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Physico-Chemical Properties of Toilet Soaps Prepared From Coconut (*Cocos nucifera L.)* OIL, MELON (*Cucumeropsis mannii*) Seed Oil and the Blends of The Two

Ohoke Francis Okemini[1], Igwebike-Ossi Clementina Dilim[1], Yahya Qasseem Mohammed[2] and Ibekwe Fidelis Chukwuneme[3]
a.Department of Industrial Chemistry, Ebonyi State University, Abakaliki.
b. Standard Organization of Nigeria, Kaduna State Office
c. Federal College of Education (Technical) Asaba, Delta State, Nigeria

Abstract
High yield of oil from melon seed and its relatively low cost suggests its usefulness in soap production. This study investigated the physico-chemical properties of soap produced from coconut oil, melon seed oil and a blend of the two. The soaps were produced using the hot process method. Each of the oil was heated to 70^oC and saponified with potassium hydroxide solution in line with its lye factor. Sodium silicate, sodium carbonate and sodium lauryl sulphate were used as additives. The soap was allowed to set for two days and their physico-chemical properties were determined and compared with a popular commercial brand in Nigerian market. The physico-chemical properties of CO soap, MSO soap and the blend respectively were found to be: pH (9.7, 10.1, 10.0); total fatty matter (72.13%, 70.26%, 71.20%); total alkali (0.33%, 0.35%, 0.34%); free caustic alkali (0.11%, 0.10%, 0.10%); moisture content (10.41%, 9.68%, 10.07%); matter insoluble in water (6.78%, 7.02%, 7.0%) and foaming capacity (3.10cm^3,2.90cm^3, 3.20cm^3). Each of the soap samples had lower value of matter insoluble in water than a popular brand used as the reference standard but compares favourably with the control in cleansing capacity and other parameters.
Key words: *toilet soap, oil, coconut, melon seed*

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I. Introduction

Soap, simply put, is the sodium or potassium salt of long chain fatty acid obtained by alkaline hydrolysis (saponification) of fats and oils [1]. Fats and oil can be saponified either by hot or cold process. Consumers mainly use soap as a cleansing agent for washing and bathing. It can also be used as lubricant in textile spinning, as emulsifier in leather, plastic and cosmetic industries [2]. In this regard, soap can generally be classified as either laundry, toilet, antiseptic or medicated. The class of soap is determined by the type of oil, alkali and other additives in the soap formulation. Potassium soaps tend to be softer and more water-soluble than others.

Fatty acids are obtained as either saturated or unsaturated. Animal fat contains mainly the saturated fatty acid while vegetable oil contains the unsaturated fatty acids. In the simplest sense, saturated oils are solid at room temperature and are regarded as hard whereas unsaturated oils are liquid at room temperature and are regarded as soft oil. The degree of hardness and softness differs according to their sources. Hard oils contributes to hardness and/or lather in soap while soft oils contribute to conditioning. The main conditioning fatty acids are oleic, linoleic and linolenic. The more unsaturated bonds, the better the conditioning and the more easily it is absorbed by the skin, but the softer the oil in soap, and the more prone to oxidation [3]. Coconut oil has a high level of saturation. Palm oil, animal tallow and hydrogenated fats are hard while groundnut oil, cotton seed oil, fish oil melon seed oil and olive oil are examples of soft oil [4][5][6].

Apart from cleansing ability, toilet (bathing) soap is formulated to have minimal reaction with human skin. Although medicated and antiseptic soaps have inherent or incorporated medicinal values, quality parameters generally considered in soap are lathering ability, cleansing ability, total alkali (TA), free caustic alkali (FCA), total fatty matter (TFM), moisture content (MC), matter insoluble in water (MIW), chloride content and iodine value [7]. These parameters wholly depend on the type of the oil or fat, the type of alkali and the process involved in the production.

