Comparative Evaluation of Mechanical and Solvent-Based Shea Nut Oil Extraction from Shea Nut Seeds

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DOI: https://doi.org/10.51244/IJRSI.2026.1307000313

Received: 24 July 2026; Accepted: 30 July 2026; Published: 17 August 2026

ABSTRACT

Solvent extraction and mechanical extraction are the two main methods for oil extraction from oil-bearing materials, each with unique advantages and drawbacks. Understanding these differences is crucial for businesses seeking efficient oil recovery. Ongoing global research aims to enhance the quantity and quality of essential oils, including shea butter derived from the seeds of the shea tree (Vitellaria paradoxa), which is prevalent in Africa. This study specifically focused on these two extraction techniques: mechanical extraction and optimized solvent extraction. In this study, the extraction of shea nut oil was optimized using Box-Behnken Design (BBD) and Response Surface Methodology (RSM). Key factors included reaction temperature (50 to 100°C), duration (30 to 60 minutes), and solid-to-liquid ratio (2 to 10). The optimized process yielded 44.39% shea nut oil through solvent extraction, with properties such as density (0.9336), viscosity (92), acid value (26.367 mg KOH/g), free fatty acids (13.184 mg KOH/g), saponification value (213.567 mg KOH/g), peroxide value (172 meq O_2/kg), iodine value (114.21 g I_2/100 g), flash point (230°C), pour point (15°C), and cloud point (17°C). The yield of 36.23 % shea nut oil was achieved by mechanical extraction with density of at 0.914, viscosity of 94, acid value of trace mg KOH/g, FFA of trace mg KOH/g, saponification value of 220.2 mg KOH/g, peroxide value of 169 milli equivalent O_2/kg, iodine value of 116.3 gram I_2/100g, flash point of 235°C, pour point of 17^0C, cloud point of 19^0C. The mechanically and solvent extracted shown to meet global standards. The mechanical extraction technique yielded superior oil compared to the solvent extraction method, making it preferable for food, biodiesel, cosmetics, medicine, and pharmaceuticals. FTIR analysis identified key functional groups, confirming the oil's moisturizing properties. Additionally, with optimal conditions at 75°C and a solid-to-liquid ratio of 6 w/v, and 45 minutes reaction time, the highest yield for solvent extraction reached 44.39%, while mechanical extraction yielded 36.23%.

Keyword: Response surface methodology, solvent extraction, shea nut seeds, shea nut oil and mechanical extraction.

INTRODUCTION

In the food and cosmetics industries, shea nuts are a crucial component. She has seen a 600% growth in export quantities since the demand for cocoa butter equivalents increased after 2000 (Wardell et al., 2021a). In a similar vein, prices for shea nuts increased fivefold between the 1990s and 2013 (Wardell et al. 2021a) (Rousseau, Gautier, and Wardell 2015). In West Africa, the majority of shea nut collectors are women and children, and they often have little alternative means of revenue to sustain their way of life (Pouliot 2012). NGOs, development organisations, and international businesses have taken notice of the rising demand for shea nuts. They often portray women's incorporation into the global shea value chain as a win-win situation in which women who gather shea nuts may attain empowerment and economic emancipation (Bockel et al. 2020; Elias and Saussey 2013; Questiaux and Pouliot 2025). Shea butter is notable for having a high percentage of content (about 8%) with therapeutic qualities. It protects and repairs damaged skin and hair and contains essential fatty acids. Vitamins A, E, and F, along with other vitamins and minerals, are known to be abundant in it naturally. In addition to providing skin collagen and functioning as anti-agents for wrinkles and other symptoms of ageing, vitamins A and E aid to balance, moisturise, and smooth the skin (Han et al., 2022). In addition, it is used as a skin unguent and to cure minor aches, pains, sprains, and dislocations. It may also be used as a lubricant and as an antibacterial agent to speed up wound healing. Additionally, you may use the nut shells to keep insects away. Most importantly, the seed may be crushed to produce vegetable oil for use in cooking, soap production, and skin and hair care (Abdul-Mumeen et al. 2013b). There are many uses in the food, cosmetics, pharmaceutical, and biodiesel sectors cannot be overstated. The growing significance of its features is justified by the extensive variety of empirical applications. Traditionally, women gather nuts and use the extraction procedure to manufacture butter (Abdul–Mumeen et al., 2013b). Shea fruit is an economically important crop, especially for many African nations. Research on shea fruit may boost agricultural production and farmers' incomes, as well as provide a number of advantages and aid in the growth of other economic sectors. Farmers may gain from higher production and profitability via better processing techniques and higher yield and quality (Kamga, 2023). Many people in the North Central States of Nigeria, particularly women, rely on shea tree activities as a source of income, however the amount of money earned is often minimal in comparison to the potential. There are several reasons for this outcome. First, poor-quality fruits and butter with cheap pricing are produced by the handling and processing techniques used, which limits access to foreign markets. This problem is made worse by collectors' and processors' ignorance of better handling and processing techniques and tools. LADGROUP Limited, a trading company that was established in 1971, has made an investment in an oilseed (shea nut) processing plant with a 39,000 metric tonnes (MT) annual capacity in Ikenna, Ogun State, Nigeria. The Ladgroup expansion was designed to satisfy the increased demand for shea nut goods made both domestically and abroad.

