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Original Research Article

Effect of Extraction Methods on the Physico-chemical Properties of Shea Butter Produced from Selected Shea Kernels

[1]Saba, A.M., *[1]Baba, A.H., [2]Mohammed, I.Y. and [1]Mohammed, A.

[1]Department of Chemical Engineering, The Federal Polytechnic PMB 55 Bida, Nigeria.
[2]Department of Chemical Engineering, Abubakar Tafawa Balewa Ubiversity, PMB 0248, Bauchi, Nigeria.
*aliyuharunababa2003@gmail.com

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ABSTRACT

Shea kernel processing method and the method used in the extraction of the shea butter significantly influence the properties of the shea butter. Traditional extraction, screw expression and solvent extraction methods were used to extract butter from both optimally processed and locally processed kernels. The variations in roasting temperature, roasting time, particle size of the kernel, solvent type and time of contact between the pulverized kernel and the solvent have significant effect on the physico-chemical properties. Shea butter produced from optimum kernel processing conditions of 4.3-day conditioning period, 86 ^{o}C processing temperature and 120 minutes boiling duration using optimum traditional extraction method at a processing condition of 30 ^{o}C roasting temperature, 60 min roasting time, 300 rpm kneading speed and 6.4 h time lag between kernel milling and Shea paste kneading resulted in physical properties of 36.05%, 1.4418, 0.92 gcm^{-3}, 30 ^{o}C, for yield, refractive index, density and melting point respectively. The chemical properties were 0.8936%, 1.53 meqkg^{-1}, 181.45 meqKOH^{-1}g^{-1} and 10.99% for free fatty acid, peroxide value, saponification matter and unsaponifiable fraction respectively. Results for shea butter produced from optimally processed kernel but using mechanical screw expression and later solvent extraction method were also reported. This work established that best quality shea butter is obtained from optimally processed shea kernel using optimum traditional extraction method.

1. INTRODUCTION

Researchers and scientist all over the world are continuously looking for ways and means of improving the yield and quality of vegetable oils to meet both domestic and industrial applications (Akihisa *et al*., 2010).

Shea butter is one of these numerous vegetable oils and it is a fatty extract obtained from the kernels of shea fruit. It also contains fatty acids usually oleic, stearic, palmitic, linoleic and aracchidic acids with oleic and stearic acids predominating and together constituting about 85% of the fatty acid content of shea butter (Coulibaly *et al.*, 2009; Julius *et al.*, 2013). The presence of these fatty acids in shea butter varies in proportion depending on the source of the shea nuts. The nut is obtained from the shea tree which is a native of Africa and it is either called *Vitellaria paradoxa or Butyrospernum parkii* in West Africa or *"nilotica"* in East Africa. Examples of countries where shea trees are found include Senegal, Mali, Ivory Coast, Burkina Faso, Togo, Ghana, Benin, Niger, Nigeria, Cameroon and further east in Uganda, Sudan and Ethiopia (Walter *et al*., 2003).

The shea tree bears fruit up to 4 cm in diameter and contains a nut. It grows wildly up to 9-12 m in height and begins to bear fruits after approximately 15–20 yrs. The tree gets matured at about 45 years and continues to bear fruits for about 200 years (Alander, 2004). NASPAN (2018) and Koloche *et al.* (2016) reported that Nigeria is well endowed with this tree accounting for about 50 to 60% of West African shea tree population. In Nigeria, these trees are concentrated in Niger, Kwara, Nassarawa, Zamfara, Adamawa, Edo , Yobe, Plateau, Kaduna State and some parts of Kebbi, Bauchi, Kogi, FCT and Oyo State. From statistics, even though not updated, Koloche (2016) has shown that 40–50% of these shea trees are found in Niger State. The tree starts flowering and fruiting in January and harvest begins between May to June through to August. After the harvest, the pulp is removed, the nuts are dried, the shell is removed and the shea kernels processed for shea butter. This shea butter is produced by three methods namely, solvent extraction, mechanical expression and a third method loosely referred to as traditional method (Abdul-Mumeen et al., 2019). The solvent extraction method is an expensive process of shea butter extraction involving the use of organic solvents such as straight chain hydrocarbons, alcohols, chlorinated hydrocarbons and ketones to recover the oil from the kernels (Lovett, 2013). The shea butter from this method is highly refined and hence contains little or no bio-active components. The mechanical expression method involves the use of expellers or hydraulic press which exerts high pressure on the heated kernels to express the shea oil. The shea oil obtained from this method has high free fatty acid (FFA) and requires refining before use (Julius et al., 2013). The traditional method of shea butter extraction is the most preferred method because of its retention of bioactive fractions (Addaquay, 2004; Lovett, 2004). It involves the use of tools like fire wood, pots, pestle and mortar, milling machine and plastic vats. The product from this method of production, if controlled gives a grade of shea butter referred to as unrefined with bio-actives (triterpines alcohol, vitamin E, phytosterols and catehins) greater than 5%. This grade A shea butter has FFA less than 1%, iodine value between 30 and 75 g/kg, peroxide value less than 10 meq/kg, bioactive fraction greater than 5 $g(kg)^{-1}$ and moisture content less than 0.05% and is the optimum in quality. It is more desirable in both cosmetics and pharmaceutical industries and attracts more premiums (William and Isemede, 2015) and is rarely obtained among shea butter producing countries including Nigeria (Lovett, 2013).

