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Original Article

Mechanical extraction of shea butter: Optimisation and characterisation studies with comparison to other methods of extraction

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Abstract

Shea butter (SB) production by mechanical extraction (ME) was optimised by box-behnken (BB) experimental design of response surface methodology (RSM). The process parameters studied (with their ranges) were sample weight (100 – 200 g), temperature (60 – 120°C) and duration of applied pressure (10 – 30 min) to optimise the yield of SB. The characteristics of the SB were determined using standard methods and Fourier transform infrared spectroscopy (FTIR). A comparative study of the SB from ME with other methods of extraction was also performed. The optimum 37% (w/w) yield of SB was obtained from 150 g sample of shea kernel at 90°C and 20 min. The R^2 (0.9957) obtained from analysis of variance showed that quadratic model of BB fitted the experimental data well. The characteristics of the SB from ME showed non-compromising quality, with a yield greater than that of traditional method but lower than with solvent method. This study showed that the extraction methods affect both yield and quality of SB.

Keywords: shea butter, mechanical extraction, box-behnken, FTIR

1. Introduction

Extraction of vegetable oils is nowadays done by chemical method using solvents such as n-hexane. This gives a high yield of vegetable oil with short processing time and low energy consumption. However, due to negative environmental impacts and potential health risks from using a solvent in the extraction of vegetable oil, particularly in industrial scale, the method is regarded hazardous (Alenyorega, Hussein, & Adon go, 2015). Ikya, Umenger, and Iorbee (2013) further substan-tiate this claim, when a solvent was used to extract SB with the relatively high 47.5% yield, but with compromised charac-teristics appearance, texture, odour, and general acceptability.

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Hence, an alternative method of extraction is needed to obtain both high yield of vegetable oil and the required quality for human consumption and other uses.

One of the available alternative methods is mechanical extraction (ME), which is the most popular globally; it is safe and simple to use. The vegetable oil from ME has been reported to be chemical free and rich in protein. This makes ME advantageous over the more efficient solvent extraction. However, the ME is relatively inefficient, leaving about 8 to 14% of the available oil in the cake (Singh & Bargale, 2000). Several efforts have been made by different researchers to improve the efficiency of ME by optimising the process parameters such as applied pressure, pressing temperature, and moisture content of the kernels. For instance, Olaniyan and Oje (2007a) studied the use of ME to extract SB with a 4^3 factorial experimental design and obtained a maximum yield of 35.1%. Further studies by Olaniyan and Oje (2011) using a model equation with Mechanical Extraction Rig (MER) for SB gave a maximum yield of 35.39%. The yields obtained were very low compared to 60% SB present in its kernel (Axtell, Kocken, & Sandhu, 1993a). Therefore, a more robust optimisation approach, such as response surface methodology (RSM), can be employed for optimising ME to improve the yield of SB without compromising the quality.

RSM is a mathematical tool that employs experimental design with the ultimate goal of evaluating operating parameters for any process, using minimum numbers of experimental runs. It is widely used as a technique to optimise, develop, or improve processes. The main advantage of RSM, apart from the minimum number of experimental runs, is that it generates enough information for statistical acceptability of the results (Akinoso, Aboaba, & Olajide, 2011). The use of RSM for ME of SB is expected to improve the yield and maintain the quality.