The extraction procedure then entails extracting the shea butter from the shea nut, which is essential to the total production and quality. These include chemical (solvent extraction), biological (enzymatic extraction), mechanical (machine), and manual (traditional). Gathering the shea nut, de-pulping, boiling, drying, removing the shell and drying the kernel, breaking into grit, roasting, milling and grinding into paste, kneading, and boiling the fat are all labour-intensive and highly engaging traditional methods that are primarily carried out by women. Because of the increased contact area, pore opening, cell wall cracking, and decrease in oil viscosity and moisture, size reduction and heat pretreatment improve oil extraction. At the farm size, cylinder-hole screw presses are the most popular, regardless of the pretreatment, and extraction yields between 65 and 90 percent are usual (Savoire Raphaelle et al., 2013). After this process and stress, shea butter was eventually produced. But the mechanical process is simple: a mechanical crusher is used to smash the roasted kernel, and a hydraulic press is used to extract the oil from the resulting paste.

This approach is less successful than solvent extraction, which entails mixing the powdered kernel from grinding and drying the shea nut for shea butter extraction with chemicals like n-hexane, ethanol, or isopropanol; the solvent is then extracted from the mixture by distillation, evaporation, and condensation. Additionally, the enzymatic process extracts shea butter from shea nuts using enzymes. The best quality and yield are produced by this extraction technique, which may be set up in moderate settings but requires an effective optimisation procedure due to the high cost of enzymes. Since this approach works well for small-scale activities, it is not thought to be ideal for large-scale or industrial application, Thinnakorn and Tscheikuna (2014). Kouame Fulbert

Oussou and his colleagues' research revealed that extraction techniques significantly influence the quality and chemical makeup of the material examined.

Despite their advancements throughout time, each of these extraction techniques has pros and cons. As a result, an improved and more ecologically friendly approach has been created and put into practice throughout history. The response surface methodology (RSM) is a systematic application of mathematical and statistical techniques used with modern technological advancements that allows researchers and engineers to obtain the best tuning of parameters, the actual temperature, pressure, time, and concentration of a substance that yields the best quality and quantity of the product. Because of its increased efficiency, improved oil quality, cost-effectiveness in large-scale production, and controllable environmental impact when contemporary procedures are implemented, the solvent extraction process has shown to be beneficial for shea nut oil extraction. The literature has documented the characterisation and optimisation of solvent extraction of shea butter (Vitellaria paradoxa) using response surface approach (Ajala, E. O., et al., 2016). Nevertheless, little is known about optimisation studies that contrast the mechanical and solvent extraction methods for shea nut oil, particularly with respect to determining the ideal variables for this investigation. This inquiry was required since there is enough data to provide conclusions that are statistically acceptable. Optimisation has effectively used response surface methods (Raymond, H. et al., 2016).