In Nigeria, the traditional method of shea butter production is done mostly by children and rural women with little or no formal training and hence produce shea butter with attendant low yield, poor quality and irregular properties (Chukwu and Adigzi, 2008; Munir et al., 2012; Obibuzor, et al., 2014; Ouattara et al., 2015; Saba et al., 2017). Consequently, the quality produced do not meet African Regional Standard Organisation (ARSO) standard.

Nigerian shea butter processing methods are labour-intensive, characterized by poor efficiency and low yield. The resultant product often does not meet required quality for export marketing. The economic return is also not attractive. There is very scanty documentation on shea kernel quality with shea butter extraction. Also lacking is adequate documentation to show variation of shea butter quality with shea kernel roasting temperature, shea kernel roasting time, kneading speed and time lag between milling and kneading. There is therefore the need to investigate the influence of extraction methods on the physico-chemical properties of shea butter produced from selected shea kernels.

2. MATERIALS AND METHODS

2.1. Material Collection

Materials used in this research are fresh shea nut obtained from Sonmaigi, Pati-Ndeji and Chengudu villages via Bida (Lat. $9^o4'N$, Long. $6^o1'E$) where shea butter was obtained. Shea shell briquette was used to generate heat for shea nut drying in the traditional oven. Local starch from cassava was also used as a binder in briquette production. The traditional oven used was constructed using clay obtained from The Federal Polytechnic, Bida.

2.2. Shea Butter Production from Traditional Extraction Method

An Atlas weighing balance was used to measure 4 kg of optimum shea kernel produced after 4.33 day of picking the fruit, boiled in water for 120 min and dried in an oven at 86 ºC. The nuts were crushed using a wooden pestle and mortar. Two kilogram (2 kg) of the crushed kernels were then processed by warming the kernels in a laboratory oven at 30 ºC for 60 min. The warm nuts were then milled with a 1.5 kW Atlas milling machine. The milled kernels in paste form was allowed to cool for 6.5 h and kneaded for shea butter using a laboratory variable mixer at 300 rpm. The shea butter obtained was analyzed for both physical and chemical properties. The procedure was repeated with locally produced shea kernel obtained from an open market in Agaie, Niger State, Nigeria.

2.3. Shea Butter Production from Mechanical Screw Press

Four kilogram (4 kg) of optimum shea kernel described in Section 2.2 was crushed with wooden mortar and pestle and mixed with 0.5 kg dried shea shell to increase the fiber content and increase the much-needed friction to aid extraction in the screw press. The press was allowed to run dry until the temperature of the barrels was 70 ºC. The mixture of the crushed kernel and shea shell was then fed into the Rosedowns mini-screw press (Plate I). As the barrel rotates in the clockwise direction, the rings compress and shea butter is expressed out through the rings arranged. The crude shea butter was placed on a 32 cm Whatman filter paper and put in a laboratory oven at 50 ºC. The filtered shea butter collected was characterized for both physical and chemical properties.