Shea butter is a product of shea fruits. Shea fruit includes green epicarp, mesocarp (pulp), and a hard endocarp that ultimately encloses the shea kernel (nut) known as the embryo (Olaniyan & Oje, 2007). The shea kernel contains about sixty percent (60%) shea butter (Axtell, Kocken, & Sandhu, 1993b), which is a major raw material for several purposes. SB can be used as a lubricant (material for greasing, engine oil, and baking industries) and as insect repellent and protection against *Simulium infection* (Ajala, Aberuagba, Olaniyan, & Onifade, 2016). Shea butter is also known to contain a relatively large amount of unsaponifiable content, between 4 and 11%. The unsaponifiable compounds include triterpenes, tocopherol, phenols, and sterols, which are anti-inflammatory with antioxidant properties (Honfo, Akissoe, Linnemann, Soumanou, & Van Boekel, 2014). Therefore, SB can be used for medicinal purposes as a sedative in the treatment of sprains, dislocations, minor aches, and pains; unguent for skin; and as an antimicrobial agent promoting the rapid healing of wounds (Ajala *et al.*, 2016). SB consists of more than 90% triglycerides, comprising more than 50% unsaturated fatty acids (oleic, stearic, linoleic and palmitic fatty acids) that are prone to oxidation (Honfo *et al.*, 2014). The presence of high amounts of unsaturated fatty acids in the SB causes oxidative degradation of the butter, whether made by traditional extraction or any other extraction procedure that involves boiling or improper processing or storage. This leads to inconsistent quality and limited shelf-life (Lovett, 2004; Masters, Yidana, & Lovett, 2004). The oxidative degradation of SB degrades the edibility of the butter, produces sensory and chemical changes, and reduces the nutritional values (Nahm, Juliani, & Simon, 2012). This has motivated a lot of interest in SB extraction and characterisation. Hence there is a need to compare alternative extraction processes, as this choice affects the yield and quality of the SB.

This study, therefore, optimised SB extraction by ME using RSM. Comparative study of extracted SB from mechanical (SBM), SB from traditional (SBT), SB from solvent (SBS) and SB from enzymatic (SBE) extraction methods was performed, assessing yield, physicochemical properties, and functional groups.

2. Materials and Methods

2.1 Materials

Shea kernels (Sk) were purchased from Ilorin South, Kwara State, Nigeria. The Mechanical Extraction Rig (MER) that was used comprised two sections. The first being the Hydraulic Press Machine (HPM) with 10 MPa capacity, model number M500 - 50 KN (Testometric Co Ltd., UK). The second section is an MER, shown in Plate 1 and Figure 1, which was developed by Olaniyan and Oje (2007a) and was used to extract shea butter from shea kernels.

Plate 1. Mechanical expression rig.

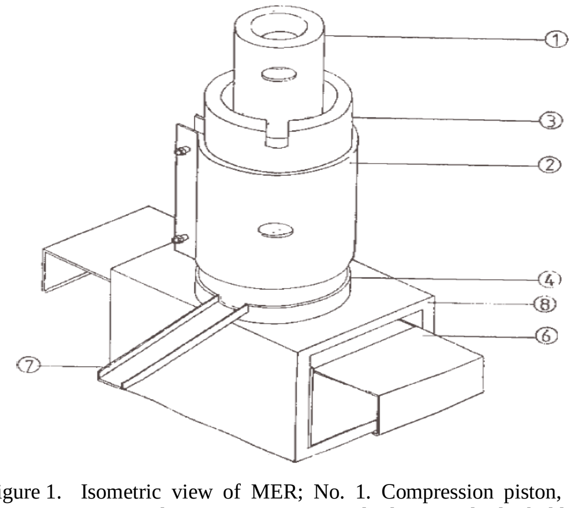


Figure 1. Isometric view of MER; No. 1. Compression piston, 2. Heating device, 3. Press cage cylinder, 4. Cylinder holder, 5. Drainage channels, 6. Oil collecting pan, 7. Oil trough, 8. Supporting platform. Source: Olaniyan and Oje (2007a).

2.2 Methods

2.2.1 Box-Behnken (BB) design with quadratic model in response surface methodology (RSM)

The quadratic model of BB is an empirical model that involves a second-order polynomial, which was employed in this study. The model was fit to experimental data to predict the yield from independent process parameters, and was used to maximise the yield by determining the optimal operating conditions for SB extraction. The model is as shown in Equation 1:

$$Y = \beta_0 + \sum_{i=1}^{n} \beta_i x_i + \sum_{i=1}^{n} \beta_{ii} x_i^2 + \sum_{i=1}^{n-1} \sum_{j=i+1}^{n} \beta_{ij} x_i x_j \quad 1$$

Where Y represents the predicted response; β_0 is the offset term; β_i is the linear coefficients, and β_{ij} is the interaction coefficients, while x_j is the independent variable process parameter.

