Optimization of solvent extraction of shea butter (*Vitellaria paradoxa*) using response surface methodology and its characterization

E. O. Ajala¹ · F. Aberuagba² · A. M. Olaniyan³ · K. R. Onifade²

Revised: 30 August 2015 / Accepted: 14 September 2015 / Published online: 24 September 2015
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Abstract Shea butter (SB) was extracted from its kernel by using n-hexane as solvent in an optimization study. This was to determine the optima operating variables that would give optimum yield of SB and to study the effect of solvent on the physico-chemical properties and chemical composition of SB extracted using n-hexane. A Box-behnken response surface methodology (RSM) was used for the optimization study while statistical analysis using ANOVA was used to test the significance of the variables for the process. The variables considered for this study were: sample weight (g), solvent volume (ml) and extraction time (min). The physico-chemical properties of SB extracted were determined using standard methods and Fourier Transform Infrared Spectroscopy (FTIR) for the chemical composition. The results of RSM analysis showed that the three variables investigated have significant effect ($p < 0.05$) on the %yield of SB, with $R^2 = 0.8989$ which showed good fitness of a second-order model. Based on this model, optima operating variables for the extraction process were established as: sample weight of 30.04 g, solvent volume of 346.04 ml and extraction time of 40 min, which gave 66.90 % yield of SB. Furthermore, the result of the physico-chemical properties obtained for the shea butter extracted using traditional method (SBT) showed that it is a more suitable raw material for food, biodiesel production,

cosmetics, medicinal and pharmaceutical purposes than shea butter extracted using solvent extraction method (SBS). Fourier Transform Infrared Spectroscopy (FTIR) results obtained for the two samples were similar to what was obtainable from other vegetable oil.

Keywords Shea butter · Extraction · Characterization · Optimization · Vegetable oil · RSM

Introduction

Shea tree (ST) is abundantly found in the wide belt of savannah (Masters et al. 2004) including West African countries like Nigeria, and further east in Uganda (Goreja 2004). It belongs to Sapotaceae family and the German botanist Carl Gaertner was the first to name it as *Vitellaria paradoxa* C.F. Gaertn (Nahm 2011) and was renamed as *Butyrospermum parkii* (Maranz and Wiesman 2003). Shea fruit like other fruits has three layers: green epicarp (the outer part); a fleshy mesocarp (pulp) and a relatively hard endocarp (shell) containing embryo known as shea kernel (Olaniyan and Oje 2007a). The shea kernel contains great percentage of butter, which had been used over the years for different purposes such as: biodiesel production, soap processing, human consumption, cosmetic, medicinal, metal cutting fluids and a substitute for cocoa butter in the chocolate and confectionary industries (Olaniyan and Oje 2007b). SB has several other applications for medicinal use (Abdul-Mumeen et al. 2013b) such as: sedative for treatment of sprains; dislocations; minor aches and pains; and as an unguent for skin. It can also be used as antimicrobial agent for the promotion of rapid healing of wounds and as lubricant. Shea butter is also suitable as: material for greasing bread tins in baking industries; insect repellent, protection against *Simulium* infection (Goreja 2004; Olaniyan

✉ E. O. Ajala
olawaleola01@yahoo.com

¹ Department of Chemical Engineering, University of Ilorin, Ilorin, Nigeria

² Department of Chemical Engineering, Federal University of Technology, Minna, Nigeria

³ Department of Agricultural and Bioresources Engineering, Federal University Oye-Ekiti, Oye-Ekiti, Nigeria

and Oje 2007a). The shea nut cake after the extraction can be used to make animal feeds (Abdul-Mumeen et al. 2013a) and as a substitute for maize in the diets of growing gilts. With these numerous application of SB and its cake; it can be said to be a source of employment and means of livelihood for rural people in the region where it is largely found.