MATERIALS AND METHODS

Materials

The primary raw materials used in this investigation was shea nut seeds, which were purchased from a local market in Benin City, Edo State, Nigeria. They were purchased fresh and used as a source of vegetable seeds for the production of shea nut oil. The chemicals and reagents utilised in this investigation were supplied by accredited producers as well as local analytical chemical dealers. They were obtained under the most favourable circumstances and grades.

Methods

Solvent Extraction Process

Response Surface Methodology (RSM), Box-Behnken Design (BBD), and optimisation of shea nut oil extraction was used in this study. Reaction temperature (50 to 100°C), reaction duration (30 to 60 minutes), and solid-to-liquid ratio (2 to 10) were factors that were independent of the process. As shown in Table 2, the design expert generated seventeen (17) experimental runs with results ranging from respond ($R_{1)}$ to respond ($R_{17)}$. In this study, the Soxhlet apparatus equipped with a heating mantle was used. The shea nut kernels were free of pollutants and impurities after being removed from the nuts. Shea nut seeds were dried until the moisture level was less than 7%. This was done to stop fungus from growing on the kernels and to deactivate the enzymes that were causing the accumulation of unwanted fatty acids.

A desktop mechanical grinder was used to crush the dried seeds into very fine particles (about 0.5mm to 0.1mm). The Soxhlet device, which was set up on a heating mantle, was filled with 200g of pulverised shea nut seeds. Laboratory/Industrial Grade of High Purity: A selection of n-hexane with 65% purity was used for optimal shea nut oil yield.

The combination of solvent-ground shea nut seeds was continuously heated and stirred using a calibrated hotplate with a magnetic stirrer to set the extraction temperatures.

During different experimental runs, the hotplate was set to extraction temperatures of 50°C, 75°C, and 100°C. In order to keep the paste kernels from adhering to the flask walls when heated, the hotplate's integrated magnetic stirrer, made it easier to automatically agitate the combination of solvent (n-hexane) and ground shea nut seeds. All the experiment runs were stirred at 100 rpm (Enoch Mbawin Alale, et al., 2024). The heating mantle was placed next to the power supply outlet on the table. The Soxhlet extraction equipment was designed to be secured by the retort stand. A rubber tube was used to connect the condenser to the Soxhlet device so that water could circulate. The power supply was linked to the heating mantle. The n-hexane's vapour was condensed back into the boiling flask using a water bath and condenser (Enoch Mbawin Alale, et al., 2024). This was established for each of the 17 experimental runs, and n-hexane was used as the solvent for the extraction process, which was repeatedly allowed to reflux. This procedure was carried out around 10 times until the whole shea nut oil was extracted from the crushed shea nut seeds. The oil was subsequently extracted from the n-hexane-oil mixture (miscella) via distillation.

Table 1: Build Information

File Version	13.0.1.0		
Study Type	Response Surface	**Subtype**	Randomized
Design Type	Box-Behnken	**Runs**	17
Design Model	Quadratic	**Blocks**	No Blocks
Build Time (ms)	1.0000		

Table 2: Shows the independent variable with the standard deviation value Factors

Factor	Name	Units	Type	SubType	Minimum	Maximum	Coded Low	Coded High	Mean	Std. Dev.
A	Reaction Temperature	0C	Numeric	Continuous	50	100	-1 ↔ 50.00	+1 ↔ 100.00	75.00	17.68
B	Reaction Time	MINUTES	Numeric	Continuous	30	60	-1 ↔ 30.00	+1 ↔ 60.00	45.00	10.61
C	Solid To Liquid Ratio	W/V	Numeric	Continuous	2	10	-1 ↔ 2.00	+1 ↔ 10.00	6.00	2.83

Mechanical Extraction Method

This section detailed the experiment performed by the mechanical extraction technique to assess its efficacy in obtaining shea nut oil. This technique used a manual grinding apparatus to pulverised shea nut kernels.

