Physicochemical characteristics of shea kernels and kinetics of shea butter press extraction

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Abstract

Morphological study of some non conventional oil seeds are of great interest for the development of some communities. In this investigation, shea kernels from 4 localities in Chad were studied. The sun dried shea kernels from Beinamar locality had the greatest length ($28.30 \pm 3,25$ mm), diameter (20.58 ± 2.46 mm), weight (5.56 ± 1.52 g) and moisture content (3.79 ± 0.14 %), whereas the kernels of Penie village had the least values (24.25 ± 2.54, 19.02 ± 2.34, 4.0 ± 1.01 and 3.54 ± 0.33 respectively) of the mentioned characteristics. On the other hand, the total lipid content of Penie's shea kernels was the highest (58.5%) whereas that of Bolo village was the lowest one (53.53 ± 0.40 %). The acid value of the 4 samples was extremely high (25.31 ± 0.71- 38.30 ± 0.76 mg KOH/g oil), whereas the acid value of the oven dried shea kernels produced using boiling method was low (1.2 - 2.1). The kinetics of press extraction of shea butter suggested that the extraction time is 12 min.

Keywords: shea; kernels; butter; boiling; physicochemical characterization; correlation; extraction; kinetics.

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1. Introduction

In developing countries, one of the ways of promoting development is through the exploitation of available local resources. This also helps to satisfy the needs of the increasing population and increase the income of the citizen [1].

Though several studies have been carried out on the physicochemical and functional properties of shea kernels as under exploited oilseeds elsewhere [2-4], there is very little information on the physicochemical characteristics of shea kernel in the producing countries in general and in Chad in particular. Meanwhile, Mbaiguinam *et al.,* (2007) [5] reported some morphological as well as some physicochemical characteristics of shea tree and shea fruit in the southern region of Chad. Their study was done in one production area (Mandoul region). But Palmberg (1985) [6] reported that to study different properties of certain species, sampling site should be as far as possible so as to account for the influence of some parameters such as site, climate and ecology. Therefore, this part of research was focused on the determination of the morphological and the physicochemical characterisation of sun dried shea kernels from different localities in the southern region of Chad, and to study the kinetics of press extraction of shea butter, because the annual production of Chadian shea kernels was estimated to be 1 390 247 tons, with about 40 % post harvest losses [7]. Therefore, 834 148 tons per year could be transformed to shea butter. Shea butter is used as an edible vegetable fat in many African countries. It can be utilized as a substitute or complete replacement for cocoa butter in various applications and plays an important role in traditional African medicinal practice [8].

2. Materials and methods

2. 1. Origin and characterisation of shea kernels

Sun dried shea kernels which were produced using the non boiling method described by Adoum (1996) [9] were bought from: Beinamar locality west of Mondou city, Bedjondo village 45 km east of Doba city, Penie village 35 km west of Koumra city and Bolo village 27 km east of Kelo city. These sites are located in southern region of Chad in Sudanian zone (Savannah's trees). The selection of these localities was based on two reasons; first, they present the main source of supply of sun dried shea kernels to local markets. Secondly, there is a lack of information on morphological characteristics of sun dried shea kernels and on the physicochemical properties of shea butter from these localities. Some geographical information (height above see level, longitude, latitude, rainfall and the type of soil) for each site are shown in table 1 [10-11]. For the purpose of comparisons, oven dried shea kernels produced following the boiling method as described by Lovett (2004) [12] were prepared from shea fruits.

Table 1: Some geographical information about the four localities in southern Chad

Geographical information	Locality			
	Bolo	**Beinamar**	**Bedjondo**	**Penie**
Height (m)	375	410	387	393
Longitude (east)	$16^{o}.00$	$15^{o}.25$	$17^{o}.20$	$17^{o}.05$
Latitude (north)	$9^{o}.20$	$8^{o}.40$	$8^{o}.35$	$8^{o}.50$
Rainfall (mm)	1000	1100	1150	1100
Type of soil	Reddish brown (Kaolinite, hematite)	Red soil (ferruginous)	Reddish (hydromorphic)	Brown (higher % of clay fraction)

2. 2. Preparation of oven dried shea kernels using boiling method

Ripe shea fruits from Bolo and Bedjondo villages were collected and brought to the laboratory of Applied Chemistry Faculty of Pure and Applied Sciences University of N'Djamena Chad. The fruits were depulped and each sample was divided in two equal lots, one lot was boiled (Bo) in water for one hour and the other was kept un boiled (NBo). Then all the samples were dehulled and dried in an oven (60 ± 2°C) for 72 hours.