Plate 1: Rosedowns mini-screw press

2.4. Shea Butter Production by Solvent Extraction

The shea butter sample from solvent extraction was produced according to the method developed by Ajala *et al.* (2016). The optimum kernel described in Section 2.2 was used for the extraction. The kernel was pulverized using a laboratory blender and then sieved in a laboratory shaker to between 1 mm – 2 mm particle

size. A 30 g pulverized shea kernel in 346 mL n- hexane was extracted for 40 min in a 500 ml Soxhlet apparatus.

2.5. Analysis of Shea Butter

The physical properties of shea butter samples (A, B, C, D and E) produced from different methods of Shea butter extraction were analyzed for yield, density, refractive index and melting point. The sample designation was as follows. Sample A= shea butter produced from optimally processed Shea kernel using optimum traditional extraction method, Sample B= shea butter produced from optimally processed Shea kernel using mechanical screw expression method, Sample C= shea butter produced from kernels obtained from Agaie market using optimum traditional extraction method, Sample D = shea butter produced from kernels obtained from Agaie market using conventional traditional extraction method and Sample E = shea butter produced from optimally processed shea kernel using solvent extraction method. The shea butter samples produced in this research were analysed according to international standards (AOCS, 1994; ISO 3960, 2005; ISO 3657, 2005; AOAC, 2000). Other physical parameters were analysed as described below.

2.5.1. Determination of percentage oil yield

The percentage oil yield was determined by using Equation 1.

$$Oil\ yield = \frac{W_u - W_e}{W_u} \times 100 \quad (1)$$

where W_u = weight of unexpressed sample (g) and W_e = weight of expressed sample (g)

2.5.2. Determination of refractive index

The refractive index of shea butter was determined using the AOAC (2000) method. Two gram of butter was melted in a trident oven and filtered through 11 cm Whatman filter paper to remove impurities. Few drops of the filtered butter were then placed on the glass slit provided on the refractomer. The temperature of the refractometer was adjusted to 40 ^oC and the refractometer was adjusted while focusing on the eye piece of the refractometer and the reading on the screen was recorded as the refractive index.

2.5.3. Determination of density

Two millilitre (2 ml) of the shea butter sample was measured in a Gay-Lussac bottle and was calibrated with water. The weight of completely dried Gay Lussac bottle was weighed and recorded as ‘a’. The weight of dried Gay-Lussac bottle filled with water was measured and recorded ‘b’. The weight of dried Gay-Lussac bottle filled with shea butter at 40 ^oC was measured and recorded ‘c’. The specific gravity was then calculated using Equation 2 and the density was calculated from Equation 3.

$$\text{Specific gravity} = \frac{c-a}{b-a} \quad (2)$$

$$\text{Specific gravity} = \frac{\text{Density of shea butter}}{\text{Density of water}} \quad (3)$$

2.5.4. Determination of melting point

The melting point of shea butter was determined by the opened-tube capillary-slip method (Girolami et al., 1999). Here, 10 g of shea butter was melted in an oven and mixed thoroughly to ensure that it was

homogeneous. A 20 mm long capillary was inserted inside the molten shea butter so that the sample was sucked to a height of about 10 mm. The tube was chilled with ice cubes to ensure that the shea butter is solidified. The tube was held in a small beaker and placed for 1 hr in a refrigerator. The tube was removed and attached with a rubber bind to the thermometer bulb so that the lower end of the capillary tube and the thermometer bulb were at the same level. The thermometer with the capillary tube containing the shea butter was placed in water of about 10 ^{o}C in the Thiele tube. The heating temperature was increase gradually by heating the side tube of Thiele tube at rate of 2 ^{o}C/min until the temperature reached 35 ^{o}C and thereafter at the rate of 0.5 ^{o}C/min. The temperature of the water was observed when the sample column begins to rise in the capillary tube. The average of 2 such separate determinations was recorded as the melting point, provided the reading did not differ by 0.5 ^{o}C.