Procedure

The prepared seeds were subjected to oven drying at 70^0C until a stable weight was achieved, a desktop mechanical grinder was used to crush the dried seeds into very fine particles (about 0.5mm to 0.1mm). A conventional manual screw press was used (Martínez, et al., 2012). The screw press comprises an elevated collecting platform that supported a perforate cylinder. A frame affixed to the cylinder supports, a rotatable screw with a piston face that ascends and descends inside the cylinder. Approximately one kilogram of ground shea nut seeds was placed into a perforated cylinder, and the experiment was conducted in three experimental runs, time duration was varied between 10 minutes and 30 minutes, where the Screw Rate: Diminished rotation rates permit greater pressure accumulation and improved extraction. The extracted shea nut oil was allowed to cool before being filtered to eliminate solid particulates. The outcomes achieved were documented.

At the conclusion of each run, the shea nut cake residue was collected, and the quantity of extracted shea nut oil was recorded. The design lacked ergonomic consideration for women, since the levers for twisting the screw

ISSN No. 2321-2705 | DOI: 10.51244/IJRSI |Volume XIII Issue VII July 2026

were short and needed upper body strength to function, particularly when more force was necessary to press the shea nuts deeper for enhanced yield.

Degumming and Purification

The oil was heated to 60^0C, and commercially acquired activated carbon was added, resulted in the oil being decolourised. The decolourised oil was completely combined with water and thereafter heated to 60^0C, agitated rapidly for 15 minutes, filtered, cooled, and the residue on the filter paper was eliminated. The purified oil was placed into a glass container and refrigerated until all examinations were finalised (Akpan et al., 2005).

Extracted shea nut oil by both production process was characterized.

The ASTM method was used to characterised the physical and chemical properties of shea nut oil extracted via both processes. The various tests performed are described below

Assessment of Moisture Content:

Fifty grams (50g) of the purified shea nut oil was measured and subjected to drying in an oven at 80^0C. Every two hours, the shea nut oil was removed from the oven and positioned in the desiccator for a 30-minute cooling period. Subsequently, it was extracted and measured (Akpan et al., 2005). The moisture content of the shea nut oil was subsequently determined from:

$$\text{Moisture content of shea nut oil} = = \frac{100(w_1 - w_2)}{w_2}\% \qquad (1)$$

where

W_1 = Original weight of shea nut oil before drying (g), W_2 = Weight of shea nut oil after drying (g)

Assessment of Specific Gravity:

The specific gravity bottle was purified using acetone and ether, then dried in an oven at 60 degrees Celsius. The mass of the vacant bottle was recorded, thereafter the container was filled with the shea nut oil specimen and securely sealed. The mass was then documented using a weighing scale, after which the shea nut oil was extracted from the container. The container was thoroughly cleaned and filled with distilled water, after which its weight was measured, and subsequently, the specific gravity was calculated using the formula provided (Akpan et al., 2005).

$$\text{Specific Gravity} = = \frac{w_0 - w}{w_1 - w} \qquad (2)$$

where, W = Weight of empty bottle (g), Wo = weight of the bottle and shea nut oil content (g), W_1 = Weight of bottle and water content (g).

Assessment of Acid Value:

A shea nut oil weighing two grams was dissolved in 50cm³ of a neutral solvent mixture (25cm³ of diethyl ether combined with 25cm³ of ethanol, which was meticulously neutralised with 0.1M NaOH using a 1% phenolphthalein solution). The solution was titrated with a 0.1M NaOH aqueous solution while continuously agitating until a pale pink hue was seen (Akpan et al., 2005).

$$\text{Acid Value} = \frac{Titre\ value \times 5 \times 0.00282}{weight\ of\ shea\ nut\ oil\ (g)} = \text{mgKOH/g} \qquad (3)$$