Traditional technique has been the major method of extraction for shea butter in most places where it is largely produced, particularly, Nigeria; but the method has been reported to be too cumbersome, tedious, time consuming, energy sapping, environmentally unfriendly and grossly inefficient (Olaniyan and Oje 2007a) with average efficiency of about 28 % (Coulibaly et al. 2009). This process comprises of five basic steps: thermal treatment of the seeds; grinding; extraction by boiling; oil recovery and drying. The process procedure and conditions had been reported to have great effect on the oil yield, extraction efficiency and quality of oil recovered; as it causes damage to the components due to application of higher temperature. This resulted in the degradation of flavour and aroma of the extracted shea butter, making it to have smoky smell (Febrianto and Yang 2011). Moreover, due to the chemical breakdown, the oil extracted contains some quantity of free fatty acid and water with unpalatable flavour (Ibrahim and Onwualu 2005). Among many other challenges with this traditional method is the technological limits of the process, that did not enable a full separation of liquid and solid phases, with no adequate monitoring of the extraction conditions (Alenyorega et al. 2015). Therefore, there is the need to find alternative method of extraction that can overcome these challenges. Although, solvent extraction have been reported in the literature for shea butter (Ikya et al. 2013), but there is paucity of information on the optimization study, to establish optima variables for the process. These necessitated this study.

RSM is an effective and important tool for statistical analysis to find the optimal conditions for different complex processes, which has been applied in the optimization of multiple variables with a minimum number of experiments (Stroescu et al. 2013) to generate sufficient information for a statistically acceptable result. RSM has been successfully used for the optimization of biodiesel production (Boonmee et al. 2010; Sara et al. 2012), and it has been applied in industries, biological, clinical, social, food, physical and engineering sciences (Akinoso et al. 2011), however, little or no information is available on its application for optimization of SB using solvent. Hence, this work was expected to provide additional information on the use of RSM for optimization study of solvent extraction of SB.

This study therefore, investigated the effect of process conditions: quantity of the seed (g); extraction time (min) and volume of the solvent (ml) on the percentage yield of SB in an optimization study using Box–behnken (BB) design. This was used to predict the response for the experimental design of solvent extraction process of SB. The physico-chemical

properties and FTIR of SBS was compared with SBT, to determine the effect of solvent extraction procedure on the quality of the SBS.

Materials and methods

Materials

Shea seed and SBT were purchased from Ilorin South, Kwara State, Nigeria. The Rudolph Research Analytical (RRA) DDM 2911 Automatic Digital Density Meter was used to determine the relative density. The Bruker ALPHA FT-IR spectrometer was used for the FTIR analysis, in the range of 4000–400 cm^{-1} . The solvents used were of analytical grade.

Methods

Shea kernel preparation

The seed were dehulled, winnowed to remove the outer covering and sundried until constant weight was observed. This was to eliminate the moisture content present in the shea kernel. The kernels were ground using mortar and pestle to rupture the nut; and to reduce the particle size. Mesh size of 2.06 mm was used to screen the kernel to the desired particle size.

Design of experiment and statistical analysis

The experimental design of three process conditions with three levels for each, gave total number of 17 runs of experiment based on BB design. The Statistical analysis was carried out through RSM using design expert software (version 8.06 Stat-Ease Inc., Minneapolis, MN). The process conditions and their ranges considered were: the sample weight of shea kernel (A, 30–50 g), solvent volume (B, 200–350 ml) and extraction time (C, 40–80 min). The particle size of shea kernel (2.06 mm), solvent type (n-hexane) and temperature (68 °C) were kept constant for the study. The regression coefficient and significant model term to fit the regression model and to determine the optimal factors level for maximum %yield of SBB were obtained. An empirical model was employed for a better understanding of the correlations between the factors and the yield (response) by using a quadratic model of a second-order polynomial as shown in Eq. 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; β_{ii} and β_{ij} are

the interaction coefficients; x_i and x_j are the independent variables.

Solvent extraction procedure

The solvent extraction of SB was carried out using 500 ml Soxhlet extractor. The required solvent volume of n-hexane was poured into a round bottom flask. The prepared sample weight of shea kernel was placed in the thimble and was inserted in the centre of the extractor. The temperature of the extraction was maintained at 68 °C. When the solvent was boiling, the vapour rose through the vertical tube into the condenser at the top. The liquid condensate dripped into the filter paper thimble in the centre, which contained the shea kernel sample to be extracted. The extract seeped through the pores of the thimble and filled the siphon tube, where it refluxed back into the round bottom flask. The process continued until the desired duration of time was attained for the extraction process; the content in the flask was poured into a rotary evaporator to remove n-hexane in the mixture. The SB obtained was placed in water bath until constant weight was recorded. The percentage yield was calculated as the ratio of the weight of oil recovered to the weight of the shea kernel before extraction. It was mathematically expressed as Eq. 2:

$$O_Y = \frac{W_{OE}}{W_{US}} \times 100\% \quad (2)$$

Where; O_Y = oil yield (%); W_{OE} = weight of oil expressed (g) and W_{US} = weight of crushed shea kernel before extraction (g).