3. RESULTS AND DISCUSSION

Figure 1 shows the yield of shea butter obtained from all the extraction methods used in this research. The values of their yield fell within 28.6% to 46.0% with optimum kernel using solvent extraction method (Sample E) having the highest yield of 46.0%, closely followed by optimum kernel using mechanical screw extraction method (Sample B) with 40.4% and local kernel using conventional extraction method (Sample D) having the lowest yield of 28.6%. The highest yield recorded for Sample E is consistent with the findings of Ajala *et al.* (2012) and also coupled with the fact that the solvent used for the extraction has a high affinity and selectivity to leach out oil from the milled kernel and form a solution with the oil (Lovett, 2013). The high yield also from Sample B may be due to high temperature and pressure generated by the barrels and addition of shea shell which increases friction and compression between the barrels and the kernels, leading to release of more oil. The optimization of the traditional extraction method (Saba, 2019) alone increased the percentage yield of shea butter from 28.6 to 30.3%, while the optimization of both the traditional extraction method and shea kernel process increases the yield of shea butter from 28.6 to 36.05% when compared with the conventional method and kernels locally produced. This increase may be as a result of optimizing the process variables both in shea kernel processing and traditional shea butter extraction (Aculey, 2012).

Figure 2 shows the trend of refractive index obtained for shea butter produced from each method. The trend shows that optimum kernel using optimum traditional extraction method (Sample A) had the lowest refractive index of 1.4418 while local kernel using conventional extraction method (Sample D) had the highest refractive index of 1.62. The value of refractive index of optimum kernel using solvent extraction method (Sample E) was found to be 1.50 while that of optimum kernel using mechanical screw extraction method (Sample B) was 1.470. This figure obtained for Sample B is not significantly different from the African Organization for Standardization (ARSO) value of 1.4635**.** This may indicate little or no impurity or lower carotene content in shea butter samples from this process as compared with ARSO, (2017). Optimization of shea kernel used in traditional method of extraction increased the brightness by lowering the refractive index from 1.62 to 1.44 while the combined effect of optimization of the traditional extraction method and shea kernel has similar effect by lowering the refractive index from 1.6 to 1.44. The figure for Sample D is higher than the ARSO standard implying lower quality than required by the standard.

Figure 3 shows the density of shea butter samples obtained from the various methods studied. The densities of shea butter obtained from all extraction methods studied are between 0.90 and 0.96 gcm^{-3} which are not significantly different from the African Organization for Standardization (ARSO) approved standard average of 0.91 g/cm^3 for shea butter. The shea butter obtained from Sample E with density of 0.90 g/cm^3 is less dense compared to others, probably because it has no any dissolved impurity and also all the bio-active fractions are dissolved in the solvent, while Sample D has density of 0.96 g/cm^3 and by implication is heavier when compared with other samples and may also contain some dissolved impurities. Also, the closeness of these densities to the African Organization for Standardization standard indicates the seemingly lack of adulteration or contamination of the shea butter samples (Rodriguez-Negrette *et al.,* 2019).

Figure 4 shows the melting point of shea butter obtained from all the methods studied in this research. The melting points obtained ranged from 28 to 34 ºC. It showed a similar trend with the density of shea butter with optimum kernel using solvent extraction method (sample E) having the lowest melting point of 28 ºC and local kernel using conventional extraction method (Sample D) having the highest melting point of 34 ºC. Melting point determines the level of adulteration of a substance and it's also the point at which a substance begins to form liquid when heat is applied (Girolami,et al., 1999). Optimisation of both the shea kernel and traditional extraction method had no significant influence on the melting point as all the values fell within the African Organization for Standardization (ARSO) average value of 36 ºC, except for optimum kernel using solvent extraction method (sample E) which has melting point of 28 ºC.

