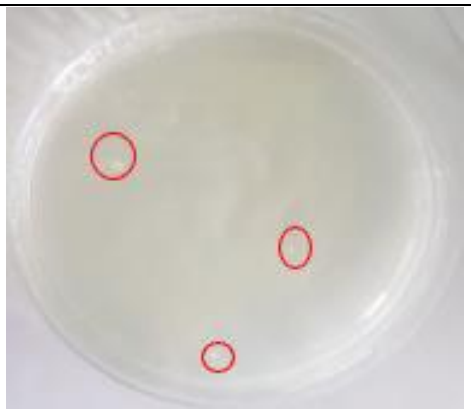
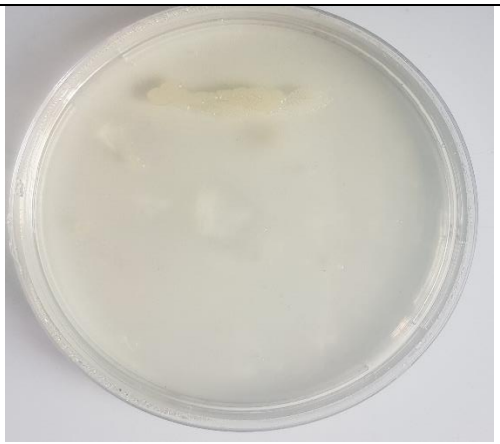
Fig.10: Foaming power of the soaps obtained.

3.4. Evaluation of the antiseptic efficacy of the soap produced.

The concept of this microbiological test is based on naming bacteria in a culture medium (CAP) before and after hand washing by soaps produced to test the bactericidal efficacy of these soaps. Before one week of the experiments, the volunteer must not wash his hands with antiseptic or disinfectant products. Note that during this test, we did not add any antiseptic compounds.

The results obtained from this test give us a first idea of the effectiveness of the soaps we produce. Figures 11, 12, 13, and 14 present the results obtained after incubation in each Petri Dish.

Table 5: The antibacterial efficacy of the soaps produced.

Type of soaps	Before Hand Washing	After Hand Washing
Cold Process Soap	 **Fig.11:** Bacteriology test before hand washing (cold soap).	 **Fig.12:** Bacteriology test after hand washing (cold soap).
Hot Soap	 **Fig 13:** Bacteriology test before hand washing (reflux soap).	 **Fig.14:** Bacteriology test after hand washing (reflux soap).

The formation of bacterial colonies and molds was observed after incubation in the four Petri dishes. Still, there was a difference in the number of colonies and molds formed before and after washing for the two types of soaps. The figures clearly show that hand washing with both types of soap reduces the number of bacterial colonies.

The results of the bacteriological samples obtained are very satisfactory. The soaps produced have a high bacterial reduction capacity, consistent with the published literature on the antibacterial efficacy of olive oil [15].

3.5. Saponification Yield

To determine the effectiveness of these techniques, we used the calculation of production yield based on the saponification reaction:

$$Olein + soda \rightarrow Soap + Glycerin$$

The yield relationship is calculated by the ratio of the experimental mass of soap produced to the theoretical mass calculated.

With:

- $n_{olein} = \frac{n_{soap}}{3}$
- $M_e = 884\ g/mol$
- $M_{soap} = 304\ g/l$

The production yield is very satisfactory for both techniques, reaching 94% for cold saponification and 89.9% for hot saponification.

Table 6: Saponification yield.

Technique	m_{exp} (g)	M_{Theo} (G)	Yield (%)
Cold saponification	176.5	187.7	94.38
Hot saponification	211.2	235.2	89.97

3.6. The effect of the fat used on the soap produced.

3.6.1. Cold Saponification

In order to evaluate the effect of the amount of fat used on the soap produced, we added other oils (castor oil, cocoa oil) to the basic formula (oil recovered from the OMWW, olive oil), which is represented as soap N°: 1. We adopt the percentages mentioned in Table 2, the soaps obtained are illustrated in the following photos.

Soap No.: 1

Soap No.: 2

Soap No.: 3

Soap No.: 4

Fig.15: The four types of handmade soaps produced.

3.7. Evaluation of the quality of the soaps produced.

The assessment of soap quality was determined using different standards or indicators. These are presented in the following table:

Table 7: The range of value of soap-making criteria.

Indicators	Value Range
Hardness	29-54
Cleaning power	12-22
Sweetness	44-69
Foaming power	14-56
Creaminess index	16-48
Iodine value	40-70 Ideal value: 50
INS	036-170 Ideal value: 160

3.7. 1. pH

The pH values for each soap-making test are in the range of 8.0 and 9.5. After adding 1 g of citric acid, we obtained pH values between 7 and 7.5.