The diameter and length of 100 sun dried shea kernels (randomly selected) from each of the four localities were determined using Vernier callipers. The mass was measured using a laboratory balance of 0.001 precision. The moisture content (%) of the kernels was determined using AFNOR, (1981) method. In this method, 5g of ground kernels was placed in a dry oven dish, and then dried in an oven at 105± 2°C until a constant mass was achieved. The experiment was repeated twice and the average value was taken.

The moisture content M_c was calculated on a wet basis and expressed in gram per 100 grams of initial sample (equation 1).

$$M_c = \frac{m_1 - m_2}{m_1} * 100 \% \qquad ....(1)$$

Where m_1 is the mass in gram of wet sample and m_2 is the mass of dry sample.

2. 3. Shea butter extraction

Solvent extraction method was used to determine the total lipid content and the later was used to determine the extraction yield of press extracted shea butter.

2. 3. 1. Determination of the total lipid content

The total lipid content represents the total fats in the oil seed. The method used was described in IUPAC (1979) [13]. This method is based on the differential solubility of lipid in the organic solvent (n-hexane or petroleum ether). The extraction was done in a soxhlet apparatus for 8 hours, after which the solvent was evaporated and the fat was dried in the oven. The detailed procedure is as follows:

20 g of ground shea kernels was introduced in a dry cellulose thimble. The thimble was put in the extraction flask and then 250 ml of petroleum ether was added. The extraction flask was then fitted onto the soxhlet and the extraction was conducted for 8 hours. After this period the flask was removed and the solvent was evaporated from the mixture using rotary evaporator. The extracted oil was then dried in the oven at 105 ± 2°C for one hour, then cooled in a crucible for 30 minutes and weighed. The experiment was repeated twice and the average mass was taken.

The lipid content (L_c) as a percentage of dry material was expressed by equation 2.

$$L_c = \frac{m_1 - m_0}{m} * 100 * \frac{100}{100 - M_c} \quad ....(2)$$

Where m_1 is the mass of extraction flask containing the oil after oven drying together with beads, m_0 is the mass of the empty flask with beads inside, m is the mass of initial ground kernels and M_c is the moisture content of the sample.

2. 3. 2. Press extraction

For the press extraction of shea butter a manual hydraulic press of model "MF-FSEA-2005" constructed in the Department of Technology of the Faculty of Pure and Applied Sciences (University of N'djamena) was used. This press was constructed based on the press (model KCT 2000 AIRE) described by Tchiegang *et al.* (2003) [14]. The press (Figure 1) is composed of a horizontal metal arm (1) joint with a vertical screw rod (2). The later is passed through a node (3) which is fixed to a frame (4). The screw rod ends on a pressing disc at its lip (5). The disc passes through a perforated external cylinder (6) which is welded to a fixed plate (7). An internal perforated cylinder and the oil recipient (8) are complementary parts.
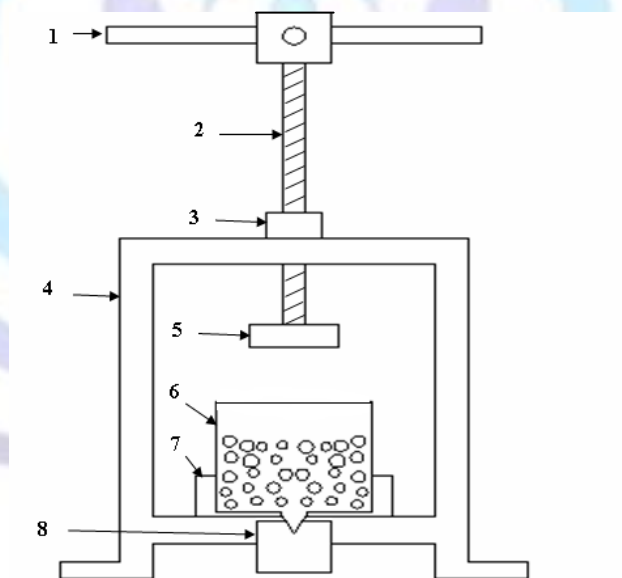
Figure 1: Sketch of a manual vertical screw press of model "MF-FSEA-2005": Horizontal metal arm (1), vertical screw rod (2), node (3), frame (4), pressing disc (5), perforated external cylinder (6), fixed plate (7) and oil recipient (8).