Assessment of Free Fatty Acids:

The quantity of free fatty acid (FFA) was determined to be half the acid value (Akpan et al., 2005), specifically,

$$\text{FFA} = \frac{\text{Acid value}}{2} = \text{mgKOH/g} \quad (4)$$

Assessment of Saponification Value:

A 0.5 M KOH solution was formulated in 95% ethanol, 2 grams of the shea nut oil sample were quantified, and $25cm^3$ of KOH was introduced, along with 25 cm^3 of the blank solution into a conical flask. The two samples were then linked to a reflux apparatus and let to boil for one hour until reflux was achieved; thereafter, $1cm^3$ of phenolphthalein was incorporated into the mixture, which was then titrated while still hot against a 0.5 M HCl solution. The quantity of acid employed to reach the endpoint was documented, and the blank measurement was performed following the aforementioned procedure until the colour transitioned from blue to clear white, after which the volume of acid utilised was recorded, and the Saponification value was calculated using the equation provided below (Akpan et al., 2005).

$$\text{Saponification value, (SP)} = \frac{56.1 \times T \times (v_0 - v_1)}{M} \quad (5)$$

Where, T= Molarity of the standard KOH solution used (M), Vo = Volume of acid used for the first titration with shea nut oil sample (cm^3), V_1= Volume of acid used for the second titration of the blank solution (cm^3), M= Mass of the shea nut oil sample used (g).

Assessment of Peroxide Value:

A specified mass (2g) of the shea nut oil sample was placed into a clean, dry boiling tube, followed by the addition of 1 gram of potassium iodide (KI) powder and $20cm^3$ of a solvent mixture (i.e., glacial acetic acid and chloroform in a 2:1 ratio). Subsequently, the boiling tube was immersed in a boiling water bath to ensure the liquid mixture reached a boil within 30 seconds, and it was permitted to boil vigorously for no longer than 30 seconds; the contents were then swiftly transferred into a flask containing $20cm^3$ of 5% potassium iodide (KI) solution, and the tube was rinsed twice with $25cm^3$ of water. Subsequently, the mixture underwent titration with 0.002 M sodium thiosulphate using a new 1% starch solution, and a blank titration was performed concurrently; the peroxide value was determined using the equation provided (Akpan et al., 2005).

$$\text{Peroxide value (PV)} = = \frac{T \times M \times 1000}{\text{weight of shea nut oil } (g)} \quad (6)$$

T represents the titre value of $Na_2S_2O_3$, defined as the sample titre minus the blank titre, and M denotes the molarity of $Na_2S_2O_3$.

Assessment of Density and Specific Gravity:

The density was assessed in accordance with the ASTM D1298 standard, whilst the specific gravity was evaluated following the ASTM D1217 standard. A 50 cc SEDI-M pycnometer vessel was meticulously cleaned with soap, water, and petroleum ether; thereafter, it was dried in an oven and weighed. The container was filled with distilled water and measured; then, it was dried and filled with the shea nut oil sample before being weighed again. According to the theory, specific gravity is defined as the mass of the oil measured divided by the mass of the water measured, while the density of the oil is equivalent to the mass of the oil per unit volume (Sahasrabudhe et al., 2017).

Flash Point Assessment (ASTM D 93):

The flashpoint of shea nut oil was established using the Pensky-Martens closed cup technique. The cup was filled with shea nut oil to the necessary level (about 75ml) and positioned in the tester. The apparatus was configured to heat at about 5^0C/min until an increase in temperature accompanied with concurrent stirring was detected. A little open flame was sustained using an external source of petroleum gas. At intervals, the flame was traversed over the shea nut oil's surface. Upon reaching the flash temperature, the shea nut oil's surface ignited; the temperature at that instant was recorded and subsequently documented as the flash point temperature.