Optimization and validation

The optimization process was carried out using BB RSM of the design expert. The empirical percentage yield of the designed experiment was analyzed using the design expert and generated a combination of actual and predicted results. The results were further subjected to optimization analysis using the design expert to obtain percentage yield of SB as the objective function and the three process conditions as constraint with the prescribed limit. The optimization result obtained from the analysis was validated by repeating the experiment in the laboratory.

Characterization of shea butter

The physical and chemical properties of SBT and SBS were characterized. The relative density was measured using the RRA. The saponification was analysed by

standard methods of AOAC (1998) and acid value (ISO 660 2003). The fatty acid component was analyzed using the technique reported by Chopra and Kanwar (1998). Iodine value was determined using the method of Akpan et al. (2006). The method of Nielsen (2003) was used to determine the peroxide value and viscosity. The pH was determined using the method reported by Warra et al. (2012). The SBT and SBS samples were further analyzed using the FTIR.

Results and discussion

The outcome of the experimental design in percentage yield of SB was presented in Table 1. The maximum yield of 66.3 % (Exp. run 5) and minimum %yield of 50.8 % (Exp. run 17) were obtained. These showed that the process conditions were relevant to the shea butter yield in a solvent extraction process, as different levels of process conditions gave different %yield of SB. The experimental and predicted results of the optimization study are shown in Fig. 1. The coefficient of determination of R^2 (0.8989) and the adjusted R^2 (0.7689) are shown in Table 2. The values are high, which showed a high significance of the model. The Adeq. Precision of 8.671, which indicate the signal to noise ratio of the model is greater than 4. This showed that the model can be used to navigate the design space.

Table 1 Experimental design and %yield of shea butter using BB of RSM

Run	Sample weight (g)	Solvent volume (ml)	Extraction time (min)	Shea butter yield (% w/w)
1	30	200	60	54.7
2	50	200	60	52.6
3	30	350	60	64.3
4	50	350	60	62.8
5	30	275	40	66.3
6	50	275	40	55.8
7	30	275	80	71.6
8	50	275	80	62.8
9	40	200	40	50.8
10	40	350	40	53.8
11	40	200	80	57.8
12	40	350	80	58.3
13	40	275	60	49.5
14	40	275	60	52.3
15	40	275	60	50.9
16	40	275	60	51.5
17	40	275	60	50.5

Fig 1 Predicted and experimental results of solvent extraction of shea butter

Model parameters

Linear parameters

The single effect of each of the linear terms are significant ($p < 0.05$) (Table 2). This implied that sample weight (X_1 , g), solvent volume (X_2 , ml) and extraction

time (X_3 , min) have individual effect on the percentage yield of shea butter.

Quadratic parameters

The quadratic terms shown in Table 2 indicate that both sample weight (X_1^2 , g²) and extraction time (X_3^2 , min²) have

Table 2 ANOVA for response (oil yield (% w/w) surface quadratic model

Model term	Coefficient estimate	Sum of Squares	df	Mean Square	F Value	p-value	
Intercept	50.94	617.95	9	68.66	6.91	0.0092	Sig.
X_1	−2.86	65.55	1	65.55	6.60	0.0370	
X_2	2.88	66.41	1	66.41	6.69	0.0361	
X_3	3.02	72.90	1	72.90	7.34	0.0302	
X_1X_2	0.15	0.090	1	0.090	9.064E-003	0.9268*	
X_1X_3	0.43	0.72	1	0.72	0.073	0.7951*	
X_2X_3	−0.56	1.27	1	1.27	0.13	0.7316*	
X_1^2	8.35	293.48	1	293.48	29.56	0.0010	
X_2^2	−0.69	2.00	1	2.00	0.20	0.6673*	
X_3^2	4.84	98.48	1	98.48	9.92	0.0162	
Residual	—	69.51	7	9.93			
Pure Error	—	4.43	4	1.11			
Cor Total	—	687.46	16				
R-Squared	0.8989						
Adj R-Squared	0.7689						
Pred R-Squared	−0.5246						
Std. Dev.	3.15						
Adeq Precision	8.671						

* Non-significant effect ($P > 0.05$)

significant ($p < 0.05$) effect on the percentage yield of shea butter. On the other hand, solvent volume (X_2^3 , ml) has no-significant ($p > 0.05$) effect on the percentage yield of shea butter.