The BB design of RSM incorporated into the Design Expert software (version 8) was used to design the experiments. The empirical results from designed experiments were provided to the software for analysis, which generated predictive models. The model was further subjected to optimisation using the software, with the yield of SB as the objective function to be maximised, and the process parameters (factors in the experimental design) as constrains within prescribed limits. Statistical analysis was also carried out through RSM using the Design Expert. The regression coefficients, significant model terms in the regression model, and optimal factors level for optimum yield of SB, were obtained.

1) Experimental design for mechanical extraction

The BB design of Design Expert was used in an optimisation study of SB extraction. The three process parameters investigated were sample weight of Sk (100 – 200 g), temperature (60 – 120°C) and duration of applied pressure (10 - 30 min). The ranges chosen for these parameters were obtained from a preliminary investigation, and are shown in Table 1a to form seventeen experimental runs matrix shown in Table 1b.

2) Experimental procedure for mechanical extraction

The Sk were ground into small particles and sieved using a mesh to obtain <2.06 mm particle size. The sieved sample Sk (100, 150 or 200 g) was weighed and poured into press cage cylinder. The sample in the cylinder was heated with the aid of a temperature-controlled heater band at desired temperature (60, 90 or 120°C) for the duration of 30 min. Thereafter, the sample Sk was compressed by the compression piston using the HPM at a pressure of 8 MPa for a chosen period of time (10, 20 or 30 min). The SB extracted was collected in the output pan and was weighed to determine the yield using Equation 2. The compression piston was lifted, the cylinder unscrewed and the residual cake was removed.

Table 1. Experimental factor levels and ANOVA results

(a) Factor levels for optimisation of SBM

Independent variables			Range and level		
Factor	Name	Units	-1	0	+1
A	Sample weight	g	100	150	200
B	Temperature	ºC	60	90	120
C	Applied pressure time	min	10	20	30

(b) Yield of SBM by experimental run

Run	Sample weight (g)	Temperature (ºC)	Applied pressure time (min)	SBM yield (%w/w)
1	100.00	60.00	20.00	27.00
2	200.00	60.00	20.00	28.01
3	100.00	120.00	20.00	29.10
4	200.00	120.00	20.00	30.00
5	100.00	90.00	10.00	33.98
6	200.00	90.00	10.00	34.01
7	100.00	90.00	30.00	34.02
8	200.00	90.00	30.00	34.00
9	150.00	60.00	10.00	27.01
10	150.00	120.00	10.00	29.99
11	150.00	60.00	30.00	28.01
12	150.00	120.00	30.00	30.00
13	150.00	90.00	20.00	37.01
14	150.00	90.00	20.00	37.02
15	150.00	90.00	20.00	37.00
16	150.00	90.00	20.00	36.99
17	150.00	90.00	20.00	37.00

(c) ANOVA for response surface quadratic model of SBM

Source of variation	Coefficient estimate	Sum of squares	Df	Mean squares	F value	P value	
Model	37.01	250.79	9	27.87	180.98	< 0.0001	Sig.
A	0.32	0.79	1	0.79	5.16	0.0574*	
B	1.15	10.63	1	10.63	69.01	< 0.0001	
C	0.0075	0.00045	1	0.00045	0.0029	0.9584*	
AB	0.055	0.012	1	0.012	0.079	0.7873*	
AC	-0.065	0.017	1	0.017	0.11	0.7501*	
BC	-0.11	0.044	1	0.044	0.29	0.6091*	
A2	-1.71	12.32	1	12.32	80.01	< 0.0001	
B2	-7.03	207.82	1	207.82	1349.7	< 0.0001	
C2	-1.28	6.90	1	6.90	44.84	0.0003	
Residual	-	1.08	7	0.15			
Pure Error	-	0.015	4	0.00368			
Cor Total	-	251.87	16				
CV = 1.22%	R^2 = 0.9957	Adj R^2 = 0.9902		Pred R^2 = 0.9324			

*Non Significant

$$\%yield = \frac{\text{weight of oil extracted}}{\text{weight of shea kernel used}} \times 100 \quad 2$$

2.2.2 Characterisation of SBM

1) Physical and chemical properties

The physical and chemical properties of the SB were determined using standard methods of analysis (AOAC, 1998). The properties determined were saponification value, free fatty acid, acid value, iodine value, viscosity, peroxide values, pH and melting point. The Rudolph Research Analytical (RRA) DDM 2911 Automatic Digital Density Meter was used to determine the relative density (Ajala *et al.*, 2016).