3.7.2. Hardness

The hardness values obtained for each soap are shown in Figure 16. After adding 20% cocoa oil and 10% castor oil, we obtain a hardness of 34 and 37, respectively, in the recommended range. Regarding soaps 1 and 2, we got values below 29, which are soft and crack quickly.

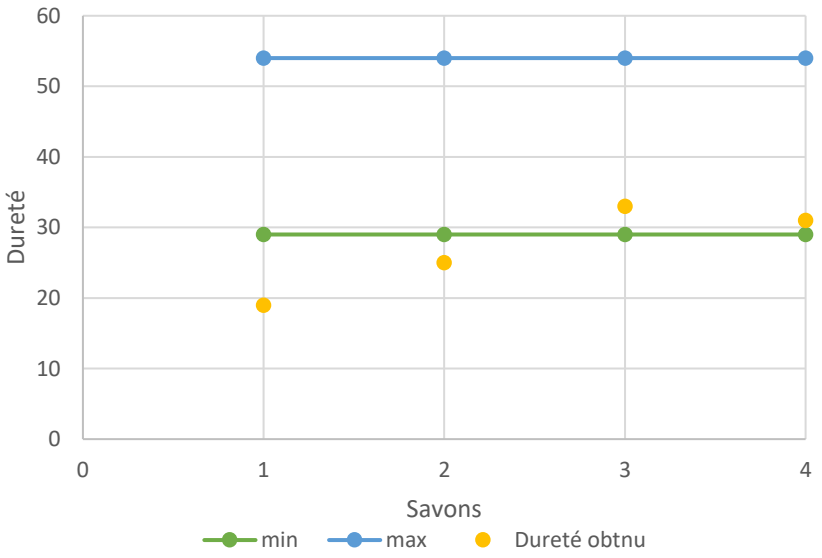


Fig. 16: The variation of hardness according to the type of soap.

3.7.3. Cleaning Power

Cleaning power is the most important criterion in the manufacture of soaps. Figure 17 shows that soaps 3 and 4 are the most recommended for use since their cleaning power values are within the standards.

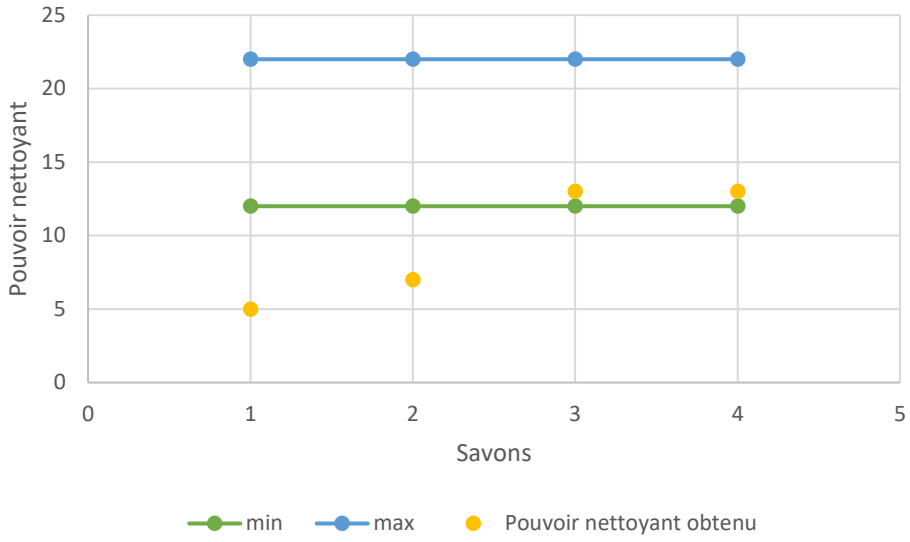


Fig.17: The variation of the cleaning power according to the type of soap.

3.7.4. The Hydration Index

The following figure illustrates the values of the mildness index or the hydration index. Concerning the first two soaps, the index has exceeded the maximum limit, and this can be explained by the presence of monounsaturated acids (oleic) and the successive addition of cocoa oil, which decrease these high levels until the standard values are obtained, 66 for soap 3 and 63 for soap 4.