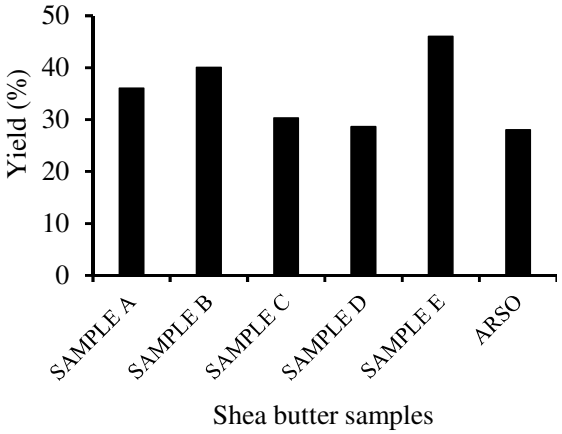

Figure 1: Yield of shea butter produced from different extraction methods

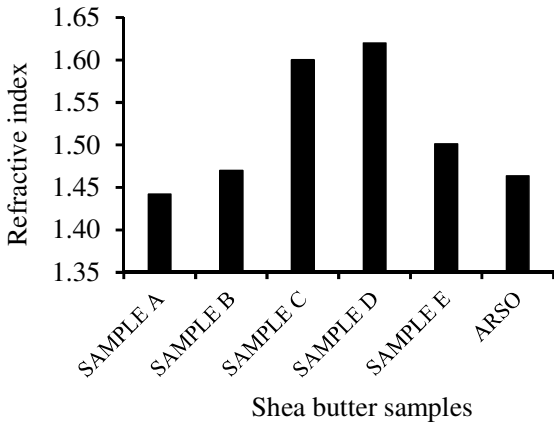

Figure 2: Refractive index of shea butter produced from different extraction methods

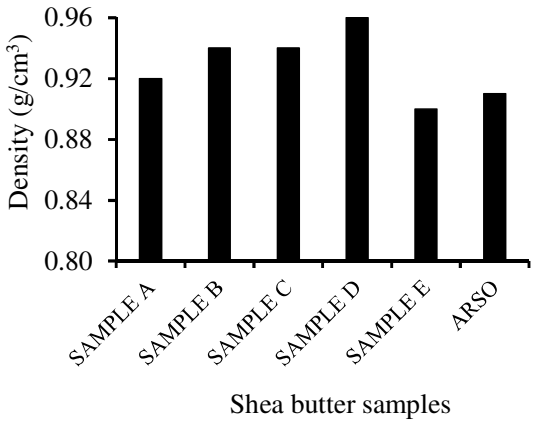

Figure 3: Density of shea butter produced from different extraction methods

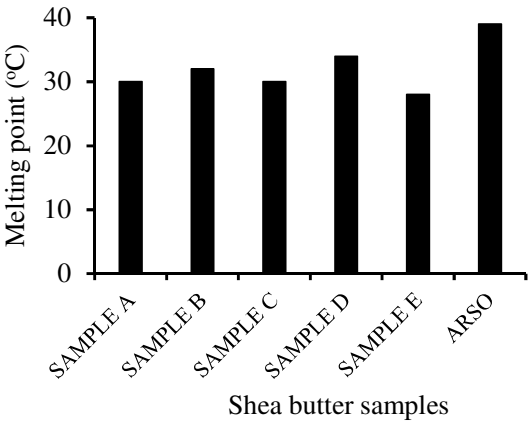

Figure 4: Melting point of shea butter produced from different extraction methods

The motivation for the comparison of the chemical properties of shea butter obtained from these methods of extraction was to analyze the effect of optimization of shea kernel, optimization of traditional extraction method and choice of extraction method on some selected chemical properties of Shea butter. Figures 5 to 8 shows the value of the chemical properties of shea butter produced from each of the methods studied. Figures 5 shows the value of the free fatty acid of shea butter produced from each of the methods. The free fatty acid of shea butter produced from all the methods investigated was found to be between 0.8936% and 4.88%. Sample D had the highest value of 4.88% while Sample A had the lowest free fatty acid of 0.8936%. Except for Sample A, all other samples of shea butter had free fatty acid values higher than the free fatty acid

requirement of ARSO (2017) of free fatty acid figure of ≤1%. The high value of free fatty acid of Sample D is consistent with the findings of Lovett (2004). This observation may be attributed to lack of control of several factors like shea nut conditioning period (SNCP), shea nut boiling duration (SNBD), shea nut drying temperature (SNDT) and other poor processing practices at the extraction stage such as roasting temperature, (RT), roasting time (Rt) and time lag between milling of kernels and kneading of paste (tLbMK) (Saba, 2019). The seemingly low value of free fatty acid of 0.8936% obtained from Sample A might be as a result of low RT of 30 ^{o}C, Rt of 60 min and low tLbMK of 6.425 h (Saba, 2019). The value of free fatty acid obtained is similar to those obtained by Obibuzor *et al.* (2014) and Ouattara *et al.* (2015). Lowering of free fatty acid is a major requirement in grade 'A' shea butter, as its high values indicates formation of peroxides and hydroperoxides (Lovett, 2013). Controlling and optimizing the processing variables from the shea kernel through to the extraction process is key to producing grade 'A' shea butter (Aculey *et al.,* 2012). Comparison of Sample D with Sample C shows that optimization of kernel only resulted in 4.7% (0.0488-0.0465)/0.0488) reduction in free fatty acid. A combination of optimization of kernel as well as optimization of traditional extraction method (Sample D and Sample A) resulted in a significantly higher reduction in free fatty acid of 82% (0.0488-0.008936)/0.0488. This indicates that optimization of shea kernel is an important criterion in achieving grade "A" shea butter.

