

Physico-Chemical Analysis and Potentials of Onion Seed Oil for Soap Production

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Abstract- This study was carried out to determine the physico-chemical properties and potentials of onion seed oil for soap production. Onion seed oil was extracted using Soxhlet extraction. The result of the chemical analysis gave the percentage of (30.20%) with the density of 0.86g/cm³. The color of the oil was light green and chemical analysis of the oil reveals saponification value for onion oil is 211.54±0.05mg KOH/g. The iodine value for onion seed is 98.13±0.39g I₂/100g. Acid value is 5.13±0.04mgKOH/g. The peroxide value is 2.75±0.25. Refractive index is 1.46±0.01 and free fatty acid value of this oil is 6.93±0.54. A simple cold-process alkali hydrolysis of the onion oil was used to produce a milky colored soap with a foam height of 60cm³ for onion soap. The chemical properties of the soap produced from the oil were total fatty matter (36.33%), total alkali (0.78%), percentage chlorine (0.48%) and pH (10.70) respectively. From the physico-chemical analysis of the oil and the soap produced, it can be concluded that oil from onion seeds has the potential in the production of soap, perfumery and pharmaceuticals.

Indexed Terms- Extraction, oil, iodine value, saponification value

I. INTRODUCTION

Soaps are anionic (negatively charged) surfactants produced from hydrolysis of fats in a chemical reaction called saponification. But the term soap usually applied to water soluble salt (Kuntom *et al.*, 1994). Soaps are water soluble potassium or sodium salt of fatty acid synthesized from and oil or their fatty acid chemically treating them with a strong alkaline substance (base). Soap may be defined as a chemical compound or mixture of chemical compounds resulting from the interaction of fatty acids or fatty glycerides with a metal radical or (organic base). The metals that are commonly used in soap production are

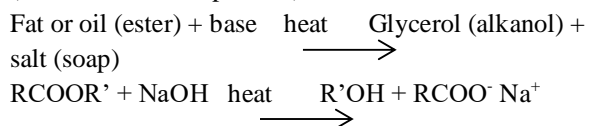
potassium and sodium, which produced water soluble soaps that are used for laundry and cleaning purposes (Kuntom *et al.*, 1994). The quality of soap is determined by the amount and composition of the component fatty acids in the starting oil. Blends of oil can be used in both the hot and cold soap production methods. Vegetable oil blend could be obtained by mixing different vegetable oil such as the mixture of coconut oil, palm kernel oil, groundnut oil and shea butter in different proportions (Kuntom *et al.*, 1994). Produced soaps of desired quality by blending various fatty acids of palm oil and palm kernel oil (PKO) and the quality of soap produced is comparable to the quality of commercially available soaps.

Fats and oil are composed of triglyceride; three molecules of fatty acids are attached to a single molecule of glycerol. It consists of sodium or potassium salts of fatty acids and is obtained by reacting common oils or fats with a strong alkaline solution in a process known as saponification. Soap belongs to the family of detergents which is a substance which improves the cleaning properties of water. In saponification, the fats hydrolyzed into free fatty acids which are then combine with the alkali to form crude soap. Glycerol (glycerine) is liberated and is either left in or washed out and recovered as a useful by-product, depending on the process employed (Cavich and Miller, 1994). Soaps are prepared by saponification of triglyceride from vegetable and animal source, for instance with a triglycerol containing 3 stearic acid (C18:0) units, the reaction with sodium hydroxide produce 3 mole of sodium stearate and 1 mole of glycerol (Francioni, 2002). This present study describes the cold process of soap produced from onion seed oil.

The cold process of saponification, is the simplest of the batch process, is limited to a small scale manufacturing. In this process, triglycerides and alkali are mixed together vigorously in a suitable container to form an ultimate mix which react slowly. The

mixture is stirred, usually with wooden stick for small scale preparation. This stirred is performed for few minute before the reactions reaches equilibrium. Solid fats such as tallow are melted separately and added once the reaction between liquid triglyceride components and alkali has begun, so as to utilize the heat of reaction to prevent solidification (Warra *et al.*, 2011).

Saponification is the chemical process of making soap that involves an exothermic reaction between sodium hydroxide and a fat (usually oils). Wash basin, (2012); (Pallas Athene Soap, 2009).