The support of the press was inclined at 20° to the horizontal to permit the flow of the shea butter at its melting point (40 - 50°C) and this was achieved by heating (about 60°C) the fixed plate before pressing. In each experiment, the shea kernels were ground and then put in a dry 100 ml beaker and covered tightly with aluminium sheet. The beaker and its contents was immersed into boiling water for 30 minutes (steam cooking) while ensuring that there was no direct contact of the water with the ground sample. A known quantity of the cooked sample was put in a cotton bag and placed in the internal cylinder in the press. Then the pressing disc was screwed down slowly inside the cylinder by rotating the horizontal metal arm up to a certain level determined by a point in the vertical screw rode beyond which the system will collapse. Oil which escapes through the perforations was collected in recipient, weighed and kept for further analysis. Using the barometer and hydraulic press,

moving the same distance mentioned latter, the pressure measured was found to vary between 17 and 18 M Pa. The dimensions of this press are figured in table 2.

Table 2: Dimensions of different parts of the manual hydraulic press of model "MF-FSEA-2005".

The part	Dimensions (cm)
Horizontal bar	Length = 42; diameter = 2
Vertical screw rod	Length = 44; diameter = 3
Frame	Length = 33; width =30; thickness = 6
Pressing disk	Diameter = 5
External perforated cylinder	Internal diameter = 6.5; height = 10.5; thickness = 0.8
Internal perforated cylinder	Internal diameter 5.2; height = 7.5; thickness = 0.5
Fixed plate	Length; 15.6; width: 10; height: 5

The quantity of the oil recovered from the ground shea kernels (Y_{ield}) was calculated as a ratio of the mass of the extracted oil to the mass of dry sample (equation 3), while the effective yield (E_{ield}) was calculated from the ratio of the amount of extracted oil to the total amount of oil available (L_c) in the sample (equation 4).

$$Y_{ield} = \frac{M_0 \ (g)}{M_d \ (g)} * 100 \quad \text{............... (3)}$$

$$E_{ield} = \frac{Y_{ield}}{L_c} * 100 \quad \text{................ (4)}$$

Where M_o is the mass of oil recovered in gram, M_d is the mass of dry sample; L_c is the total lipid content.

For the kinetics study, raw sun dried shea kernels were used. The kernels were ground and sieved using sieves of 2 - 3 mm square mesh, steamed cooked for 30 min in a water bath and then pressed. Each experiment was replicated twice and the average values were taken. Extractions were done for 3, 6, 12, 15 and 18 min. The yield of shea butter from the four samples, as a function of extraction time was then plotted.

2. 4. Shea butter characterisation

2. 4. 1. Acid value

The acid value is defined as the milligrams of potassium hydroxide required to neutralise the free carboxyl groups in one gram of oil or fat. The method used for the determination of acid value was described in (AFNOR, 1981) [15]. The determination of acid value is based on the dissolution of a known quantity of fat in a mixture of 95% ethanol and diethyl ether 1:1 (v/v), followed by the titration of FFAs with ethanolic solution of KOH in the presence of 1% phenolphthalein solution in 95% ethanol as indicator.

The procedure was as follows: 1 g of fat was weighed in 250 ml conical flask, then 10 ml of the mixture of 95% ethanol and diethyl ether was added to the beaker with shaking, then three drops of the phenolphthalein solution were added. The mixture was titrated with ethanolic solution of KOH (0.5N), and then a blank test was carried out. Each experiment was repeated twice and the average values were calculated. The acid value (A_v) was expressed by equation 5

$$A_V = \frac{56.1 N (V - V_0)}{m} \quad \text{.............. (5)}$$

Where V is volume of KOH solution used in the titration of test portion, V_0 is the volume of the KOH solution used in the blank test, N is the exact normality of KOH solution, *m* is the mass of the test sample.