Besides providing fatty acids, different vegetable oils contain different levels of vitamins which protect the skin from harsh conditions. Vegetable oil like neem oil has antibacterial, antifungal and antiseptic properties used in pet soap, shampoo and bars to repel ticks, fleas and some pathogens [8]. Almond oil is readily absorbed through the layers of skin and prevents clogging of skin pores. Some vegetable oil acts as a softener, putting down a protective layer that holds moisture to the skin [9]. Therefore to get the best product entails choosing a combination of oils with different degrees of hard and soft proportions that give the required conditioning, lathering and possibly cosmetic and medical properties.

1.2 Coconut Oil

Coconut oil is obtained from the nut of coconut fruit. It contains a high proportion of glycerides of lower chain fatty acids. The oil is highly stable towards atmospheric oxidation. The oil is characterized by a low iodine value, high saponification value, high saturated fatty acids content, omega 6 and omega 3 fatty acids and is a liquid at room temperature of 27^0C [10]. It is one of the commercially important sources of medium chain fatty acids [11]. The most abundant fatty acid in this group is lauric acid. More than 90% of the fatty acids of coconut oil are saturated. The saturated character of the oil imparts a strong resistance to oxidative rancidity and accounts for its low iodine value [4].

It has been reported that increase in molecular weight of fatty acids in the oil increases solubility, improves the lathering properties up to lauric acid and deteriorate from lauric acid upwards, increases the stability of the lather, increases the ability to hold filler in solution, increases mild skin action but decreases the detergent action of soap [3]. This explains the reason why coconut oil soap lathers readily and profusely but not stably. It is hard but dissolves more readily in water than do soaps from the hard oils. It can also retain a good amount of water, and take up large quantities of fillers like sodium carbonate [4].

1.3 Melon Seed Oil

Melon (Citrullus lanatus) thrives in tropical, subtropical, desert and temperate regions of the world [12]. Research studies have shown that its seeds contain 42 – 57% oil [12][13][14]. The high oil yield suggests its usefulness in soap, paint and printing ink industries, and can also be considered a better alternative when compared to other vegetable oil. The fatty acid profile of melon seed oil has been reported to compose of palmitic, stearic, oleic, linoleic and linolenic acids [15]. The total saturated and unsaturated fatty acids contents of the melon seed oil are in the range of 18.9 -38.1% and 71.9 - 81.9%, respectively. The palmitic acid is the predominant saturated fatty acid while linoleic acid is the most abundant (62%) of the unsaturated acids [12][15].

Melon seed oil is a semidrying oil [15] and has iodine value of 114.94 gI_2/100g. Semidrying oils, on prolonged exposure to oxygen, do not form a hard dry film. This property is utilized in their application for soap making [16][17]. The high saponification value of melon seed oil is comparable to palm oil, olive oil, soyabean oil and linseed oil. Therefore it is good for soap making and possibly shaving cream [15]. The free fatty acid value of melon seed oil is below 5% recommended as the maximum for non-rancid oil [18]. In the same vein, the low peroxide values of the oils indicate that they are less liable to oxidative rancidity at room temperature [9]. Melon seed oil also contains monoglycerides, phosphatides, cerebroside, steroid, terpenes, fatty alcohol and fatty acid-soluble vitamins which are sources of antioxidant and bioactive compounds [19] that are useful in soap making. In this work, a comparative study of soaps from coconut oil, melon seed oil and their blends was done.

II. Materials And Method

3.1 Materials

Melon seed and coconut used in this study were bought from a market in Abakaliki, Ebonyi State, Nigeria. Potassium hydroxide, sodium silicate, sodium carbonate, foaming agent, perfume were supplied by Glisco Laboratory Chemicals Limited, Enugu, Nigeria.

3.2 Methods

(i) Extraction of coconut oil

Coconut oil was extracted using the Wet Mill Method [20]. Mature and healthy coconut seeds were shelled to obtain the nut. The nut was washed with clean water shredded with scraper and blended with electric blending machine to obtain coconut paste. The paste was wrapped in a muslin cloth and, by squeezing; the milk was filtered into a transparent glass jar. The milk was left in the jar at room temperature for 24h. The coconut milk and oil separated and a layer of curd appeared at the top of the jar. The curd was scooped out with a spoon and discarded; leaving the pure virgin coconut oil in the jar.