The production of quality soaps at cheaper rate has been a hitch to many soap producers in Nigeria; which has led to the production of soaps with poor qualities and high cost of production. Various vegetable oil of different qualities and prices for soap production could go a long way in the production of quality soaps for laundry, bathing and general cleaning purposes, still at minimized cost of production. The aim of this work is to produce soap from the extracted from onion seeds and determine the physicochemical characteristics of the prepared soap.

II. MATERIAL AND METHODS

- **Sample Collection and Preparation:** The *allium cepa L.* seed was procured from onion commercial producers at Aliero Onion market in Aliero town Kebbi State, Nigeria. The dried seeds were crushed into powder using mortar and pestle and were stored in a plastic container for oil extraction.
- **Extraction of Oil from Onion Seed:** The hexane extract was obtained by complete extraction using the soxhlet extractor, the extraction of onion seed oil was carried out using a soxhlet extractor and n-hexane as solvent. 150ml of n-hexane was poured into the round bottom of the soxhlet apparatus. 50g of the crushed onion seed was introduced into the thimble and fitted into the soxhlet extractor. The apparatus was assembled. The solvent in the set-up was heated to 70°C and the vapour produced was subsequently condensed by water flowing in

and out of the extraction set-up. This process of heating and cooling continued until a good quantity of onion seed oil was obtained for about 4hours. After the extraction, the thimble was removed while the remaining solvent in the extractor was recharged into the round bottom flask for the process to be repeated (Warra *et al.*, 2012). After the extraction, the solution (oil/ n-hexane mixture) was transferred back into the extractor for refluxing in order to separate the n-hexane from the oil. The heat mantle was the switched on at a temperature of 70°C, the solution started boiling and the n-hexane was distilled off from the oil, condensed by the condenser and collected in a stopper conical flask at the thermometer reading of 75°C when almost all the n-hexane suspected to have evaporated, the heating mantle was switched off and the oil in the flask was transferred into a beaker. The n-hexane which did not evaporate on recycling was evaporated directly using a heating plate. The oil was then allowed to cool after which it was weighed and stored in the refrigerator for further analysis.

III. METHODS

- **Physiochemical Analysis**
 - **Colour Determination of the Oil Seed:** The colour of the produced oil sample was determined by using several independent competent individuals. Oil colour was correlated using colour charts (Warra *et al.*, 2011).
 - **Determination of Percentage Yield:** Percentage yield was determined as reported by (Warra *et al.*, 2011); the extracted oil was transferred into a measuring cylinder which was placed over water bath for 30 minutes at 70°C to ensure a complete evaporation of solvent and the volume of the oil was recorded.
- $$\text{Oil Content (\%)} = \frac{\text{Weight of the oil}}{\text{Weight of the sample}} \times 100$$
- **Specific Density:** The specific density of the oil was determined as reported by (John, 2003). 25ml of the oil was measured; the weight of the oil was obtained by subtracting the weight of the cylinder

from the weight of the oil. The specific density of the oil was obtained using equation below.

$$\text{Density of Oil} = \frac{W_1 - W_0}{\text{Volume of the oil used}}$$

Where: W_1 = Weight of empty measuring cylinder + oil, W_0 = Weight of measuring cylinder

- Iodine Value Determination: 0.50g of the oil was dissolved in carbon tetrachloride in 100ml conical flask. 5ml of wjys iodine was added to the flask and allowed to stand for 2h in the dark at 25°C. 5ml of potassium iodide (KI) solution was added and the mixture titrated with 0.1M sodium thiosulphate ($\text{Na}_2\text{S}_2\text{O}_3$) using starch indicator. A blank determination was carried out and the iodine value was calculated using the formula.

$$\text{Iodine value} = \frac{C(V_1 - V_2)}{W}$$

Where: C = concentration of sodium thiosulphate solution, V_1 = volume (ml) of thiosulphate solution used in blank, V_2 = volume (ml) of thiosulphate solution used in the determination, W = weight of the sample (0.50g).