2) FTIR

The Bruker ALPHA FT-IR spectrometer was used for the FTIR analysis of the extracted SB, in the range of 4000–400 cm^{-1} (Ajala *et al.*, 2016).

3. Results and Discussion

3.1 Statistical analysis of the mechanical extraction of shea butter

The %yield of SB from ME in the experiments by the BB design of RSM is shown in Table 1b. The ANOVA for fitting the second-order response surface model by least squares is shown in Table 1c. From the table, the p <0.0001 of the model demonstrates a very high significance to predicting the response and its suitability. The high F value of the model (180.98) and a very low p-value (<0.0001) show that the model is highly significant. The significance of all the coefficients is established by p – values shown in Table 1c.

The second order terms of the three process parameters considered were statistically significant, each of them having $p < 0.0005$. Also, the low CV (1.22%) shows that the results of the model are reliable. The quality of the model is shown by $R^2 = 0.9957$, which indicates that 99% of the experimental variation was explained by the predictive model. The high Adj. R^2 (0.9902) also supports the significance of the model, while the high value of predicted R^2 (0.9324) indicates reasonable precision of the fitted model.

3.2 Effect of process variables on the yield of SB from ME

The effect of sample weight of Sk and temperature on the %yield of SB is shown in Figure 2a. This figure shows that the sample weight of 150 g at 90ºC gave the maximum yield of 37% (w/w) SB. It was observed that as the sample weight and temperature deviate from the centre, the yield of SB decreases. Figure 2b shows a plot of sample weight of Sk and duration of applied pressure against yield of SB. From the plot, the sample weight of 150 g and applied pressure of 8 MPa for 20 min gave the maximum 37% (w/w) yield of SB and deviation of the two parameters from the centre causes a decrease in the yield. The interaction between the sample weight and duration of applied pressure was significant to the yield. The interaction between duration of applied pressure and temperature is also shown in Figure 2c. The figure reveals that the centre point in this plot at a fixed sample weight of

Table 2. Percentage yield of SB obtained from different methods of extraction.

Extraction Method	%Yield	Source
Traditional	(a) 27.2	Akingbala *et al.* (2006)
	(b) 28	Coulibaly *et al.* (2009)
	(c) 34.1	Ikya *et al.* (2013)
Solvent	(a) 47.5	Ikya *et al.* (2013)
	(b) 53.77	Nkouam *et al.* (2007)
	(c) 66.90	Ajala *et al.* (2015b)
Supercritical CO_2	39.57	Nkouam *et al.* (2007)
Enzymatic	42.95	Ajala *et al.* (2015a)
Mechanical	(a) 24.0	Akingbala *et al.* (2006)
	(b) 35.90	Olaniyan and Oje (2011)
	(c) 37	This study

150 g gives the maximum 36% yield of SB. The interactions show similar trends in Figs. 2b and 2c. The results show that the maximum 37% (w/w) yield of SB was obtained with sample weight 150 g, temperature 90ºC and duration of applied pressure 20 min, which is similar to the report of Olaniyan and Oje (2011), where heating temperature, applied pressure and loading rate of 82.24°C, 9.69 MPa and 2.50 mm min^{-1}, respectively, gave the maximum 35.39% yield of SB in a 4^3 factorial experimental design. However, Mohagir, Bup, Abi, Kamga, and Kapseu (2015) obtained a better yield of 45.7% SB, when RSM with Doehlert experimental design was used for the optimisation of kernel preparation conditions before press extraction of SB. However, the roasting temperature (160 - 225ºC) used was rather too high and may have affected the quality of the SB produced.