(ii) Extraction of Melon Seed Oil

Healthy melon seeds were shelled, screened and cleaned to remove contaminants. They were ground to paste using electric blending machine. The oil was extracted by continuous extraction in soxhlet apparatus

(cehmglass) for six hours using petroleum ether (40-60^{o}C) as solvent according to the method described by Edidiong et al. 2013 [9] and Oluba et al. 2008 [12]. At the end of the extraction, the extraction solvent was evaporated in a rotary evaporator.

(ii) Production of Soap

The concentration and amount of alkaline solution used in the formulation was obtained using the saponification value of the oil. Saponification value is the amount of alkali (KOH) required to saponify a particular amount (1g) of oil. The saponification value of coconut oil is 0.26 while that of melon seed oil is 0.19 [5]. The amount of water required for 100g of oil was calculated as shown below.

(a) Amount of lye required for 100g of oil = 100 x sap value.

(b) Amount of water required for 100g of oil = $(\frac{sap\ value}{0.3} - sap\ value)\ x100g$

(c)The amount of water required for 100g of mixed oil = $(\frac{mean\ of\ sap\ values}{0.3} - mean\ of\ sap\ values)\ x100g$

The recipes used for production of soap from coconut oil soap, melon seed oil and blended coconut and melon seed oils are shown in Table 1.

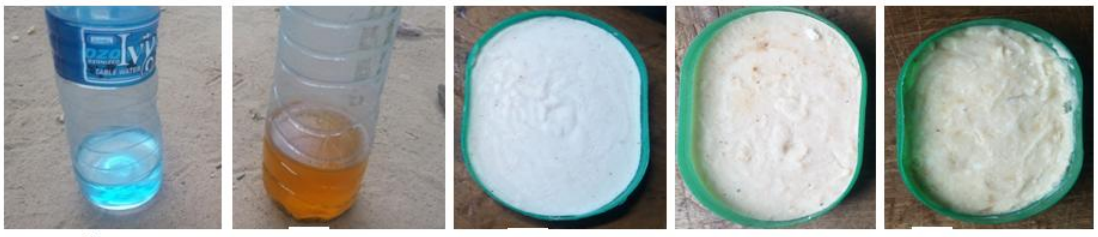
Procedure

Coconut oil soap was produced using the hot process method [21]. Coconut oil (100g) was weighed into the saponification vessel. Sodium silicate (2g), sodium carbonate (1g) and sodium lauryl sulphate (0.5g) were poured into the vessel and stirred. Potassium hydroxide (26g) was measured and 60ml of water slowly added to it. The mixture was stirred till all the alkali dissolved. The coconut oil was heated to 70^{0}C and the potassium hydroxide solution was then added in a thin stream while stirring continuously. The stirring was done in one direction to prevent phase separation. The heating was continued till the mixture became thick and consistent. The soap was poured into moulds and allowed to cool for 24h. It was then removed from the moulds and allowed to harden for two days.

With variance in lye component, this same procedure was repeated for melon seed oil soap. For blended oil soap, 70g of coconut oil, 30g of melon seed oil and same additives were used. The lye was obtained by dissolving 23.9g of potassium hydroxide in 55.8 ml of water. The same procedure for coconut oil soap was also repeated.

Table 1: Recipes used for production of soap from coconut oil, melon seed oil and their blend

Type of soap	**Coconut oil (CO)**		**Melon seed (MSO)**		**Blended CO and MSO**	
Ingredients	Wt%	Wt (g)	Wt%	Wt (g)	Wt%	Wt (g)
Potassium hydroxide	13.68	26.00	11.38	19.00	13.07	24.00
Oil	52.63	100.00	59.88	100.00	54.67	100.00
Soda ash	1.05	2.00	1.20	2.00	1.09	2.00
Silicate	0.53	1.00	0.60	1.00	0.55	1.00
Water	31.58	60.00	26.35	44.00	30.07	55.00
Perfume	0.26	0.5	0.30	0.5	0.27	0.5
Sodium lauryl sulphate	0.26	0.5	0.30	0.5	0.27	0.5
Total weight of soap	100	190	100	167	100	182.9



a b c d e

Fig. 2: a = coconut oil, b = melon seed oil, c = coconut oil soap, d = melon seed oil soap, e = blended oil soap

Determination of the Physicochemical Properties of the Produced soap

Standard procedures as described by Mark-Mensah and Firempong[8], Onyango et al. [7], Ngobizitha et al.[21] and AOCS [22] were used for the analysis of physicochemical properties of soap with slight modifications as explained. EVA soap, a popular commercial product in Nigeria was used as control.