- Determination of Free Fatty Acid: 0.5GA of oil was boiled with 5cm³ of ethanol allowed to cool and 2 drops of phenolphthalein indicator was added, then titrated with 0.1M NaOH until pink colour disappear (AOAC, 1998).

$$\text{Free Fatty Acid} = \frac{V \times N \times 0.0282}{W} \times 100$$

Where: V = Titre value, N = Molarity of acid, F = equivalent weight of free fatty acid, W = weight of sample

- Saponification Value: 2g of the oil sample was added to the flask with 30cm³ ethalonic KOH and the flask was then attached to a condenser for 30 minutes to ensure the sample was fully dissolved. After the sample was cooled, 1cm³ of phenolphthalein was added and titrated with 0.1m HCl until a pink end point was obtained. The analysis was carried out with blank; blank was also prepared using the same reagents without the oil.

$$\text{Saponification Value} = \frac{(S - B) \times M \times 56.1}{\text{Sample weight (g)}}$$

Where: S = sample titre value, B = blank titre value, M = molarity of the HCl, 56.1 = molecular weight of KOH

- Peroxide Value: 2g of oil extracted was added to 22cm³ of a solution mixture of 12cm³ of chloroform and 10cm³ acetic acid. 0.5cm³ of saturated potassium iodide (KI) was added to the flask. It was cooled and allowed to stay with occasional shaking and titrated against 0.1M of $\text{Na}_2\text{S}_2\text{O}_3$ until yellow colour is almost gone. 0.5cm³ of starch indicator was quickly added and titration continued until blue colour just disappeared. A blank titration was also carried out at the same condition.

$$\text{Peroxide value} = \frac{(s - B) \times M \times 100}{\text{Sample weight (g)}}$$

Where: Peroxide value = meq peroxide per 100g of sample, S = volume of titrant (cm³) for sample, B = volume of titrant (cm³) for blank, M = molarity of $\text{Na}_2\text{S}_2\text{O}_3$ solution (in Eq/cm³), 100 = conversion of units (g/kg), W = weight of sample

- Acid Value: 100cm³ of neutral ethyl alcohol was heated with 2g of oil sample in a 250ml beaker until the mixture boils. And titrated with 0.1M KOH solution, using two drops of phenolphthalein as indicator with consistent shaking until a permanent pink colour was obtained.

$$\text{Acid Value} = \frac{M \times C \times TV}{W}$$

Where: M = molar mass of KOH (56.1), C = conc. Of KOH (0.1M), Tv = Titre value, W = weight of oil sample

- Refractive Index: This was determined at 20°C using an Abbe Refractometer (Reichert AR 700). The measurements were performed in triplicate and results were averaged.

- Soap Making: The cold process of saponification was used as reported by (Warra *et al.*, 2012). 50g of caustic soda was dissolved in 250ml of volumetric flask and fill to the mark with 150ml of distilled water, the same volume was prepared in another volumetric flask of soda ash. The concentration of both the caustic soda and soda ash solution was determined by hydrometer. The temperature of the solution was measured before the hydrometer was carefully immersed in the solution and held there until it floats. 72ml of onion seed oil was warm gently and transfer into a clean container, 32ml of caustic soda solution was added

and 18ml of soda ash solution was also added, the oil and alkali are mixed together vigorously in a suitable container to form an intimate mixture, the stirring was performed for about 10 minutes using wood stirrer until the reaction attained equilibrium. The saponification mixture was poured into a mould and allowed to dry and get hardened and formed a soap bar.

IV. ANALYSIS OF SOAP PRODUCTION FROM ONION SEED OIL

- Total Fatty Matter: 3g of finished soap was weighed and added with 75ml of distilled water and heated. The soap was dissolved in 10ml of 15% H₂SO₄ and heat until a clear solution was obtained. The fatty acid on the surface of the resulting solution was solidified by adding 3.0g of bee wax and reheated. It was allowed to cool to form cake. The cake was removed and blotted to dry and weighed to obtain the total fatty matter using the formula (Roila *et al.*, 2001).