Iodine Index:

The iodine value signifies the level of unsaturation in shea nut oil constituents, measured by halogen absorption. The iodine value offers an assessment of these quality metrics, since the degree of unsaturation is linked to the melting point and oxidative stability. Elevated iodine readings are associated with increased unsaturation and enhanced vulnerability to oxidation. The iodine value of the extracted shea nut oil was assessed by placing 1 gram of the oil into a conical flask, introducing 10 millilitres of chloroform (CCl_4), applying gentle heat, and allowing the mixture to cool for 10 minutes. 25ml of Wij's reagent (iodine trichloride solution) was added to the flask and stirred vigorously; thereafter, the mixture was let to sit in darkness for 30 minutes to avert any reaction with sunlight. Following the designated time period, the solution was titrated with sodium thiosulphate until a yellow colouration appeared. Thereafter, 20ml of 10% KI and 150ml of purified water were included. The yellow solution obtained was titrated with sodium thiosulphate, using a 1-percent starch solution as an indicator, until a transition from indigo/black to colourless was reached (endpoint). The same parameters were used for a control experiment (Yildz, et al., 2019).

$$\text{Iodine Value} = \frac{(V - V_O) \times M \times N \times 100 \times 126.9}{M} \quad (7)$$

Where: V = volume of $Na_2S_2O_3$ needed for titrated of blank in ml, V_0 = volume of $N_2S_2O_3$ needed for titraton of test sample in ml, W = mass of shea nut oil sample in gram and M= molecular mass of iodine = 126.9 in gram.

Measurement of Viscosity (ASTM D 445):

A Brookfield NDJ-5S rotating viscometer was used to ascertain viscosity. The suitable spindle number was determined and chosen for the test specimen and carefully affixed to the apparatus. A 250ml beaker was thoroughly washed, and the shea nut oil was added until it reached the 200ml line. The beaker was thereafter positioned in a water bath set to a stable temperature of 30^0C and let to stabilise for 10 minutes. The spindle and temperature sensor of the apparatus were thereafter immersed in the shea nut oil, and the power switch was activated. The correct spindle number and velocity were chosen on the display interface and then activated by the run button. The apparatus was thereafter permitted to measure the viscosity until a consistent value was achieved and documented (Sahasrabudhe et al., 2017).

RESULTS AND DISCUSSION

Yield comparison:

The yield of shea nut oil from mechanical extraction was much lower than that from solvent extraction. This difference was shown in small-scale manufactured using rudimentary equipment, such as a manual grinding machine; the mechanical extraction method is less effective than Soxhlet extraction. We acknowledged that further studies using advanced mechanical extraction equipment might produce diverse results and provide a more comprehensive understanding of the possibilities of this technology. As seen in Table. 3 and Table 6, the Solvent extraction technique achieves a yield of 44.39%, markedly above the mechanical extraction yield of 36.23%. We shall notify our informed colleagues to use the solvent extraction process for obtaining shea nut oil from shea nut seeds for industrial, large-scale production and the mechanical extraction process for obtaining shea nut oil from shea nut seeds is preferable for food, biodiesel, cosmetics, medicine, and pharmaceuticals products due to it traces of acid values and unbounded free fatty acid in shea nut oil.