Free Fatty Acid (FFA) is the equivalent of oleic acid content of the fat. It is about one-half (0.504) the acid value. Both characteristics indicate the quality of the oil with respect to acidity, and have been used to follow the course of fat or oil refining [16].

$$R - COOH + KOH \rightarrow R - COOK + H_2O$$
Fatty acid   Soap
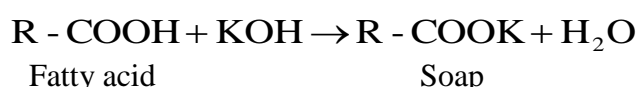
2. 4. 2. Peroxide value

Peroxide value is used to determine the degree of oxidation of oil and fats. It is defined as the content of reactive oxygen in terms of milligrams of fat (1 millimole = 2 ml equivalent) or ml equivalent of oxygen per kilogram of fat. The primary oxidation products of oils and fats are hydroperoxides. The amount of hydroperoxides is quantitatively measured by determining the amount of iodine liberated by its reaction with KI. The technique used is phenolphthalein method described in AFNOR, (1981) [15].

The procedure is detailed as follows: In each experiment, 2 g of the fat sample was weighed into 250 ml conical flask and dissolved by adding 10 ml of chloroform with shaking. Then 15 ml of acetic acid and 1 ml of saturated solution of KI were added successively to the flask, then it was stoppered immediately, shaken for one minute and kept in the dark for 5 minutes after which 75 ml distilled water was added and the content of the flask was titrated with sodium thiosulphate solution (0.01N) in the presence of starch solution as indicator. Each experiment was repeated twice. At the end a blank test was carried out and the peroxide value (P_v) was expressed by equation 6.

$$P_V = \frac{1000N(V - V_0)}{m} \quad \text{.............. (6)}$$

Where N is the normality of sodiumthiosulphate solution, V is the volume of sodiumthiosulphate used for the test with the fat, V_0 is the volume of sodiumthiosulphate used for the blank test and m is the mass in gram of the test sample.

3. Results and discussion

3. 1. Some physicochemical characteristics of shea kernels and shea butter

The determination of some characteristics of sun dried shea kernels was done with the objective to making available information on this commodity which is produced in different areas in the southern region of Chad. The results are presented in table 3

Table 3: Some physicochemical characteristics of raw sun dried shea kernels and shea butter from four localities in the southern region of Chad

Characteristic	Locality			
	Bolo	Beinamar	Bedjondo	Penie
Length (mm)	$26.1^b \pm 3.6$	$28.3^c \pm 3.3$	$25.4^b \pm 2.8$	$24.3^a \pm 2.5$
Diameter (mm)	$19.3^a \pm 2.4$	$20.6^c \pm 2.5$	$19.2^a \pm 2.0$	$19.0^b \pm 2.3$
Weight (g)	$4.80^b \pm 1.7$	$5.6^c \pm 1.5$	$4.5^b \pm 1.0$	$4.0^a \pm 1.0$
Moisture content	$3.68^a \pm 0.21$	$3.79^a \pm 0.14$	$3.69^a \pm 0.27$	$3.54^a \pm 0.33$
Total lipid content (%)	$53.53^a \pm 0.40$	$55.10^b \pm 0.73$	$56.59^c \pm 0.44$	$58.45^d \pm 0.23$
Acid value	$38.30^c \pm 0.76$	$25.30^a \pm 0.71$	$33.66^b \pm 0.21$	$25.81^a \pm 0.78$

Means in the same row with different superscripts are significantly different (p < 0.05)

The mean and the standard deviations for the physicochemical properties of shea kernels and shea butter in table 3 were calculated from the randomly selected 100 sun dried shea kernels from each locality. The moisture content for the four samples is lower than the optimum one (10-15%) required for optimum storage which was reported by EL Warraki, (1995) [17]. Also, from table 3 it is observed that the acid value of the shea kernel samples from the 4 localities is extremely higher than that permitted for edible oils and fats [18]. These higher values of acidity are an indication of oil degradation in the shea kernels during the process of sun drying.