Interaction parameters

The interaction terms of the variables did not shown any significant effect ($p > 0.05$) on the percentage yield of shea butter (Table 2).

Effect of variables on percentage yield of shea butter

Effect of sample weight (X_1 , g)

The effect of shea kernel quantity on shea butter extraction is as shown in Fig. 2. The figure showed that as the sample weight of shea kernel increases, the percentage yield of shea butter decreases. It was observed that at 30 g of sample weight of shea kernel, maximum yield of 61.9 % shea butter was recovered and as the weight was increased to 40 g, the yield decreases to 45 %. Further increase in the weight to 50 g resulted to an increase in the percentage yield of SB to 53 %. These results indicate that the quantity of the shea kernel has effect on the percentage yield of shea butter. When the effects of sample weight and solvent volume were considered simultaneously as shown in Fig. 2, it was noted that at 30 g of shea kernel and 200 ml of solvent volume, the percentage yield was 60 % and when shea kernel quantity was increased to 50 g alongside increase in solvent volume to 350 ml, the corresponding percentage yield remain constant (60 %).

Similar observation was made in Fig. 3 where sample weight and extraction time interaction was considered, the percentage yield of SB remain constant. These results showed that the interaction of sample weight with the other two variables considered in this study did not have effect on the percentage yield of shea butter.

Effect of solvent volume (X_2 , ml)

The effect of solvent volume on percentage yield of shea butter from its kernel is shown in Figs. 2 and 4. These results showed that when solvent volume increases, the percentage yield increases. It was noted that at 200 ml of solvent volume (n-hexane), maximum yield of SB was achieved as 52.5 %. Further increase in solvent volume to 350 ml, the percentage yield increased to 67 %. Similar result was shown in Fig. 4 where solvent volume was plotted along with extraction time. It was observed that the interaction of solvent volume at 200 ml with extraction time of 80 min gave yield of 66 %, and when 350 ml and 40 min were considered (Fig. 4), the yield was also 66 %. Similar observation was made in Fig. 2 where solvent volume was plotted along with sample weight. These results showed that the interaction of solvent volume with the two other variables considered would not have effect on the percentage yield of SB.

Effect of extraction time (X_3 , min)

The effect of extraction time on percentage yield of shea butter from its kernel is shown in Figs. 3 and 4. These figures showed that when extraction time increases, the percentage

Fig 2 Effect of shea kernel sample weight, solvent volume and extraction time of 40 min on %yield of shea butter

Fig 3 Effect of shea kernel sample weight, extraction time and solvent volume of 350 ml on %yield of shea butter

yield of SB would increase accordingly. From Fig. 3, it was observed that at extraction time of 40 min, maximum SB yield of 62 % was obtained and when the extraction time was increased to 80 min, the percentage yield of SB increased to 72.5 %. Similar result was observed in Fig. 4 when extraction time was plotted along with solvent volume.

Optimization and model validation

Table 3 showed the result of the optimization as compared with the experimental result. The optimum

process conditions that gave optimum yield of 66.90 % SB as model predicted were: shea kernel sample weight of 30.04 g, solvent volume of 346.04 ml and extraction time of 40 min. The experiment was carried out using the optima conditions aforementioned to validate the model prediction. The optimum yield of 66.47 % SB was obtained, and the error between the predicted and the validated result was 0.43 %. This result showed that the model prediction is in good agreement with the experimental result. The optimization solution is thus expressed in Eq. 3.