Figure 6 shows the peroxide value of shea butter produced from each of the extraction methods. The peroxide value of the shea butter samples produced in this study varies between 1.501 and 4.2. $meqkg^{-1}$. All these values were below the African Organization for Standards value of 10 $meqkg^{-1}$ and fell within the grade 'A' shea butter threshold of 10 $meqkg^{-1}$. Optimization of the traditional method of extraction reduces the peroxide value by 18% when Sample C and Sample D were compared. The combined effect of both kernel optimization and traditional extraction method reduces the peroxide value by 64% when Sample D and Sample A are compared. The choice of extraction method also had significant influence on the peroxide value of Sample A, Sample B and Sample E, as it reduces the peroxide values by 64, 37 and 64% respectively. Sample B has the highest peroxide value possibly due to nature of the extraction method involving higher temperature, pressure and possibly longer time of exposure of the oil to oxygen before filtration (Abdul-Mumeen, et al., 2019). The peroxide values obtained in this studies is comparable with those obtained by Chukwu and Agidzi (2008), Okullo *et al.* (2014), Ouattara *et al. (*2015), Rodriguez-Negrette *et al.* (2019) and ARSO (2017) . This may be as a result of instant analysis of peroxide value as production is done implying no much exposure to oxygen and sunlight which catalyzes the production of free fatty acid and their oxidation into peroxides (Lovett, 2013).

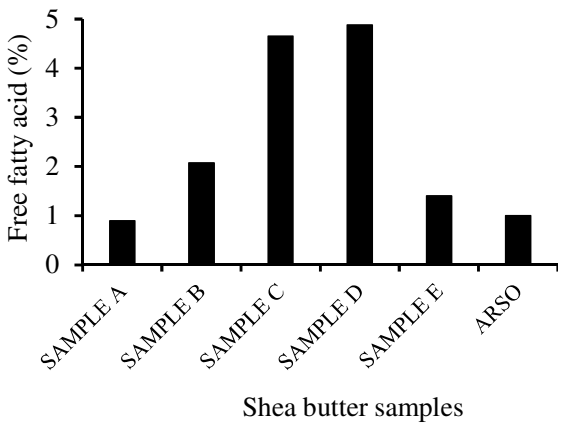

Figure 5: Free fatty acid of shea butter produced from different extraction methods

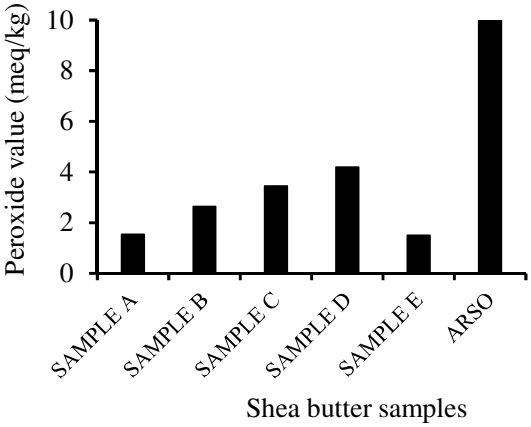

Figure 6: Peroxide value of shea butter produced from different extraction methods

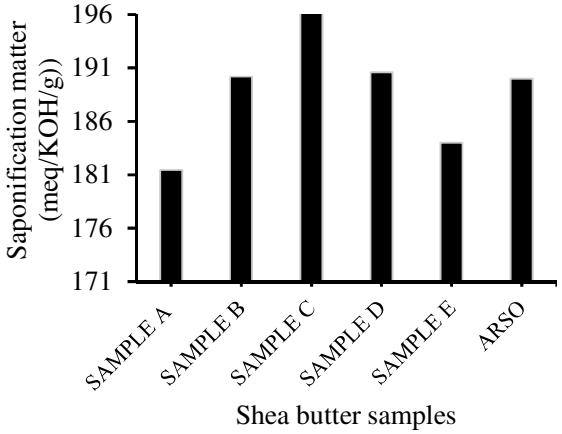

Figure 7: Saponification matter of shea butter produced from different extraction methods