Also it is observed that the sun dried shea kernels from Beinamar locality has the highest length, diameter, weight and moisture content, whereas the kernels of Penie village has the least values of the mentioned characteristics. It is observed that, sun dried shea kernels obtained from the highest locality (410 m) characterised by larger diameter, length and weight, while that from the lowest locality (365 m) characterised by small diameter, length and weight. The Pearson correlation coefficients between the diameter, length and the weight of shea kernels and the height, longitude, latitude and rain fall of the sites, showed that, the diameter, length and the weight of the kernel were positively correlated to the height and negatively to the longitude of the sampling site. The low correlation appeared in table 4 is due to the small variation in the height.

Table 4: Correlation coefficients between some geographical parameters and physical characteristics of sun dried shea kernels obtained from 4 localities in southern Chad.

	Height (m)	Longitude (east)	Latitude (north)	Rainfall (mm)	Diameter	Length	Weight
Height (m)	1,00	**-0,25**	**-0,84**	**0,65**	**0,17**	**0,18**	**0,14**
Longitude (east)	**-0,25**	1,00	**-0,23**	**0,41**	**-0,23**	**-0,41**	**-0,38**
Latitude (north)	**-0,84**	**-0,23**	1,00	**-0,96**	-0,06	-0,01	0,01
Rainfall (mm)	**0,65**	**0,41**	**-0,96**	1,00	0,00	-0,06	-0,07
Diameter	**0,17**	**-0,23**	-0,06	0,00	1,00	**0,60**	**0,79**
Length	**0,18**	**-0,41**	-0,01	-0,06	**0,60**	1,00	**0,83**
Weight	**0,14**	**-0,38**	0,01	-0,07	**0,79**	**0,83**	1,00

On the other hand, based on the total lipid content, shea kernels from Penie had the highest value (58.5%). This value is close to that reported by Tano Debrah and Ohta (1994) in Ghana (59%). The variation in the total lipid content from one locality to another is usually attributed to the diversity in natural soil and composition of their derivation or other man-made cumulative effects, in addition to the possible change in environmental and geological conditions from one region to another [19].

Moisture and lipid content of shea kernel, together with the acid and peroxide values of extracted shea butter from Bolo and Bedjondou villages are reported in table 5.

Table 5: Some physicochemical characteristics of shea kernels and of shea butter obtained using boiling and oven drying method.

	Origin of the sample			
Parameter	Bolo **NBo**	Bolo **Bo**	Bedjondou **NBo**	Bedjondou **Bo**
Moisture content (%)	$7.50^a \pm 0.17$	$8.60^b \pm 0.22$	$4.22^a \pm 0.37$	$6.12^b \pm 0.32$
Lipid content (%)	$45.96^a \pm 1.06$	$53.14^b \pm 0.87$	$40.57^a \pm 0.84$	$45.14^b \pm 1.21$
Acid value	$2.10^a \pm 0.49$	$1.40^a \pm 0.60$	$1.40^a \pm 0.67$	$1.21^a \pm 0.50$
Peroxide value	$2.13^a \pm 0.16$	$7.38^b \pm 0.21$	$2.63^a \pm 0.18$	$5.13^b \pm 0.14$

*Means in the same row for the same locality with different superscripts are significantly different ($p < 0.05$); **NBo**: Un boiled; **Bo**: Boiled*

From table 5 it is noticed that boiling of shea nuts in water for 60 min significantly increased the moisture content of shea kernels, increased the extraction yield, increased the peroxide value and decreased the acid value of shea butter. The observed increase in the moisture content of shea kernels could be attributed to the absorption of water by the kernel during the boiling process, while the increase of the peroxide value could be relatively attributed to the oxidation of shea butter during boiling process. These results agree well with those reported by Womeni *et al.* (2006) [20]. The increase in extraction yield as a result of boiling of shea nut could be attributed to denature of proteins. This action reduces the affinity of oil for solid surfaces and therefore oil could flow out easily as confirmed by Norris (1982) [21].

The decrease in acid value by boiling process might be due to the deactivation of lipase enzymes that naturally exist in oilseeds. Lipase catalyses the hydrolysis of oil in oilseeds and liberates free fatty acids. Ladurelle (1984) [22] reported that lipases in oilseeds can be deactivated at 80°C after an hour or at 100°C after 10 min.