3.3 Optimisation of mechanical extraction of shea butter

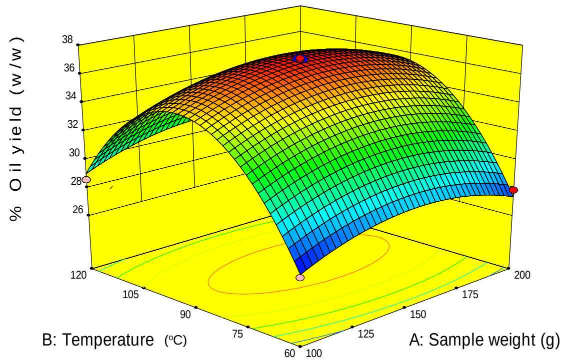
The coefficients of the response surface model for ME of SB as provided by Equation 3 were evaluated. The optimal variables that gave maximum percentage yield of SB for ME were obtained by using the regression fit, in the Design Expert software.

$$Oil\ Yield\ \left(\%\frac{w}{w}\right) = -51.68250 + 0.21086 \times A + 1.44502 \times B + 0.56395 \times C + 3.66667E-005 \times A \times B - 1.30000E-004 \times A \times C - 3.50000E-004 \times B \times C - 6.84200E-004 \times A^2 - 7.80611E-003 \times B^2 - 0.012805 \times C^2 \quad (3)$$

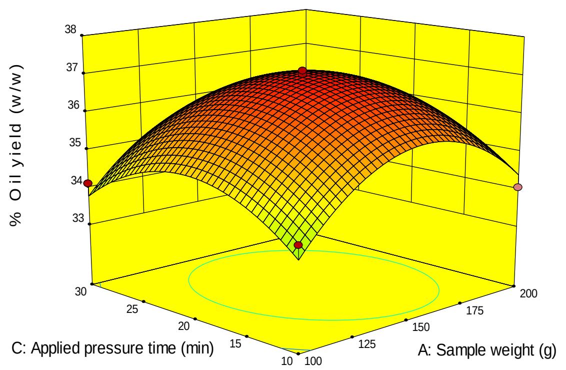
The optimum %yield of SB (37%, w/w of Sk) was found at sample weight 154.67 g, temperature 92.49ºC and duration of applied pressure 19.97 min. Experiments were carried out to validate the model predictions of the parameters, and the yield of SB obtained was 36.87% (w/w). Thus, the verification experiments confirmed validity of the predictive model. The error between the predicted and the validated result was 0.353, which is low, and within the limits (0–5%) of allowable error, and this result indicates good reproducibility of the experiments. This shows that the model predictions are in good agreement with the experimental results.

Table 3. Physico-chemical properties of SBT, SBS, SBM, and SBE.

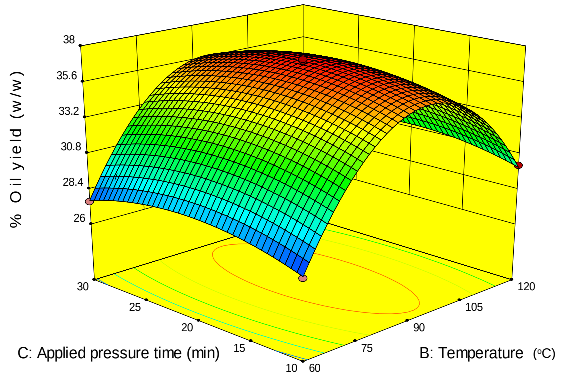
Properties	SBT	SBS	SBE	SBM
Relative Density	0.908	0.851	0.931	0.912
Kinematic Viscosity (mm^2s^{-1})	30.68	44.84	19.72	26.57
Melting point (°C)	33.0	40.5	30.0	37.5
Free fatty acid (%)	9.0	23.89	6.85	8.10
Acid value (mgKOH/g oil)	21.85	48.63	13.21	18.39
Peroxide value (meq O_2/kg)	9.80	11.00	12.10	13.80
Iodine value (gI_2/100 g oil)	61.90	70.30	67.28	58.50
Saponification (mgKOH/g oil)	208.00	202.9	193.90	180.20
pH value	6.09	5.02	5.53	5.38
Sources	Ajala *et al.*, 2015b		Ajala *et al.*, 2015a	This Study



(a) Weight and temperature (Time fixed at 20 min)



(b) Weight and time (Temperature fixed at 90°C)



(c) Temperature and time (Weight fixed at 150 g)

Figure 2. Effect of weight, temperature and time on %yield of SBM in 3D plots

This study has clearly demonstrated the applicability and reliability of RSM for the optimisation of extraction variables in SB extraction using ME method.