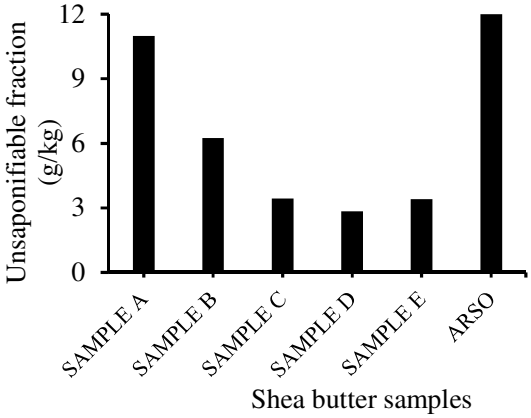

Figure 8: Unsaponifiable fraction of shea butter produced from different extraction methods

Figure 7 shows the value of the saponification matter of shea butter produced from each of the extraction methods. Saponification matter is measure of the number of milligrams of potassium hydroxide required to completely neutralize 1 g of a fatty acid. The saponification matter of the shea butter samples obtained in this study varied between 181.45 and 196.4 $meq(KOH)^{-1}g^{-1}$. Except for Sample C which was slightly above the average value of 190 meqKOH/kg for African Organization for Standardization, all other samples have saponification matter within the grade 'A' classification of ARSO (2017). The saponification matter of Sample C was found to be 196.4 meqKOH/kg, which is slightly higher than the African Organization for Standardization average value of 190 meqKOH/kg. Optimization of the traditional method of extraction alone increased the saponification matter by 3% when Sample C and sample D were compared. The combined effect of both kernel optimization and traditional extraction method reduced the saponification matter by 5%. This shows little or no significant effect on saponification matter when Sample D and Sample A were compared. The choice of extraction method had no significant influence on the saponification matter of Sample A , Sample B and Sample E as all the saponification matter of the samples (181.45 meqKOH/g, 190.2 meqKOH/g and 184.0132 meqKOH/g respectively) fell within the grade 'A' classification of shea butter provided by ARSO (2017) . Sample B had the highest saponification matter possibly due to nature of its extraction involving higher temperature and pressure giving rise to oil with higher total fatty matter and hence more saponification matter (Julius et al., 2013)**.** The saponification matter obtained in this study are comparable with those obtained by Chukwu and Agidzi (2008), Okullo *et al.* (2014), Ouattara *et al.* 2015 and comparable to values in ARSO, (2017).

Figure 8 shows the value of the unsaponifiable fraction of shea butter produced from each of the methods. The unsaponifiable fraction of shea butter produced varies from 2.84 to 10.99 $g(kg)^{-1}$and all are within the ARSO (2017) average standard of 12.0 $g(kg)^{-1}$. The low values of unsaponifiable fraction in shea butter from Sample C and Sample D maybe due to non-optimization of the shea kernels, while that of sample E may be as a result of dissolution of the bioactive components into the solvent and or evaporation of the volatile bioactive components consequent upon temperature of extraction (Ajala et al., 2016).

4. CONCLUSION

The research result shows that Grade A shea butter is obtained by the optimization of both the shea kernel processing and the subsequent shea butter extraction method. Grade 'A' shea better ($FFA < 1\%$, peroxide value $< 10\ meqkg^{-1}$ and un-saponifiable fraction $> 5g(kg)^{-1}$) can be obtained from optimally processed shea kernel (4.3 days shea nut conditioning period, 86 ºC drying temperature, 120 minutes boiling duration) using optimum traditional extraction method (30 ºC of roasting temperature, 60 min of roasting time, 300

rpm of kneading speed and 6.4 h of time lag between milling of kernel and shea paste kneading). Optimization of both the shea kernel and the processing shea butter conditions increased the yield of shea butter for traditional method, mechanical expression and solvent extraction from 28.6% to 36.05%, 40.4% and 46% respectively. Economically, these percentage increase in yield is a motivating factor for optimization of both the kernel and the process of shea butter extraction from all methods studied.

5. ACKNOWLEDGMENT

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6. CONFLICT OF INTEREST

There is no conflict of interest associated with this work.

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