$$\% \text{ Total fatty matter} =$$

$$\frac{A-X}{W} \times 100$$

Where: A= weight of wax+ oil, X= weight of wax, W= weight of soap

- Total Alkali: This was determined by titrating excess acid contained in the aqueous phase with standard volumetric NaOH solution. 1 gram of finish soap was weighed and 5ml of ethanol was added, 0.5 ml of 1M H₂SO₄ solution was added to the mixture and heated until the soap sample dissolved. Test solution was titrated against 1.0M NaOH using phenol phenolphthalein as indicator. The total alkali was obtained (AOCS, 1997).

$$\% \text{ Total Alkali} = \frac{VA - VB}{W} \times 3.1$$

Where: VA= volume of acid, VB= volume of base, W= weight of soap

- Percentage of Chlorine: 5grams of finished soap was weighed and 50ml of distilled water added and heat to dissolve. 250ml volumetric flask was used and 10ml of 15% Ca (NO₃)₂ was added and shake until the soap was dissolved, distilled water was added to the mark. The solution was filtered and methyl red 50ml was added to the filtrate. The solution was titrated against 10M H₂SO₄ until a pink colour was obtained. The solution was titrated against 0.1M AgNO₃ using K₂Cr₂O₇ as indicator, till a brick-red colour was obtained (Onyegbado *et al.*, 2002).

$$\% \text{ Chlorine} = \frac{\text{titre volume} \times 0.585}{\text{weight of soap}}$$

- pH Determination: The pH was determined using a pH meter (827 PH Metronm Model). 5g of the soap shaving were weighed and dissolved with distilled water in a 100ml volumetric flask. The electrode of the pH meter was inserted into the solution of the soap and the pH reading was recorded (Warra *et al.*, 2012).
- Form Ability Test: 2g of soap was weighed and 100cm³ distill water was added in a 500cm³ measuring cylinder. The mixture was shaken vigorously so as to generate foams. After shaking for some time, the cylinder was allowed to stand for 10minutes. The height of the foam in the solution was measured.
- Solubility Test: 5.0g of soap was added in 100cm³ of distilled water in 500cm³ of measuring cylinder. The solution was left for some minute and the time taken for complete dissolution was recorded (Warra *et al.*, 2011).

V. RESULTS AND DISCUSSION

Table:1. Physical and Chemical characteristics of oil Extracted from Onion Seed Oil

Parameters	Values
Colour	Light green
Physical state at room temperature	Liquid
Oil yield (%)	30.20±0.03
Specific density (g/cm ³)	0.86±0.02

Saponification value (mgKOH/g)	211.54± 0.05
Iodine value (gI ₂ /100g)	98.13±0.39
Acid value (mgKOH/g)	5.13±0.04
Peroxide value meq H ₂ O ₂	2.75±0.25
Refractive index	1.46±0.01
Free fatty acid	6.93±0.54

Values are mean ± standard deviation of three replicate results.

Table: 2. Physicochemical Characteristics of Soap production from Onion Seed Oil

Parameter	Value /Observation
pH	10.70±0.08
Solubility in water	Slightly soluble
Texture	Soft
Colour	Milky
Foam height (cm ³)	60.00±3.61
Total fatty matter (%)	36.33±3.51
Total alkali (%)	0.78±0.16
Chlorine (%)	0.48±0.02

Values are mean ± standard deviation of three replicate results.

VI. DISCUSSION

The oil extracted from onion seed using soxhlet extractor with n- hexane as solvent, liquid at room temperature. The onion seed has a percentage (%) oil yield of 30.20% which is lower than 50.28% as reported for onion seed oil (Warra *et al.*, 2012) and higher compared to 22.5% reported for garlic oil by (Gafar *et al.*, 2012), which indicates that the onion seed has high oil content. The colour of the oil was light green.

Density of the seed oil was found to be 0.86± 0.02g/cm³. This value is higher than 0.82±0.01 reported for onion seed oil by (Warra *et al.*, 2012) and lower than 0.90±0.02 reported for garlic oil (Gafar *et al.*, 2012). The onion seed oil with low density is an indication that it contains low molecular weight fatty acids (Afolabi *et al.*, 2008).

The saponification value of the oil was found to be 211.54±0.05 mgKOH/g which is higher when compared to the 192±1.00mgKOH/g obtained for garlic seed oil as reported by (Gafar *et al.*, 2012) and also higher than 203.00±0.71mgKOH/g reported for onion seed oil (Warra *et al.*, 2012) which is useful in

soap making. This indicates that the onion seed oil could be used for soap making since its saponification value falls within the range of these oils. High saponification values justify the usage of oil in soap making.

