



MANUFACTURING SOAP FROM WASTE COOKING OIL USING COLD AND HOT PROCESSES

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ABSTRACT

The conversion of waste cooking oil to soaps using endothermic (hot) and exothermic (cold) soap making processes was investigated. Waste cooking oils were collected locally and characterized using standard methods and found to have acceptable saponification value (258.9 g). Various soaps were prepared by factorial combinations (A, B, C) using the following parameters: amount of NaOH, oil content and molding time. The soaps prepared gave a pH range of 9.85 ± 0.45 to 11.20 ± 0.30 and 10.65 ± 0.65 to 12.20 ± 0.10 for the cold and hot processes respectively. The % moisture contents of the soap made using cold process were between the ranges of $25.20 \pm 3.60\%$ to $38.36 \pm 2.84\%$ while the soaps made from hot process had a % moisture content of $21.90 \pm 3.50\%$ to $35.74 \pm 3.54\%$. Total fatty matter (TFM) content of the cold process soaps gave values of 10.60% - 60.00% while the hot process soaps reported values ranging from 30.00% - 71.00%. Relatively similar foam heights were observed for soaps made by both processes with values of 0.70 - 2.50cm and 0.60 - 2.50cm for both cold and hot processes respectively. From the results obtained for the effectiveness in cleaning of the soaps, cold process soaps had the highest number of soaps being more effective and most effective, whereas hot process soap making method had majority of soaps being just effective. The treatment A2B1C2 (Endothermic method + 10% NaOH + 4 days molding time), with molding during of 4 days, was considered the best treatments based on its TFM value of 71.00% and effectiveness in cleaning being most effective. In general, the soaps obtained from both cold and hot process, had TFM, % moisture and pH values comparable to the commercially available soaps (Eva, Joy and Premier). The use of waste cooking oils from this study was found to be a good alternative feedstock for the manufacture of soap and as mitigation for environmental pollution.

Keywords: Soap, oil, saponification, pH, moisture content

INTRODUCTION

Due to the abundance of waste material, there is a need to produce soap from these wastes. Waste cooking oil (WCO) is in liquid form which is not properly disposed either in homes, restaurants and catering outlets. This waste disposal issue has resulted in blockage of sewage drains and aquatic life destruction. From this waste cooking oil, soap can also be produced that will serve the dual purpose of taking care of the waste management problem as well as employment creation for the local village folk.

Soap is made from an acid (animal fat in our case) and base (sodium hydroxide solution) reaction that produces salt (soap), water and glycerin. The process is called saponification reaction, also known as the cold process for making soap, is the alkaline hydrolysis of triacylglycerols. These esters are the major constituents of vegetable oils and animal fats; they can react with a strong mineral base like sodium hydroxide, in aqueous medium, to produce the sodium salts of the hydrolysed free fatty acids (the opaque soap) and glycerol (Panda, 2003; Hill, 2004; Mabrouk, 2005).

The preparation of these soaps using the cold process however, involves combining natural oils (coconut, palm, olive or butter), hydroxide and water (Ruiz, 2008; Helmenstine, 2017). The texture have been known to be very smooth or fine, typically, although only 95% of the oils have been known to be converted into soap; the other 5% of the oils remain in the soap which is thereafter added to the excellent conditioning of soap qualities (Warra et al., 2011). This study was therefore aimed at manufacturing soap from waste cooking oil collected from fast-food restaurants using cold and hot processes.

MATERIALS AND METHODS

Waste cooking oil samples were collected from different fast-food restaurants within Benin City, Edo State. The samples were stored at room temperature until use.

The following pre-treatment processes were carried out on the oil. The waste cooking oil (WCO) samples were warmed slightly to a temperature of about 40°C and filtered to remove debris. Saponification value was determined according to standard methods (AOAC, 2000). 2.00g of oil sample was added into a 250ml conical flask, 25 ml of 0.5M potassium hydroxide was then added. The flask was gently heated for 30 minutes with occasional shaking. The solution was allowed to cool and then titrated against 0.5 M HCl. The endpoint was recorded when a colour change from pink to light yellow was observed. A blank was also prepared without the oil sample. The colour change of the blank was from pink to colourless.

The saponification value was calculated using the formula:

$$SpV = \frac{56.1(R_{AB} - R_{AS}) \times N}{W_o} \dots\dots\dots (1)$$

Where: SpV= Saponification Value, RAB=Required acid "HCl" by blank (ml), RAS= Required acid"HCl""by sample (ml), N=Normality of the standard HCl, Wo = weight of the oil (g).