Fig 4 Effect of extraction time, solvent volume and shea kernel sample weight of 30 g on %yield of shea butter

Table 3 Optimization and validation results

	Experimental		Predicted
	Max	Mini	Optimum
Sample weight (g)	50	40	30.02
Solvent volume (ml)	350	200	350
Extraction time (min)	80	40	40.00
Maximum oil yield (%)	71.6	49.5	66.96

Final Equation of the Optimization in Terms of coded Factors:

$$\begin{aligned}
 \text{Oil Yield} \left(\% \frac{w}{w} \right) = & 50.94 - 2.8625 \times A + 2.88125 \times B \\
 & + 3.01875 \times C + 0.15 \times A \times B \\
 & + 0.425 \times A \times C - 0.5625 \times B \\
 & \times C + 8.34875 \times A^2 - 0.68875 \\
 & \times B^2 + 4.83625 \times C^2 \quad (3)
 \end{aligned}$$

Characterisation of shea butter

The physico-chemical properties of SBT and SBS are shown in Table 4. The relative density for SBT was 0.908, and SBS was 0.851. The difference between these results is due to the impurity and presence of water molecules in SBT (Honfo et al. 2014). The kinematic viscosity was 30.68 and 44.84 mm²s⁻¹ for SBT and SBS, respectively. Olaniyan and Oje (2007a) reported that the more viscous oil is, the better its lubricating properties, therefore SBS has higher lubricating property than SBT. The melting points were 33 and 40.5 °C for SBT and SBS, respectively. These results indicates that SBT may be more suitable as a base for ointment and medicines than SBS, since the closer the melting point of shea butter to body temperature, the better its suitability for ointment and medicines (Honfo et al. 2014). The free fatty acid (FFA) was 9 and

23.89 % for SBT and SBS, respectively. These showed that FFA of SBS is higher than SBT, and high FFA is not suitable for cosmetic and food uses because it produces irritation on the tongue and in the throat (Honfo et al. 2014). The FFA > 1 % is also not suitable for biodiesel production (Ajala et al. 2015). Therefore, SBT may be more suitable for different purposes than SBS. Similarly, the acid values (mgKOH/g oil) of the samples were 21.85 (SBT) and 48.63 (SBS). The higher acid value showed the extent of decomposition of the glycerides in the butter by lipase or heat and light. It is also an indication for the condition and edibility of the oil. Therefore, SBT may be reported to be preferred to SBS, since it has lower acid value. The iodine value is 21.43 g I₂/100 g oil for SBT and 13.19 g I₂/100 g for SBS. These results showed that SBT is more saturated, with higher degree of unsaponification and shorter shelf-life than SBS (Honfo et al. 2014), because the FFA and iodine value is the measure of the degree of unsaturation of vegetable oil (Syed et al. 2012). The changes in these properties can therefore be used in monitoring deterioration of SB (Okullo et al. 2010). For this study, the peroxide values obtained were 29.5 and 44.01 (mg/100 g) for SBT and SBS, respectively. The recommended value for cosmetic and food uses is less than 10 meq O₂/kg (Honfo et al. 2014). These values showed that SBT is closer to the recommended value for cosmetic and food industries, hence suitable than SBS. The saponification values obtained in this study are 167.4 and 202.9 (mgKOH/g oil) for SBT and SBS, respectively. The pH of SBT and SBS are 6.09 and 5.02, respectively. The pH values showed that SBS is more acidic than SBT. This indicates that unsaturated fatty acid is present in SBS than SBT (Nwabanne 2012).

The FT-IR spectra of SBT and SBS were obtained, and by visual examination of the spectra, it was difficult to notice any difference between the two samples. Table 5 was therefore used to evaluate the peaks for differences in the two samples by using series of relevant data available on vegetable oil in the literatures (Pandurangan et al. 2014; Poiana et al. 2012; Vlachos et al. 2006). The FT-IR spectra of SB under study have few bonds with difference, both in intensities and forms. Based on these absorbance observed at different wavenumber, it was categorized into two major regions of functional and finger print. The functional region was further divided into three: bonds of hydrogen's stretching, double bond's stretching, and other bonds deformations and bendings, in the range of 4000–400 cm⁻¹.