(i) pH

A method described in Nqobizitha et al.[21] was modified and used to determine the alkalinity of the soaps. Ten grams of each of the soap samples was weighed and dissolved in distilled water and made up to 10% soap solution. The pH of the soap solution was determined using a pH meter (Hanna pH 211 microprocessor pH meter) and recorded.

(ii)Total alkali

The total alkali is determined by titrating excess acid contained in the aqueous phase with standard volumetric NaOH solution. A method described by Onyango et al [7] and Mak-Mensah and Firempong [8] was modified and used. Ten grams (10g) of finished soap was weighed and 100 ml of neutralized alcohol was added to it. Five milliliters (5ml) of 1 N H_2SO_4 solution was added to the mixture and heated till the soap sample dissolved. Test solution was titrated against 1 N NaOH using phenolphthalein as indicator. The total alkali was obtained with the formula;

% Total alkali = $\frac{Va-Vb}{W}\ x\ 3.1$

Va = Volume of acid, Vb= Volume of base and W= weight of soap.

(iii) Free caustic alkali

Free caustic alkali was determined using the method described by Onyango et al [7]. Five grams of finished soap was weighed and dissolved in 30 ml of ethanol. A few drops of phenolphthalein indicator and 10 ml of 20 % $BaCl_2$ were added. The resulting solution was titrated against 0.05 M H_2SO_4. Free caustic alkali- the volume of the acid obtained was calculated using the formula; NaOH $= \frac{Va}{W}\ x\ 0.31$,

Va = Vol. of acid and W = weight of soap.

(iv) Total Fatty Matter (TFM)

A method described by Mak-Mensah and Firempong [8] was used. The total fatty matter test is carried out by reacting soap with acid in the presence of hot water and measuring the fatty acids obtained. Ten (10) grammes of finished soap was weighed and 150 ml distilled water was added and heated. The soap was dissolved in 20 ml of 15 % H_2SO4 while heating until a clear solution was obtained. Fatty acids on the surface of the resulting solution was solidified by adding 7g of bees wax and reheated. The set up was allowed to cool to form cake. Cake was removed and blotted to dry and weighed to obtain the total fatty matter using a formula: % TFM = $\frac{A-B}{W}\ x\ 100$,

A= weight of wax+ oil, B = weight of wax and W= weight of soap.

(v) Moisture Content (MC)

Five grams of the soap was weighed into a Petri dish and kept in the oven at a temperature of 105± 2^{o}C for 1.5h. It was cooled in desiccators and weighed after successive heating until a constant weight was reached. The moisture content of the soap sample was calculated using the formula: MC = $\frac{C+S-F}{S}\ x\ 100$

C = Weight of the Petri dish, S = weight of soap sample, F = final weight after drying.

(vi) Matter Insoluble in Water (MIW)

Analysis of matter insoluble in alcohol was determined by a method described in AOCS [22], with modifications. Ten grams of soap sample was dissolved in 150 ml of water in the oven at 60^{o}C. Then the solution was filtered with a known weight of filter paper. The residue was then washed with hot water and dried in the oven at 105^{o}C for 30 minutes. Matter insoluble in water was calculated using the formula: MIW = $\frac{W2-W1}{S}\ x\ 100$

W_1 = weight of filter paper, W_2 = weight of filter paper + residue, S = Weight of sample.

(vii) Foaming Capacity (FC)

The method reported by Nqobizitha *et al.*[21] for jatropha soap detergents was used. Exactly 0.2 grams of each of the soap samples was added to a 100 ml measuring cylinder containing 10 ml of distilled water. The soap mixtures were shaken vigorously so as to generate foam. After shaking for about 2 minutes, the cylinder was allowed to stand for 10 minutes. The height of the foam in the solution was measured and recorded. The foaming capacity was calculated from difference in height.