The free fatty acid (Oleic acid) determines the suitability for the oil for edibility or industrial uses. The free fatty acid value obtained from the onion seed oil was found to be 6.93±0.54 which is higher than the value for garlic seed oil 2.10±0.05(Gafar *et al.*, 2012) this shows that the onion seed oil it is suitable for eating.

The iodine value obtained was 98.13±0.39 in which there is no significance difference in the iodine value of the oils at 5% level of significance. The iodine values obtained is less than 100 which shows that the oil could be classified as non-drying oil. Non-drying oils have iodine values less than 100 which are useful in the production of soap.

The acid value of 5.13±0.04 mg KOH/g was obtained and is higher than value obtained for garlic oil which is 4.18±0.01 mgKOH/g (Gafar *et al.*, 2012) and 0.03± 0.01mgKOH/g for onion seed oil reported (Warra *et*

al., 2012). Thus the higher the acid value of oil the lower its storage quality and vice-versa. Therefore since the value obtained for onion seed oil high this indicated that it has a good storage quality.

The peroxide value 2.75 ± 0.25 meq H_2O_2 which is low than 3.00 ± 0.01 meq H_2O_2 reported for onion seed oil by (Warra *et al.*, 2012) and higher when compared to 2.50 ± 0.05 meq H_2O_2 reported for garlic oil (Gafar *et al.*, 2012) the value obtained for onion seed oil is low when compared to Palm Kernel Oil (PKO) 3.58 (Ogbuagu, 2008). This shows that oil seed oil can be kept for a very long period of time (Ogbuagu, 2008).

The refractive index was 1.46 ± 0.01 higher than refractive index of 1.44 ± 0.01 reported for onion seed oil (Warra *et al.*, 2012). The refractive index measures the purity of oil. Therefore the oil seed is pure. The onion oil soap was prepared by a product of saponification reaction between NaOH solution and oil extracted from onion seed. The prepared milky coloured soap has a foam height of 60cm^3 and the soap forms a clear solution and slightly soluble in water. Although foam generation has little to do with cleansing ability (Mainkar, 2000) it is of utmost importance to the consumer and therefore considered as a parameter in evaluating soaps.

The pH of the prepared soap was 10.70 ± 0.08 this value is slightly lower than pH of 11.03 ± 0.02 reported for onion oil soap (Warra *et al.*, 2012) and higher than 10.04 ± 0.04 for neem oil soap (Oyedele, 2002). The pH value obtained is comparably within the higher pH range of 9-11 but favorably higher than the pH range of 8-10 which are considered as high and low level respectively by the National Agency for Food and Drug Administration and Control (NAFDAC). Due to incomplete alkali hydrolysis resulting from the saponification process, this can be overcome by the addition of excess fat or oil any other super fatting agent to reduce the harshness of the soap. Super fatting soaps with 1-2% neutral oils or glycerin also resulted in the better quality of soaps that are free of cracks (Kuntom *et al.*, 1999).

The percentage of total fatty matter of onion soap was reported to be $36.33 \pm 3.51\%$ the value is closer to 36.66 ± 0.02 reported for onion oil soap by (Warra *et al.*, 2012) and lower than $63.75 \pm 0.07\%$ reported for

neem soap (Mak-mesah *et al.*, 2011). The differences in total fatty matter is responsible for high moisture content. Low total fatty matter values are due to the presence of unreacted NaOH in the mixture (Riola *et al.*, 2001). Soap which is high in moisture. However, dry skin needs total fatty matter of 80% this rehydrates the skin making it smooth and in addition to the high oil content within the soap acts as lubricant throughout the day (Oyedele, 2002).

The percentage of chlorine level in the soap is important as excess amount causes cracks of the soap. The % of chlorine value of (0.48) is lower than 0.053% reported for onion oil soap 1.15% reported for neem soap by (Mak-Mensah *et al.*, 2011). The % chlorine value of (0.48%) reported in this study indicates the value obtained is good enough to sustain the soap and prevent it from cracking.