In the hydrogen's stretching region, the following peaks were seen: 2920.42; 2852.71 (SBT) and 2917.82; 2851.18 (SBS). These bonds signal the presence of symmetric and asymmetric stretching vibration of the aliphatic CH₂ group. Thus, there were no notable differences in the signal registered at this frequency. In the second spectral region of double bond's stretching, frequency of 1741.02 and 1741.23 cm⁻¹ occurred, which indicates ester carbonyl functional group of

Table 4 Physico-chemical Properties of SBT and SBS

Properties	SBT	SBS
Relative density	0.908	0.851
Kinematic viscosity (mm ² s ⁻¹) at 40 °C	30.68	44.84
Melting point (°C)	33.0	40.5
Free fatty acid (%)	9.0	23.89
Peroxide value (mg/100 g)	29.5	44.01
Acid value (mgKOH/g oil)	21.85	48.63
Iodine value (gI ₂ /100 g oil)	21.43	13.19
Saponification (mgKOH/g oil)	167.4	202.9
pH value	6.09	5.02

Table 5 Evaluation of the FT-IR spectrum for SBT and SBS

Identification of peaks by region	Wavenumber (cm ⁻¹)		Assigned functional group
	SBT	SBS	
1. Region of functional groups			
1a. Region of hydrogen's stretching			
1	2920.42, 2852.71	2917.82, 2851.18	Symmetric and asymmetric stretching vibration of the aliphatic CH ₂ group
1b. Region of double bond's stretching			
1	1741.02	1741.23	Ester carbonyl functional group of the triglycerides
2	—	1707.13	Free fatty acids shoulder
1c. Region of other bonds deformations and bendings			
1	1459.31	1460.44	Bending vibrations of the CH ₂ and CH ₃ aliphatic groups
2	1369.87	1375.75	Bending vibration of the CH ₂ groups
2. Finger print region			
1	1242.62, 1166.95	1248.50, 1170.32	Stretching vibration of the C-O ester groups
2		941.99	C=O bonds
3	720.41	721	Overlapping of the CH ₂ rocking vibration and the out-of-plane vibration of cisdisubstituted olefins

the triglycerides is present. These regions are found in almost all the vegetable oils and significantly characterize the oils with high saturated fatty acids content (Poiana et al. 2012), but for SBT, peak were observed at wavenumber 1707.13 cm⁻¹, which signify a different in that region for shea butter extracted using two different methods. This peaks means it has free fatty acids shoulder. This confirmed why FFA of SBS is higher than that of SBT. The third region of deformation and bending in functional group showed bonds of 1459.31 and 1460.44 as well as 1369.87 and 1475.75 cm⁻¹ for SBT and SBS, respectively. The peaks at 1459.31 and 1460.44 cm⁻¹ indicates bending vibrations of the CH₂ and CH₃ aliphatic groups while 1369.87 and 1475.75 cm⁻¹ peaks showed bending vibration of the CH₂. In the finger print region, the bond that brought the important information on the butter occurred at 1242.62, 1166.95, and 1248.50, 1170.32 cm⁻¹ for SBT and SBS, respectively. These bonds signal the stretching vibration of the C-O ester groups. The bond recorded at 941.99 cm⁻¹ is characteristic to vibration of C=O. These results revealed that the absorbance at this wavenumber was observed for SBS only and was not seen in SBT.

The results obtained in this study correspond with the peaks identified in other vegetable oils. The functional group (C=O, C=H and CH stretch) identified in Table 5 are typical of vegetable oils. SBT and SBS have similar functional group and fingerprint regions like any other vegetable oil. In all the results shown, C=C was absent, this corroborate with the iodine and peroxides value obtained, which is low iodine values (<100 mg/I₂), this showed that the butter is saturated and the low value of peroxide value also indicate that the self-life is longer than those with C=C. The absence of C=C indicates that reaction at this region is impossible. This showed that this

particular butter cannot be used for some reactions that involves C=C.

Conclusion

This study revealed the effect of three independent process conditions on the percentage yield of shea butter. The results suggested optima values as: shea kernel sample weight of 30.04 g, 346.04 ml of n-hexane and extraction time of 40 min for 66.90 % yield of shea butter. The physico-chemical properties of the SBS showed similar properties with other vegetable oils which made the butter suitable for many applications but rather, SBT is more suitable for biodiesel production, cosmetics, medicinal and foods use, than SBS. The results of FT-IR in this work showed that FT-IR spectral regions could be very useful for determination of the effect of traditional and solvent extraction method on SB. Although the differences in the FT-IR spectra are few, of course, most vegetable oils are expected to contain some content such as fatty and triglyceride. The bond presence identified at 1707.13 cm⁻¹ and 941.99 cm⁻¹ for SBS only can be said to determine the effect of method of extraction on SB.

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