3.4 Comparative analysis of SB from ME with other methods of extraction

3.4.1 Percentage yield of SBM

Table 2 shows the yields of SB obtained from different methods of extraction. The table shows that the maximum yield obtainable for SB was from solvent extraction, as reported by Ajala *et al.* (2016) (66.90% (w/w)) and Ikya *et al.* (2013) (47.5%). Nkouam, Kapseu, Barth, Dirand, and Tchatchueng (2007) corroborate the fact that solvent extraction of SB gave higher yield (53.77%) than even supercritical CO_2 methods (39.57%, w/w). The maximum yield for mechanical extraction of SB was 35.90% (Olaniyan & Oje, 2011) and that of enzymatic extraction was 42.9% (Ajala, Aberuagba, Olaniyan, & Onifade, 2017). From this study, 37% SB (w/w) yield was obtained, which clearly shows that ME could not yield over 40% SB. However, the yield of ME (37% SB) was more than those of traditional extraction method reported by Akingbala, Falade, Adebesi, Baccus-Taylor, and Lambert (2006), and Coulibaly, Ouédraogo, and Niculescu (2009), as 27 and 28% SB (w/w), respectively.

3.4.2 Physico-chemical properties

Table 3 shows the physicochemical properties of SBM as compared with other samples of SB from other extraction methods.

1) Relative density and kinematic viscosity (mPa.s)

The relative density (RD) of SBM is 0.912 (Table 3); an indication that the RD of the SBM is relatively high compared to SBT (0.908) and SBS (0.851) but lower than SBE (0.931) as shown in Table 3. This may be as a result of fine particles and impurities present in the SBM after gravitational settling. The RD of SBM falls between 0.870 and 0.917 as reported by Olaniyan and Oje (2007a), which is also similar to the find of Hee (2011).

The kinematic viscosity (Kv, mm^2 s^{-1}) was 26.57 for SBM (Table 3). The Kv of SBM is the lowest among SBT and SBS

Table 4. Evaluation of the FT-IR spectrum of SBM.

Identification of Peaks by Region	Band wave number (cm^{-1})	Assigned functional group
Region of functional groups		
Region of hydrogen's stretching	2918.74, 2852.03	Symmetric and asymmetric stretching vibration of the aliphatic CH_2 group
Region of double bond's stretching	1741.30	Ester carbonyl functional group of the triglycerides (C=O stretch)
Region of other bonds deformations and bendings		
i.	1461.01	Bending vibrations of the CH_2 and CH_3 aliphatic groups
ii.	1375.90	Bending vibration of the CH_2 groups
Finger print region		
i.	1246.25, 1168.66	Stretching vibration of the C-O ester groups
ii.	720.02	Overlapping of the CH_2 rocking vibration and the out-of-plane vibration of cisdisubstitutted olefins

as shown in Table 3, and lower than 80 obtained by Olaniyan and Oje (2007a), but greater than that of SBE (Table 3). The difference in these results may be due to a temperature differences in the extraction process (Olaniyan & Oje, 2007b). The SBE is the least viscous among the samples, which may be due to the presence of water in the extraction process.

2) Melting point

The melting point (mp) of SBM is 37.5°C which falls within the 20 - 45°C range reported by Honfo, Akissoe, Linnemann, Soumanou, and Van Boekel (2014). This value is similar to the 37.0°C obtained by Olaniyan and Oje (2007b) and close to the human body temperature, hence suitable for different purposes such as a base for ointment (Ajala *et al.*, 2016). Comparatively, as shown in Table 3, the mp of SBE is the lowest, followed by SBT. This might be due to the presence of water and/or impurities in the extraction process of SBE. The mp of SBM is a little lower than that of SBS. The lower mp might be due to the hydrolysis of triacylglycerols and oxidation of unsaturated fatty acids, as a result of heating for 30 min (Gunstone, 2004; O'Brien, 2009).