Soap Preparation

The saponification process was carried out using the method as reported by Mak-Mensah and Firempong (2011). The soap was prepared according to the components formulated from the block design with factorial arrangement AxBxC, where, A=Methods (A1: Exothermic and A2: Endothermic), B= Percentage of NaOH (B1: 10 %, B2: 15 %, B3: 20 % and B4: 30 %), C= Molding time (C1: 2 days and C2: 4 days).

Cold Process soap preparation:

The oil and lye solution were mixed together in a ratio of 50 % oil and 50 % of the lye solution to produce each soap sample. The saponification reaction was initiated by adding NaOH according to the concentration of each treatment (i.e. 10 % NaOH, 15 % NaOH, 20 % NaOH and 30 % NaOH) and stirred properly till it turned semi-solid. Then it was transferred to the mold and allowed to set for the respective number of days. The soaps were all prepared at room temperature. After demolding, the prepared soaps were all rinsed with distilled water to remove any excess sodium hydroxide on the surface and impurities. They were then air dried for 3 weeks before being analysis.

Hot Process soap preparation:

The oil and lye solution were mixed together in a ratio of 50 % oil and 50 % of the lye solution to produce each soap sample. The saponification reaction was initiated by adding NaOH according to the concentration of each treatment (i.e. 10 % NaOH, 15 % NaOH 20 % NaOH and 30 % NaOH), the beaker was then placed in a water bath at 68°C-75°C. It was stirred continuously till it turned to a gel having a glossy appearance before it was then transferred to the mold.

Determination of total fatty matter (TFM)

The total fatty matter content was determined using the method reported by Roila *et al.*, (2001) with slight modifications. Ten grams of the prepared soap sample was weighed and 150 ml distilled water was added and heated. The soap was then dissolved in 20 ml of 15 % H₂SO₄ with heating until a clear solution was obtained. The solution was allowed and set.

Total fatty matter using a formula:

$$\% TFM = \frac{(A-X)}{W} \times 100$$

where A= weight of crucible+ oil after drying (g)

X= weight of crucible and (g)

W= weight of soap (g).

Determination of Percentage (%) moisture Content

Approximately 5 g of samples was accurately weighed using analytical balance into dried, tarred moisture dish and dried in an oven (DHG-9023A) for 2 hrs at 101 ± 1 oC and repeated until a constant weight (difference between two measurement not exceed 0.5 mg/g of sample) was reached. % moisture was calculated using the following formula, (AOCS 1997).

$$\% \text{ moisture} = \frac{(C_s - C_h)}{(C_s - C_w)} \times 100$$

Where: C_w = weight of crucible (g)
 C_s = weight of crucible + sample (g)
 C_h = weight of crucible + sample after heating (g)

Determination of pH

Ten grams of the crushed soap was weighed and dissolved in distilled water in a 100ml volumetric flask. It was made up to the 100ml mark with the distilled water. The pH of the soap solution was determined using a pH meter (Hanna Instrument PH-009 IA).

Determining foaming height

Foaming height was determined using a method as reported by Isah (2006) with modification. About

5.0g each of soap shavings was added to a 500ml measuring cylinder containing 100ml of distilled water. The mixture was shaken vigorously so as to generate foams. After shaking for about 2 minutes, the cylinder was allowed to stand for about 10 minutes. The height of the foam in the solution was measured and recorded. This was repeated for all soap combinations.

Test for effectiveness in cleaning

To determine the cleaning property of the prepared soaps, a drop of oil was placed on four separate strips of filter paper. The filter papers with the oil spot were immersed in a separate test tubes containing soap solution (2g soap shavings/100ml distilled water) each was shaken vigorously for 1 minute. The filter papers were removed and rinsed with distilled water and the degree of cleanliness in each filter paper was observed.

RESULTS

Table 1 below represents the properties (moisture content, pH, total fatty matter and foam height) obtained for the different soap combinations prepared and compared with commercially available brands.