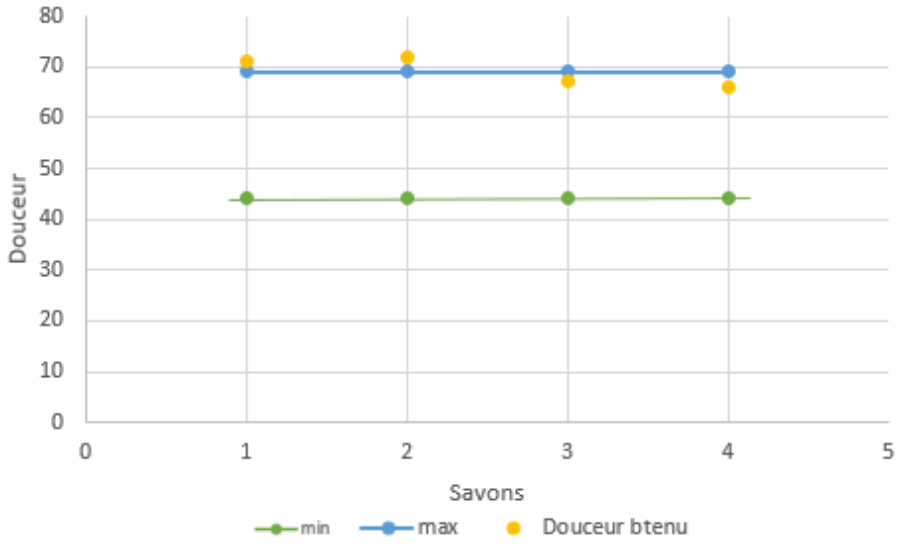


Fig.18: The variation of the mildness index according to the type of soap.

3.7.5. The Foaming Power Index

The foaming power index values are shown in Figure 19, which measures the soap's ability to lather and produce bubbles. From these results, we notice that all the other formulas produced from foams, except for the basic soap (soap 1), are due to the presence of cocoa oil, which is responsible for the production of the bubbles.

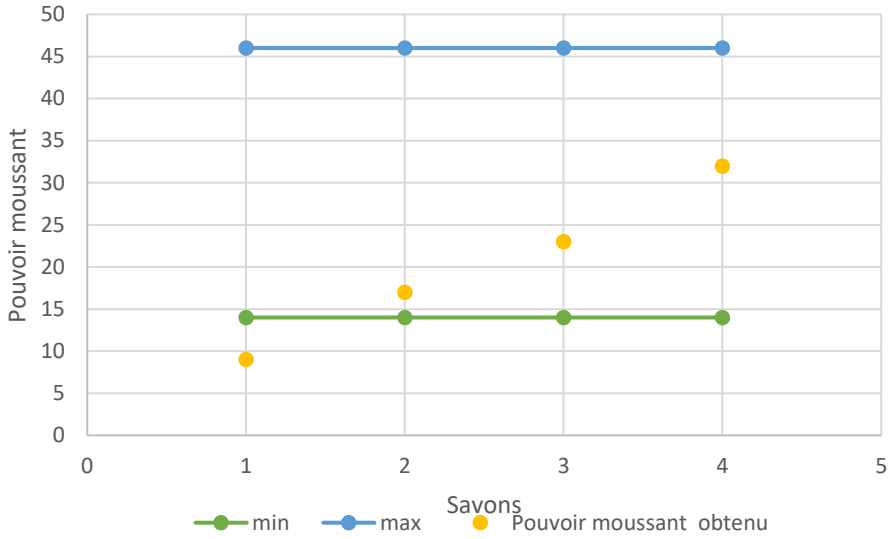


Fig.19: The variation of foaming power according to the type of soap.

3.7.6. The Creaminess Index

The creaminess index (the strength of the soap) is shown in Figure 20. We notice that all the values are around 16 except for soap N° 3; the index value reaches 27, so the foam is not very creamy but remains within the standards. To increase these values, add a quantity of cocoa or castor oil.

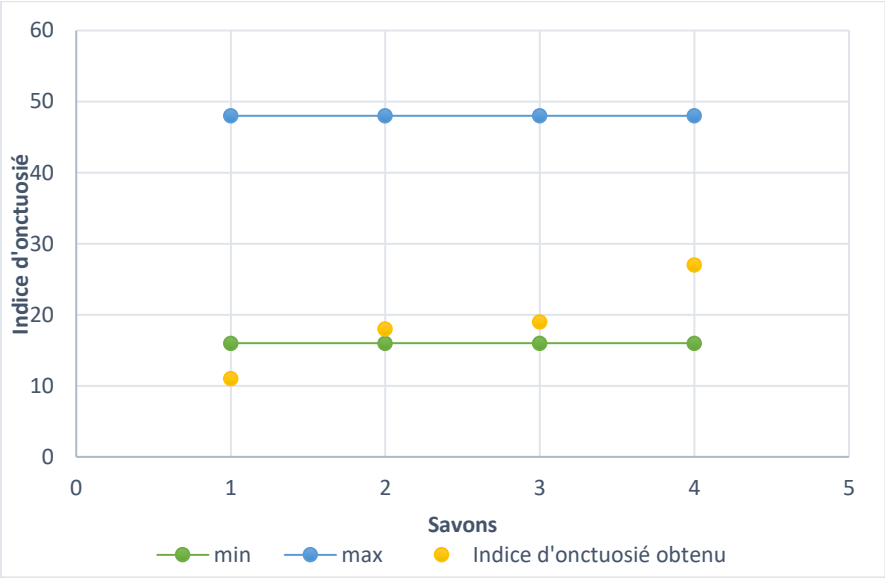


Fig.20: The variation in the creaminess index according to the type of soap.