The mp of SBM is also closer to that of cocoa butter (32 - 35°C), therefore SBM can be recommended as a substitute for the more expensive cocoa butter in the production of confectioneries (Akingbala *et al.*, 2006).

3) Acid value and free fatty acid

The acid value (Av) of SBM is 18.39 mgKOH/g, which falls within the range 0 – 21.2 mgKOH/g reported by F. G. Honfo *et al.* (2014) and Nkouam *et al.* (2007). However, the SBM is lower than the 47.7 mgKOH/g reported by Olaniyan and Oje (2007b). In comparison with previous studies shown in Table 3, acid value of SBM is the lowest among SBT and SBS, but slightly higher than that of SBE. This indicates that SBM and SBE are in good condition and edible with long shelf life; and suitable for industrial uses such as paint making, cosmetics and food applications, compared to the other two (Nitièma-Yefanova, Poupaert, Mig nolet, Nébié, & Bonzi-Coulibaly, 2012). This is because Av of vegetable seeds increases with storage duration depending on the conditions (Hee, 2011; Honfo *et al.*, 2014).

The FFA value of SBM is 8.10% which is the lowest among those of SBT and SBS, but higher than that of SBE, as shown in Table 3. This shows that SBM is the best among SBT and SBS. However, SB with FFA>1% is not suitable for biodiesel production, and not good for cosmetic and food uses due to irritation of tongue and throat (Ajala *et al.*, 2016), but rather it can be used as a lubricant, because of the inherent lubricating properties. The FFA of SB from all the methods of extraction analysed was >1%; this may be due to the hydrolysis of triglycerides caused by the lipolytic activity of the fruit lipase and microorganisms (Nitièma-Yefanova *et al.*, 2012).

4) Peroxide value

Peroxide value (Pv) is a measure of the extent to which rancidity reactions occur during storage and measures of oxidation of unsaturated fats and oils. In cosmetics and food industries, the recommended value of Pv for any vegetable oil is <10 mEq O_2/kg (F. G. Honfo *et al.*, 2014).

The Pv of SBM is 13.80 mEq O_2/kg which slightly exceeds the recommended level (<10). However, it is within the range from 0.5 to 29.5 mEq O_2/kg reported by Dandjou ma, Adjia, Kameni, and Tchiegang (2009) and Njoku, Eneh, Ononogbu, and Adikwu (2000). Table 3 shows that SBM has the highest Pv, but lower than 44.9 mEq O_2/kg observed by Olaniyan and Oje (2007b). This might be due to the processing conditions (Hee, 2011), as the activation of lipases and tocopherol of natural antioxidants occurs with heating for 30 min at 90°C (Akingbala *et al.*, 2006).

5) Iodine value

The iodine value (Iv) for SBM (58.50 g I_2/100 g oil) and the other cases are shown in Table 3. From the table, the Iv of SBM is the lowest among the cases, and also lower than 82.1 g I_2/100 g oil obtained by Olaniyan and Oje (2007b), but higher than the 50.2 g I_2/100 g oil observed by Akingbala *et al.* (2006). The differences in the Iv may be due to processing conditions or the extraction approach. These results showed that SBM is less saturated, with a lower degree of saponification and longer shelf-life than the others (Ajala *et al.*, 2016).

6) Saponification value

The saponification value (Sv) for SBM is 180.2 mgKOH/g oil (Table 3) and it is within the acceptable range of 188 - 190.5 mgKOH/g oil (Akingbala *et al.*, 2006). Table 3 also shows the Sv for SBT, SBS, and SBE, and it was seen that SBM has the lowest Sv which is also lower than 261.3

mgKOH/g oil obtained by Olaniyan and Oje (2007a). The reason may be due to the 90ºC press temperature with 30 min heating time, as the temperature is inversely proportional to Sv (Olaniyan & Oje, 2007a).

7) pH value

The pH of SBM is 5.38 (Table 3). The pH values show that SBM is acidic, though less acidic than SBS but more acidic than SBE (Table 3). The acidity in the SB is due to the unsaturated fatty acids (Nwabanne, 2012). Generally, the physicochemical properties of SBM observed in this study may be affected by the experimental procedures, as well as quality and pre-treatment of the kernels before crushing, as was reported by Coulibaly *et al.* (2009).