TABLE 1: Percentage (%) Moisture content of the soaps produced

Sample	Moisture content (%)	pH	TFM Values (%)	Foam Height (cm)
A ₁ B ₁ C ₁	25.20 ± 3.60	9.85 ± 0.45	40.41 ± 0.01	1.52 ± 0.02
A ₁ B ₁ C ₂	26.40 ± 7.60	10.40 ± 0.10	10.61 ± 0.01	2.02 ± 0.02
A ₁ B ₂ C ₁	30.10 ± 6.10	10.15 ± 0.35	60.02 ± 0.02	2.49 ± 0.01
A ₁ B ₂ C ₂	37.35 ± 2.75	10.60 ± 0.00	56.81 ± 0.01	2.48 ± 0.01
A ₁ B ₃ C ₁	36.31 ± 6.10	10.65 ± 0.45	32.45 ± 0.35	0.71 ± 0.01
A ₁ B ₃ C ₂	38.36 ± 2.84	10.80 ± 0.20	54.10 ± 0.10	1.69 ± 0.01
A ₁ B ₄ C ₁	37.10 ± 3.10	11.20 ± 0.30	29.05 ± 0.05	1.02 ± 0.02
A ₁ B ₄ C ₂	35.80 ± 3.80	11.15 ± 0.25	36.82 ± 0.02	0.92 ± 0.02
A ₂ B ₁ C ₁	21.90 ± 3.50	10.65 ± 0.65	36.72 ± 0.02	2.02 ± 0.02
A ₂ B ₁ C ₂	22.90 ± 4.10	10.70 ± 0.60	71.10 ± 0.10	2.52 ± 0.02
A ₂ B ₂ C ₁	25.57 ± 4.17	10.80 ± 0.80	55.65 ± 0.07	2.51 ± 0.01
A ₂ B ₂ C ₂	26.69 ± 1.69	10.95 ± 1.05	62.15 ± 0.15	2.01 ± 0.01
A ₂ B ₃ C ₁	33.20 ± 0.80	11.30 ± 0.50	49.82 ± 0.02	1.99 ± 0.01
A ₂ B ₃ C ₂	33.13 ± 8.33	12.20 ± 0.10	43.42 ± 0.02	1.31 ± 0.01
A ₂ B ₄ C ₁	31.59 ± 0.22	11.99 ± 0.02	41.12 ± 0.12	0.73 ± 0.03
A ₂ B ₄ C ₂	35.74 ± 3.54	11.89 ± 1.09	30.10 ± 0.10	0.62 ± 0.02
Premier soap	17.70 ± 3.70	10.20 ± 0.31	41.15 ± 0.15	18.02 ± 0.02
Joy soap	16.40 ± 3.00	10.63 ± 0.08	87.25 ± 0.05	13.91 ± 0.01
Eva soap	8.40 ± 3.20	10.43 ± 0.18	86.67 ± 0.07	16.71 ± 0.01

Mass of soap used = 5.0 g

Values obtained revealed a moisture content range of 21.90-38.36%. The combination of A1B3C2 gave the highest value of 38.36% ± 2.84 while sample combination A₂B₁C₁ gave the least moisture content 21.90 % ± 3.50. Most of the values obtained were comparable with the three commercially available soap with a range of 8.40%, 16.40% and 17.70% obtained for Premier soap, Joy soap and Eva soap respectively.

A pH range of 9.85 – 12.20 was obtained for the different soaps prepared (Table 1). Soaps produced using the cold process method was observed to have pH values ranging from 9.85 -11.20 while the soaps produced from hot process method have pH values ranging from 10.65 – 12.20. All the samples exhibited

basic pH with $A_2B_3C_2$ giving the highest pH 12.20 ± 0.10 and $A_1B_1C_1$ giving the lowest pH 9.85 ± 0.45 . The commercially available soaps were found to have a pH values of 10.20 - 10.63.

The foam height was found to be in the range of 0.60cm-2.5cm. The highest foam height of 2.5 cm was observed for a number of samples - $A_1B_2C_1$, $A_1B_2C_2$, $A_2B_1C_2$ and $A_2B_2C_1$ while the lowest foam height of 0.6 cm was observed for sample $A_2B_4C_2$. The commercially available soap gave a higher range of foam height with a range of 13.90 - 18.00 cm obtained.

The total fatty matter for the combinations was found to be in the range of 10.60% - 71.00 %. The highest total fatty matter of 71.00 % was obtained in the combination - $A_2B_1C_2$ while lowest total fatty matter of 10.60% was observed in the combination -10.60 %.The commercially available soap gave a total fatty matter value ranging from 41.00 % - 87.20 %. The cleaning efficiencies of the soaps were also assessed and found to have a higher cleaning efficiency when compared with the commercially available soap. Combinations $A_1B_1C_2$, $A_1B_3C_1$, $A_2B_1C_1$, $A_2B_1C_2$ and $A_2B_3C_1$ exhibited the best cleaning property even higher than the commercially available soaps (Table 2)

Table 2: Cleaning effectiveness of the soap samples prepared

Samples	Cleaning effectiveness
$A_1B_1C_1$	Effective
$A_1B_1C_2$	Most effective
$A_1B_2C_1$	More effective
$A_1B_2C_2$	Effective
$A_1B_3C_1$	Most effective
$A_1B_3C_2$	Most effective
$A_1B_4C_1$	More effective
$A_1B_4C_2$	More effective
$A_2B_1C_1$	Most effective
$A_2B_1C_2$	Most effective
$A_2B_2C_1$	Effective
$A_2B_2C_2$	Effective
$A_2B_3C_1$	Most effective
$A_2B_3C_2$	Effective
$A_2B_4C_1$	Effective
$A_2B_4C_2$	More effective
Premier soap	Most effective
Joy soap	More effective
Eva soap	More effective

This study was designed to explore an alternative use of waste oils, instead of indiscriminate disposal in drainage systems, soil, and the environment to cause environmental pollution.