3.7.7. Iodine Value

Regarding the fragility of the soap manufactured, we study the iodine value illustrated in the following figure. We observe that the iodine value of the first soap is close to the limit value of 70, so it is riskier to be oxidized and blackened, and its shelf life is shorter than that of other soaps with values below the standards.

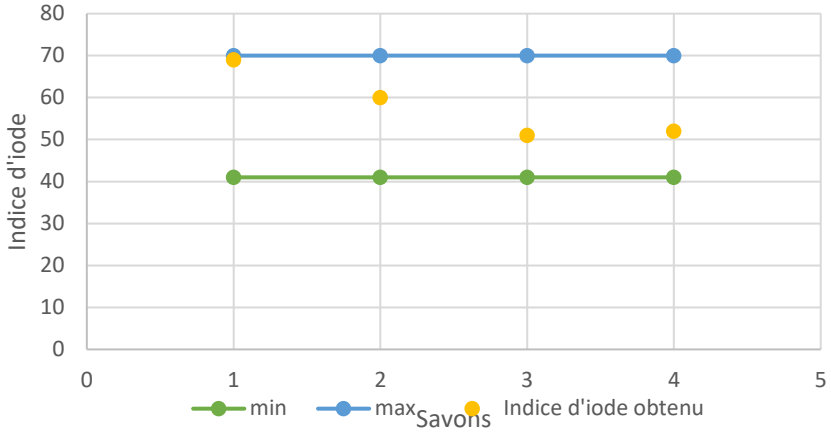


Fig.21 : The variation in iodine value according to the type of soap.

3.7.8. Iodine Number Saponification

The final endpoint of the study is the equilibrium parameter, Iodine Number Saponification (INS), which provides an overall rating for the soap and uses the parameters we've already mentioned: hardness, detergent, hydration, and lather. According to the results mentioned in Figure 22, we notice that the best soap among the four soaps is soap 4, since its shelf life is the longest, as its INS is the highest (INS N° 4 = 152).

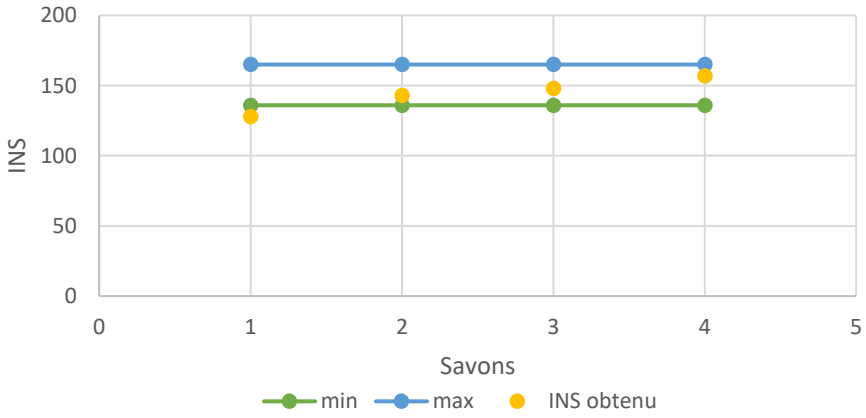


Fig.22: The variation of INS according to the type of soap.

IV. Conclusion

The liquid discharges formed during the olive oil extraction process contain a remarkable amount of oily residues. The latter is characterized by a very high acidity equal to 2.73%, which exceeds the value of the acidity of consumable olive oil with a saponification index of around 186.2 mg KOH/g, which makes them a perfect source (fat) for saponification. The valorization of the OMWW in soap manufacturing has yielded the following results:

- Characterizing cold-produced and hot-produced soaps leads to the recommendation of using cold-process soaps.
- The production efficiency is 94% for cold saponification and 89.9% for hot saponification.
- The quantities of fatty acids used influence the effectiveness of the soaps; the appropriate formula found is as follows: 40% oils recovered from the OMWW, 30% olive oils, 20% cocoa oil, and 10% castor oil.

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