Generally, it could be reported that the acid value of shea butter that was produced using the boiling method and oven drying of shea kernels, is low compared with that of shea butter produced using non boiling method and sun drying. In addition, boiling significantly improved the extraction yield of shea butter.

3. 2. Kinetics of press extraction of shea butter

The kinetic curve was found to follow equation 7 (exponential rise to maximum).

$$y = a(1 - e^{-bt}) \quad .... (7)$$

Where **y** is the yield (g/g), **t** extraction time (min), **a** and **b** are constants.

From figure 2, it is observed that the extraction rate of shea butter from the sun dried shea kernels seemed to be very fast in the first 3 minutes, and then it had a moderate increment in the next 9 minutes. After 12 minutes the rate was almost constant. This behaviour was supported by the results of y_t/y_{t-3} appeared over figure 2 in which y_9/y_{12} gave 95% oil yield. Also it is noticed that Penie sun dried shea kernels gave the best yield of shea butter, followed by Beinamar sample. In fact, from

the kinetic plots, the yield was related to the total lipid content. These results permitted us to select the sample of Penie locality for the optimisation of press extraction process.
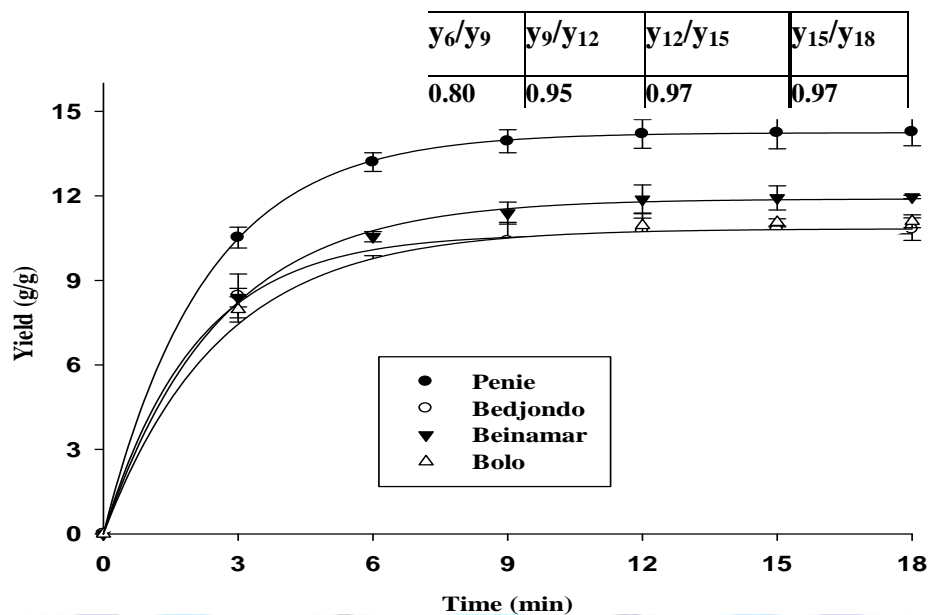
y_6/y_9	y_9/y_{12}	y_{12}/y_{15}	y_{15}/y_{18}
0.80	0.95	0.97	0.97

Figure 2: Variation of yield and press extraction time of shea butter

CONCLUSION

This study permitted us to conclude that the sun dried shea kernels from Beinamar locality has the biggest size: length (28.30 ± 3,25 mm), diameter (20.58 ± 2.46 mm), weight (5.56 ± 1.52 g) and moisture content (3.79 ± 0.14 %), whereas the kernels from Penie village are the smallest in size: (24.25 ± 2.54, 19.02 ± 2.34, 4. 0 ± 1.01 and 3.54 ± 0.33 respectively). On the other hand, based on the total lipid content, Penie's shea kernels have the highest (58.5%) and Bolo village showed the lowest (53.53 ± 0.40 %).

The acid value of the 4 samples of sun dried shea kernels is extremely high (25.31 ± 0.71- 38.30 ± 0.76), whereas the acid value of the oven dried shea kernels produced using boiling method is low (1.2 - 2.1). The kinetics of press extraction of shea butter suggested that the equilibrium extraction time is 12 min.

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