The saponification value of the oil 258.9 was similar to the saponification value of coconut oil 257.0 (Abayeh *et al.*, 1998). The value is higher than that of olive oil (192.0) and sunflower oil (188.7). This indicates that the oil could be used in soap making since its saponification value fall within the range of these oils. It could also substantially be

used in the preparation of cosmetic products (Warra, 2009). Oils with high saponification values such as coconut oil (257.0) and palm oil (199.1) are better used in soap making (Abayeh *et al.*, 1998). Some soap manufacturers blend their oils with coconut oil (with high saponification value) to get yield of better quality soaps.

Moisture content is a parameter that is used in assessing the shelf life of a product. High moisture content in soap would lead to reaction of excess water with un-saponified fat to give free fatty acid and glycerol in a process called hydrolysis of soap on storage. For % moisture content, the combination $A_1B_3C_2$ has the highest percentage moisture content $38.36 \% \pm 2.84$, followed by the combination $A_1B_2C_2$ $37.35 \% \pm 2.75$ and $A_1B_4C_1$ $37.10 \% \pm 3.10$, in these treatments no significant statistical difference. The combinations $A_2B_1C_1$ have the lowest %moisture content $21.90 \% \pm 3.50$ as compared to the rest combinations. The controls have low % moisture, Eva 08.40 %, Joy 16.40 % and premier 17.70 %. The % moisture content obtained after analyzing were generally higher than the controls. The moisture content was similar to the recommended by Salguero *et al.*, (2017).The results obtained from this analysis were found to be similar to those obtained by Sanaguano *et al.*, (2017),with their highest moisture content being 43.24 % in the combination $A_1B_1C_1$ and the least being 25.73 %, combination $A_2B_2C_2$. This may be due to difference in the soap preparing methods and difference in the ratio of lye solution to oil in the mixture. This could be as a result of excess lye due to high concentrations. The pH of the soaps was rather on the high side as compared to the control. The pH of the commercially available soap is 10.43, 10.63 and 10.20 for Eva, Joy and Premier respectively. The combination with the highest pH value is $A_2B_3C_2$ with pH value of 12.20 ± 0.10 , followed by $A_2B_4C_1$ with pH 11.99 ± 0.02 and $A_2B_4C_2$ with pH value 11.89 ± 1.09 while the combination $A_1B_1C_1$ and $A_1B_2C_1$ have the lowest pH values of 9.85 ± 0.45 and 10.15 ± 0.35 respectively, in these treatments, there's no significant statistical difference. The pH of NaOH is about 14.00. For bathing soaps, the pH value should be around 07.50-09.00. Cold process soap is naturally alkaline with a pH of around 09.00-10.00. The results obtained were slightly higher than those obtained by Sanaguano *et al.*, (2018) and Mak-Mensah *et al.*, (2011) in their work on neem oil soap. The high pH values of the soap owe to high percentage of lye solution used in making the soap, also due to incomplete hydrolysis resulting from the saponification process. The remedy to high pH value is super fatting (i.e. adding excess fat). More oil or fat can be added to the mixture to reduce the pH, thereby reducing the harshness of the soap. High pH means the soap will be harsh on the skin and different skins have different pH.

The foaming abilities of the different soap samples were also analysed with respect to the foam

height the produce. The controls gave significant foam heights as compared to the soaps made. The additives added could be responsible for this, but no additive was added to the prepared soap samples. Premier soap gave the highest foam height of 18.00cm, followed by Eva 16.70cm and joy soap 13.90cm. The combination with the highest foam height is $A_1B_2C_1$, $A_1B_2C_2$, $A_2B_2C_1$ and $A_2B_1C_2$, with 2.50cm foam height. The combinations $A_1B_1C_2$, $A_2B_1C_1$, $A_2B_3C_1$ and $A_2B_2C_2$ have a foam height of 2.00cm. The combinations with the lowest foam heights are $A_1B_3C_1$, $A_2B_4C_1$ and $A_2B_4C_2$, with foam heights of 0.70cm, 0.70cm and 0.60cm respectively.