(viii) Cleansing Ability

A measured amount of blue ink (2g) was dropped on three pieces of white cloth of size 6cm x 6cm each. Each of the pieces of cloth containing the ink spot was placed in each of the beakers containing each of the soap solutions. The piece of cloth was removed after 2 minutes of continuous shaking in the beaker and rinsed with tap water. The level of disappearance/ dispersion of the ink spot was assessed and recorded as the cleansing ability of the soap.

III. Results

Table 2: Determined parameters for soaps produced from coconut oil (A), melon seed oil (B) and their blend (C).

Sample	pH	TFM (%)	TA (%)	FCA (%)	MC (%)	MIW (%)	FC (Cm^3)	CA
A	9.7	72.13	0.33	0.08	10.41	6.78	3.10	Cleaned
B	10.1	70.26	0.35	0.11	9.68	7.02	2.90	Cleaned

C	10.0	71.20	0.34	0.10	10.07	7.0	3.20	Cleaned
Control	10.2	73.01	0.36	0.09	9.42	11.26	3.10	Cleaned

A = Coconut oil soap, B = melon seed oil soap and C = blend oils soap. TFM = total fatty matter, TA = total alkali, FCA = free caustic alkali, MC = moisture content and MIW = matter insoluble in water, FC = foaming capacity and CA = cleansing ability.

IV. Discussion

From the results, the pH of coconut oil soap (10.1), melon seed oil soap (9.7) and the blended oil soap (10.0) were lower than that of the Control (10.2) but consistent with the normal pH range for soap 8-10.5 (http://www.soap-body-and-spa.com) and 9-11 [23]. They are lower than pH values of 9.38 reported by Warra et al. [24] for cotton seed oil soap and 5.5 - 8.0 reported by Mak-Mensah and Firempong [8]. Soap, as the salt of a weak acid and a strong base, should be alkaline (pH~10) in aqueous solution. High pH value which results from incomplete alkali hydrolysis during saponification can be controlled by the addition of excess fat or oil to reduce the harshness of the soap [24]. Although alkalinity is important since it favours detergency [25], alkaline substances neutralize the body's protective acid mantle that acts as a natural barrier against bacteria and viruses. Healthy skin has a pH 5.4 to 5.9 (http://www.godsebook.org/soap). The pH values of the soap samples in this study indicate that all the soap samples are not corrosive to the skin.

Total fatty matter (TFM) is a parameter that identifies the amount of fatty matter present in soaps. The total fatty matter (TFM) of coconut oil soap was 72.13%, melon seed oil soap was 70.26%, and that of blended oil soap was 71.20%. These values were found to be lower than the control (73.01%), higher than 58% reported by Warra et al.[24], 55.45% obtained by Ogunsuyi and Akinnawo [26] and 62% reported by Beetseh et al. [27], but consistent with 65-70% obtained by Taiwo et al, [28], 71% - 84% reported by Viorica et al. [29], and 63% - 75% obtained by Mak-Mensah et al, [8]. The differences in TFM account for the variations observed in moisture contents of soap and it depends on the type of fatty materials and the saponification method used. The accepted percentage value for toilet soap is between 76-77% [4]; although dry skin prefers soap with TFM as high as 80%. The oil re-hydrates the skin, making it smooth, and acts as a lubricant [7]. However, the TFM values for the soaps investigated were slightly below the range of TFM required for toilet soap.

Total alkalinity (TA) is the total alkaline material present in soap. They include alkaline components such as hydroxides, sodium (II) oxide, carbonates and bicarbonates. From Table 2, it is evident that coconut oil soap had the highest value of TA (0.35%), followed by blended oil soap (0.34%) and then melon seed oil soap (0.33%). Each of the soap prepared had TA value lower than that of the Control (0.36%) and 0.57% obtained by Warra et al [24] but higher than 0.24% obtained by Mak-Mensah and Firempong [8]. However, it is consistent with the acceptable value (0.34%) [7].