Predicted And Actual Value Parity Plot Values

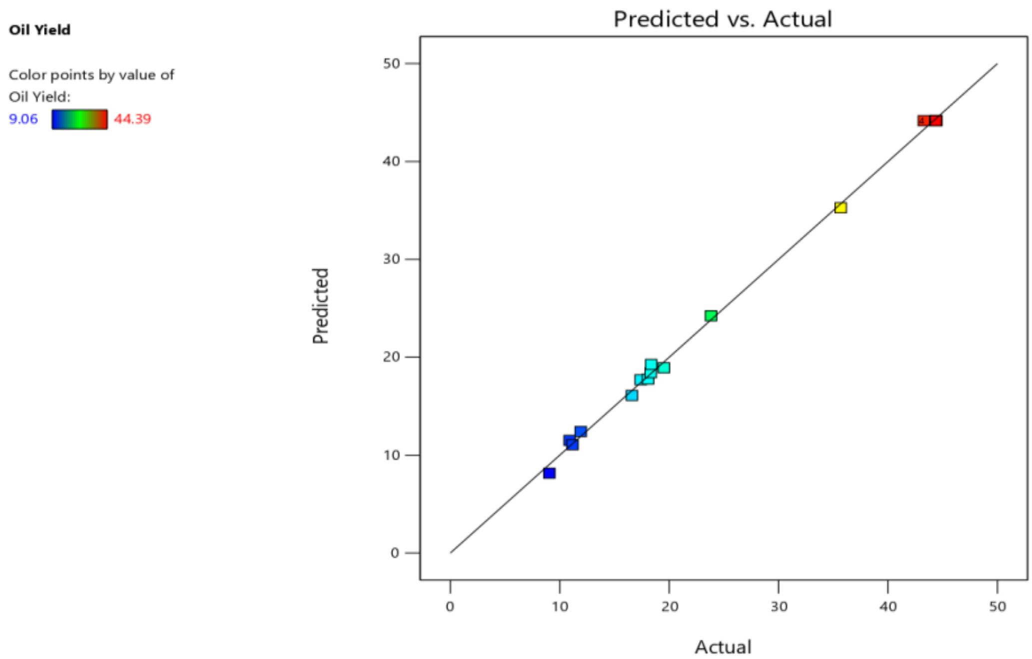


Figure 1: Lack of Fit for the Quadratic model.

Figure 1 illustrated the relationship between projected and actual yields for shea nut oil extracted by the solvent extraction method and the Box-Behnken experimental design. The graphs showed that the data points were spread near a straight line. The high correlation (R^2 value close to unity) indicated that the data fits well with the models and provides a reliable estimation of the system's reaction within the tested ranges.

Process Modelling, Optimization and determination of extraction yield by Solvent Extraction

The experimental design of the solvent extraction of the shea nut oil, and the predicted responses were presented in Table 3. Equation 8 represents the quadratic model (actual-basis).

$$\textbf{Shea nut oil yield \%} = +44.17 - 0.8675\,A + 0.9763\,B + 4.61\,C + 1.67\,AB - 7.27\,AC - 0.5175\,BC - 9.99\,A^2 - 18.25\,B^2 - 11.69\,C^2 \quad (8)$$

Equation 8 indicated that reaction temperature A, interaction factors (AC and BC), and quadratic factors (A^2, B^2, and C^2) adversely affected the yield of shea nut oil via solvent extraction, whereas reaction time (B), solid to liquid ratio (C), and interaction factor (AB) positively influenced the yield of shea nut oil through solvent extraction. Models with a p value below 0.05 are considered significant at the 95% confidence level. This indicated that the yield % of shea nut oil was substantially influenced by variations in the values of the input variable indicated by that model term. Conversely, model terms with p values over 0.05 are deemed insignificant, indicating that fluctuations in the input variables would not substantially influence the yield of shea nut oil derived by solvent extraction.

The quadratic models A^2, B^2, and C^2 from Table 4 were shown to be significant in forecasting the shea nut oil output by the solvent extraction technique, with p values below 0.0001. The study using factor coding revealed a substantial model characterised by an F-value of 521.87 and a Type III partial sum of squares, indicated an only 0.01% probability that this outcome was attributable to random variation. A, B, C, AB, AC, A^2, B^2 and C^2 are significant model terms with P-values below 0.0500. Values beyond 0.1000 are deemed inconsequential (Ayoub, M., et al., 2016). In scenarios with several extraneous factors, model reduction may be advantageous. According to Wenjun Zhang (2022), to transform the scientific research paradigm, it is essential to establish a framework of numerous repeated experiments and multi-sample testing, with various parties verifying each other to enhance the legitimacy and reproducibility of findings.

Table 3: Percentage Shea Nut Oil Yield From Shea Nut Seeds (Soxhlet Extraction).