The % total fatty matter values gotten from analyzing the soap were comparable to the controls, Premier 41.00 %, Joy 87.20 % and Eva 86.60 %. The combination with the highest TFM value is $A_2B_1C_2$ 71 %, followed by the combination $A_2B_2C_2$ and $A_1B_2C_1$, with TFM value of 62.00 % and 60.00 % respectively, this is not statistically different. The combination $A_1B_1C_2$ and $A_1B_4C_1$ have the lowest TFM value of 10.60 % and 29.00 % respectively. The lower TFM value is due to the presence of unreacted NaOH in

the mixture. Additives added like EDTA, glycerin, can also increase the value of TFM as the react with the excess NaOH to some extent. High TFM can be achieved by putting in high quantities of C8 Oleic acid.

The soaps made can clean generally but some cleaned better than the others within the given contact time. The combinations $A_1B_1C_2$, $A_1B_3C_1$, $A_1B_3C_2$, $A_2B_1C_1$, $A_2B_1C_2$, $A_2B_3C_1$, were most effective using the control as a means of comparison.

CONCLUSION

From the study, the preparation of soap from waste cooking oil was carried out. Analysis of the chemical characteristics revealed values comparable to commercially available soap; the treatment $A_2B_1C_2$ gave the best treatment in % total fatty matter, having a value of 71 % and amongst the best in effectiveness in cleaning. This work show revealed that the waste cooking oil could be used to make soaps with favourable characteristics to industries for domestic purposes, as an alternative use of this waste to partially mitigating the negative effects on environment.

REFERENCES

- A.O.A.C, (2000). Official Method 920.160-Saponification Number of Oils and Fats/IUPAC 2.202 I.S.I Hand Book of Food Analysis (Part XIII 1984), A.O.A.C, 17th edition.
- Abayeh, O. J., Aina, E. A. and Okuonghae, C. O. (1998). Oil content and oil quality characteristics of some Nigerian oil seeds. *Journal of Pure and Applied sciences* 1(1): 17 – 23.
- Food and Nutrition Board, Institute of Medicine of the National Academies (2005). Dietary Reference Intakes for Energy, Carbohydrate, Fiber, Fat, Fatty Acids, Cholesterol, Protein, and Amino Acids (Macronutrients). *National Academies Press*. p. 423 and p. 504.
- Helmenstine, A. M. (2017). Endothermic and Exothermic Reactions. ThoughtCo, Feb. 8, 2017, thoughtco.com/definition-of-exothermic-reaction-604462
- Hill, M. (2004). Product and process design for structured products. *AIChE J.* 50 (8), 1656 –1661.
- Isah, A. G. (2006). Production of Detergent from Castor oil. *Leonardo Journal of Practices and Technologies*, 9: 153-160.
- Mabrouk, S. T. (2005). Making usable, quality opaque or transparent soap. *J. Chem. Educ.* 82 (10), 1534 – 1537.
- Mak-Mensah, E. E. and Firempong, C. K. (2011). Chemical characteristics of toilet soap prepared from neem (*Azadirachta indica A. Juss*) seed oil. *Asian Journal of Plant Science and Research* 1 (4): 1-7.
- Panda, H. (2003). Herbal Soaps and Detergents Hand Book. National Institute of Industrial Research, Delhi, India.
- Ruiz, J. (2008). Fabrication de jabron Casero, un ejemplo de reaccion quimica de saponification. *Rev. Digital Ennov. Exp. Edu.*, 45 (6): 8
- Salguero, H.S., León, A. T., Quintana, A. L. and Morejón, F. B. (2017). Recovery of Waste Cooking Oil Used in the Biodiesel Production in Ecuador. *International Journal of Scientific and Engineering Research* 8 (5): 123 – 127.
- Sanaguano, H., Tigre, R. A., Bayas Morejón, I. F. and Ruilova, M. (2017) Nutritional value and sensory properties of cookies prepared from flour mixes of carrot (*Daucus carota*), lupine (*Lupinus perennis*) and barley (*Hordeum vulgare*). *European Scientific Journal*, 13(9): 378-385.
- Standard Test Method for fat and oil (D5558). (2012). Saponification value of fat and oil. TIB99307. 1: 1 - 4.
- Warra, A. A. (2009). Extraction and Chemical Analysis of Indigenous Shea nut oil. *Proceedings of the 1st National Conference of the Academic Staff Union of Polytechnics. WUFP Chapter Nigeria*. pp 160-162.
- Warra, A. A., Wawata, I. G., Guru, S. Y. and Atiku, F. A. (2011). Soap preparation from Soxhlet extracted Nigerian cotton seed oil. *Advances in Applied Science Research*. 2(5): 617 –623.