The free caustic alkali is the amount of alkali that prevents soap from becoming oily. It is one of the parameters that determine the abrasiveness of any given soap [30]. This mostly results from incomplete saponification. From this analysis, free caustic alkali value of 0.08, 0.11 and 0.10 were obtained for coconut oil soap, melon seed oil soap and blended oil soap respectively. These vaues are below 0.2% obtained by Ogunsuyi and Akinnawo [26] for toilet soap, 2.95% obtained by Beetseh et al. (2013), and 1.24% obtained by Taiwo et al. [28]. In line with requirement of International Standard Organization (ISO 1999), the maximum permissible FCA value of soap is 0.2%. Excess free caustic alkali causes skin irritation. This study showed that the free caustic alkalinity of all the analyzed soap samples were below the maximum set limits, hence the amount of free caustic alkalinity in the analyzed soap samples had no adverse effect on cloth or skin

Moisture content is a parameter that is used to assess the shelf life of soap. On storage, high moisture content leads to the reaction of excess water with un-saponified fat to give free fatty acid and glycerol. From the values obtained in the analysis, moisture content of 10.41%, 9.68% and 10.07% were obtained for coconut oil soap, melon seed oil soap and blended oil soap respectively. These values were slightly above 9.42% obtained for the Control, but below 10% - 16.2%) reported by Viorica et al. [29] and 18.8% - 22.5% obtained by Osuji et al. [31] for palm oil soap. The results obtained in this study fall within the standards of 10 – 15% [32].

Matter insoluble in water (MIW) is a parameter that is used to determine the purity of soap (Viorica et al, [29]. It is the measure of non-soap ingredients known as builders or fillers such as sodium silicate, sodium phosphate, sodium carbonate and minor constituents such as bleachers, whitening agents and fluorescing agents in the finished product. From the values obtained in the analysis (Table 2), coconut oil soap had the lowest level of matter insoluble in water (6.78%), followed by blended oil soap (7.0%) and melon seed oil soap (7.02%). These values are below 11.28% obtained for the control. The results obtained compare well with the results obtained by Ogunsuyi and Akinnawo [26], of 3.4% to 14.80%. It is expected that in industries, many non-soap ingredients are added to modify the soap qualities and reduce cost. This probably makes the difference in MIW between commercial soap and laboratory soap. The soap with high MIW values suggests that it contained high level of impurities which may also result from the impurities of alkali used [26][29].

Foaming capacity (lathering power) refers to the amount of foam formed by a given soap solution. Although foam generation has little effect on cleansing ability, it is an importance parameter to be evaluated in soap [23]. Foam helps to suspend dirt by creating greater surface tension in water and trapping dirt for easy removal through rinsing. Foam, which is generated by agitating surfactant solutions, cushions the effect of rubbing the skin which occurs during bathing. The foam height of coconut oil soap, melon seed oil soap and mixed oil soap were 3.10cm, 2.90cm and 3.20cm respectively. The results showed that melon seed oil soap had lower foaming capacity than the control (3.10), but mixed oil soap had a superior foaming capacity than the control. Therefore mixed oil soap had the highest foaming capacity among the three soap samples.

As shown in the results for cleansing ability (Table 2), all the ink stains on the piece of cloth were washed off by the three formulated soaps and the control. Each of the three soap samples is therefore comparable to the reference standard in terms of its ability to emulsify oily dirt from clothing and human skin.

V. Conclusion

Coconut oil soap, melon seed oil soap and mixed oil soap were prepared from mechanical pressing of coconut and melon seed. From literature search and the results obtained in this study, it could be concluded that coconut oil soap is grade 1 soap and melon seed oil soap is grade 2 soap. The need to produce relatively low cost soap that strikes a balance among the physicochemical parameters necessitated the use of mixed oil. Minimal matter insoluble in water, minimal moisture content, high amounts of total fat matter and low levels of caustic alkalinity are the basic consumer demands. Soap produced from mixed coconut oil and melon seed oil balanced on these parameters and can be regarded as high quality soap. Therefore production of soap from blends of coconut oil and melon seed oil helps to reduce cost and at the same yields high quality soap.

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