3.4.3 FTIR analysis

Figure 3 shows the FT-IR spectra of the various samples of the SB, but no subjective differences were noticed among the spectra. Table 4 gives the chemical compositions shown by the spectra, using relevant information available in the literature (Pandurangan, Murugesan, & Gajivaradhan, 2014; Poiana *et al.*, 2012; Vlachos *et al.*, 2006). In the hydrogen's stretching region, the signal was observed at 2918.74 and 2852.03 for SBM; an indication of symmetric and asymmetric stretching vibrations of the aliphatic CH_2 group. A similar signal was observed for the other samples. In the second region of double bond's stretching, the band at 1741.30 is seen; an indication of ester carbonyl functional group of the triglycerides. Of course, this region is found in almost all vegetable oils and significantly indicates oils with high saturated fatty acids contents (Poiana *et al.*, 2012). Comparatively, the signal was observed at 1707.13 for SBS, which signifies a difference in that region. This indicates that SBS has a free fatty acids shoulder, which may confirm higher FFA in SBS than in SBM.

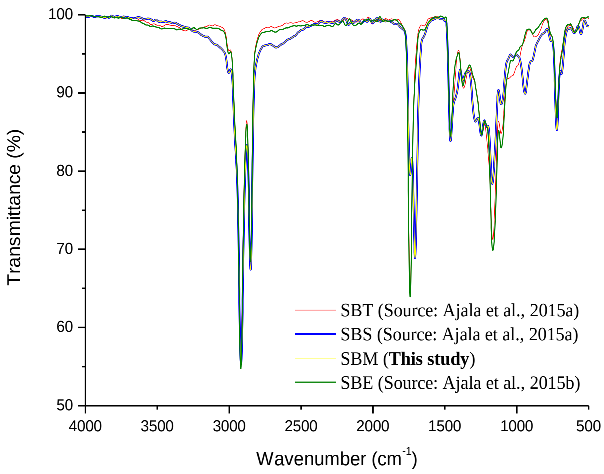


Figure 3. FTIR spectra of the different samples of SB

The third region of deformation and bending in the functional group showed bands at 1461.01 and 1375.90 for SBM. The peak at 1461.01 showed bending vibrations of the CH_2 and CH_3 aliphatic groups, while 1375.90 peaks showed bending vibrations of the CH_2.

In the fingerprint region, the bands occurred at 1246.25, 1168.66 cm^{-1} for SBM which signaled the stretching vibration of the C-O ester groups and gave the important information about the sample. Only SBS has signal at 941.99, which characterises C=O bond vibrations.

The SBM has similar functional groups and fingerprint regions like other samples considered; an indication that the method of extraction may not have a significant effect on the functional groups. In all the results shown, C=C was absent; this corroborates the iodine and peroxide values obtained. The low iodine values (<100mg/I_2) show that the SBM and the other samples are saturated, matching the low peroxide values that show deterioration of the samples; an indication that the shelf-life is longer than cases with unsaturated C=C bonds.

4. Conclusions

The optimum ME yield was 37% SB at the optimal process parameters of Sk sample weight (154.67 g), temperature (92.49ºC) and duration of applied pressure (19.97 min). This study concluded that RSM was not able to significantly improve the yield of SB from ME. The 37% yield obtained in this work is nearly the same value of 35% SB (w/w) reported in literature. In comparison with the four methods of extraction, solvent extraction gave the highest yield of SB. However, ME is more environmentally friendly, easy to use, and more suitable for SB extraction than the other methods. The physicochemical properties of SB obtained by ME showed superior quality among the SB extraction methods tested. The FTIR analysis showed no significant differences between the different methods of extraction. The study concludes that ME gave a higher 37% yield of SB than the traditional extraction method with 28% yield, while the solvent (66.9% SB) and enzymatic (43% SB) extraction methods had even higher yields. This study revealed that the method of extracting SB can significantly affect yield and characteristic quality of the butter.

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