The percentage of total alkali is the total alkaline materials present in the finished soap. This includes hydroxide, sodium (II) oxide, carbonates and bicarbonates. This is the decomposition of soap by known volume standard volumetric mineral acid solution, extraction and separation of liberated fatty matter (Riola *et al.*, 2001). The soda total alkali value was $0.78 \pm 0.16\%$ which is lower than $0.92 \pm 0.02\%$ as reported for onion seed oil soap and also higher than $0.24 \pm 0.01\%$ reported for neem oil soap (Mak-Mensah *et al.*, 2011).

CONCLUSION

From the results obtained in the analysis of the oil and the soap produced, it can be concluded that oil from onion seeds has the potentials in the soap production.

REFERENCES

- [1] Afolabi, S.I. (2008). Chemical qualities of oils from fresh and market vegetable crops within Kwara State Nigeria. *Niger. Soc. Experim. Biol.* 20(2): 71-75.
- [2] AOAC (1997). Official Methods of Analysis of the Association of Official Analytical Chemists, 7th Edn., Champaign, USA.

- [3] AOAC (1998). Official Methods of Analysis of the Association of Official Analytical Chemists, 6th Edn. Gaithersb Org, USA.
- [4] Cavitch and Miller S. (1994). The Natural Soap Book. Storey Publishing, ISBN 0-88266-888-9.
- [5] Francioni, J.B AND Callings M.L. (2002). Soap Making. Louisiana State Extension Circular. Available [www.http://ebookbrowse.com/gdoc](http://ebookbrowse.com/gdoc). [Accessed 22nd January, 2013].
- [6] Gafar, M.K., Itodo, A. U., Warra, A.A., Abdullahi, L. (2012). Extraction and Physicochemical Characteristics of Garlic (*Allium sativum* L.) oil. *Int. J. Food Nutr. Sci.* 1(2): 4-7.
- [7] John, K. (2003). Analytical Chemistry for Technicians. 3rd Edn. Lewis Publishers, *Kalyan Publisher*, New Delhi, India. Pp. 432-433.
- [8] Kuntom, A., Siew, W.L., Tan, Y.A. (1994). Characterization of Palm acid Oil *J.Am.Oil Chem. Soc.* 71, 525-528.
- [9] Mainkar, A.R. Jolly, C.L. (2000). Evaluation of Commercial Herbal Shampoos. *Int. J. Cosmetic Sci.* 22: 385-391.
- [10] Mak- Mensah, E.E. Firempong, C.K. (2011). Chemical Charactristics of Toilet Soap Prepared from Neem (*Azadirachtaindica A. Juss*) Seed Oil. *Asian Journal of Plant Science and Research* 1(4): 1-7.
- [11] Ogbuagu, M. N. (2008). Inhibitory Effect of Onion and Garlic Extracts on the Rancidity of Palm and Palm Kernel Oils. *Journal Chemistry Society of Nigeria*, vol. 33 (1) Pp 43-44.
- [12] Oyedele, A. O (2002). *Nigeria J. Nat. Products and Med.* 66: 26-29.
- [13] Pallas Athene (2009). Soap High-Quality Sodium Hydroxide (NaOH) and Potassium Hydroxide (KOH) for Making Soap. Organic , Vegan, Handmade Natural Soap. Available online at [http:// www. Certified-lye.com/lye-soap.htm](http://www.Certified-lye.com/lye-soap.htm) [Accecced 20th January, 2013].
- [14] Riola, A. Salmiah, A. Razmah, G.J (2001). Properties of sodium soap derived from palm-based hydroxystearic acid . *J. Oil Palm Res* 13 (2): 33-38.
- [15] Warra, A.A., Wawata, I.G and Gunu, S.Y (2011). Chemical Analysis and base – promoted hydrolysis of locally extracted shea nut fat. *Chem. Search Journal* 2(1): 12-15.
- [16] Warra, AA. Wawata, I.G., Umar, R.A., and Gunu, S.Y. (2012). Soxhlet extraction, Physicochemical Analysis and Cold process Saponification of Nigeria *Jatropha curcas* L. Seed oil. *Canadian J.Pure and Appl. Sci.* 6(1): 1803- 180719.