C		Factor 1	Factor 2	Factor 3	Response 1
Std	**Run**	**A: Reaction Temperature (°C)**	**B: Reaction Time (minutes)**	**C: Solid to Liquid Ratio (W/V)**	**Oil Yield (%)**
12	1	75	60	10	18.36
15	2	75	45	6	44.39
7	3	50	45	10	35.68
11	4	75	30	10	18.32
3	5	50	60	6	16.6
17	6	75	45	6	44.39
13	7	75	45	6	44.39
1	8	50	30	6	17.1
14	9	75	45	6	43.28
6	10	100	45	2	23.83
9	11	75	30	2	9.06
10	12	75	60	2	11.17
2	13	100	30	6	11.92
16	14	75	45	6	44.39
5	15	50	45	2	10.91
4	16	100	60	6	18.08
8	17	100	45	10	19.52

Shea Nut Oil Yield Anova For The Quadratic Model

Table 4: Responsed, shea nut Yield

Source	Sum of Squares	Df	Mean Square	F-value	p-value	
Model	**3061.08**	**9**	**340.12**	**521.87**	**< 0.0001**	**Significant**
A – Reaction Temperature	6.02	1	6.02	9.24	0.0189	Significant
B – Reaction Time	7.62	1	7.62	11.7	0.0111	Significant
C – Solid to Liquid Ratio	170.29	1	170.29	261.29	< 0.0001	Significant
AB	11.09	1	11.09	17.01	0.0044	Significant
AC	211.41	1	211.41	324.38	< 0.0001	Significant
BC	1.07	1	1.07	1.64	0.2407	Not significant
A^2	420.44	1	420.44	645.11	< 0.0001	Significant
B^2	1402.41	1	1402.41	2151.8	< 0.0001	Significant

ISSN No. 2321-2705 | DOI: 10.51244/IJRSI |Volume XIII Issue VII July 2026

C^2	575.42	1	575.42	882.9	< 0.0001	Significant
Residual	**4.56**	**7**	**0.6517**	—	—	—
Lack of Fit	3.58	3	1.19	4.84	0.0809	Not significant
Pure Error	0.9857	4	0.2464	—	—	—
Cor Total	**3065.64**	**16**	—	—	—	—

Fit Statistics

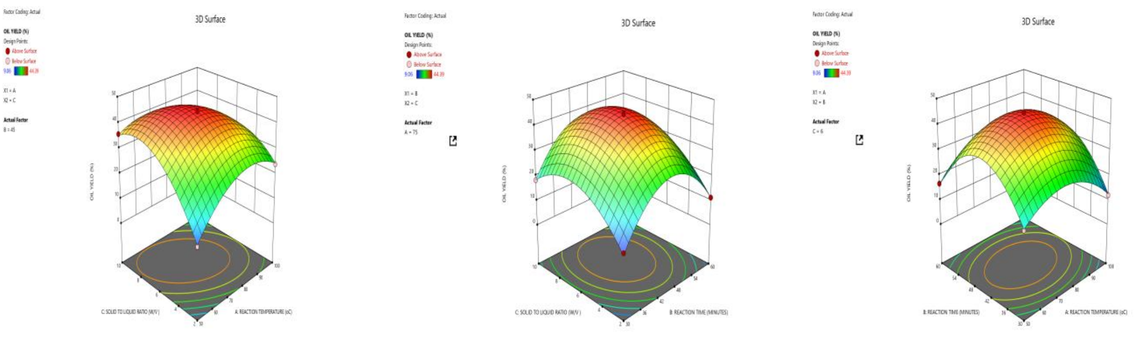
Table 5: Fit statistics

R^2	0.9985
Adjusted R^2	0.9966
Predicted R^2	0.9808
Adeq Precision	58.2197
Std. Dev.	0.8073
Mean	25.38
C.V. %	3.18

The fit statistical data revealed a R^2 of 0.9985, an adjusted R^2 of 0.9966, and an expected R^2 of 0.9808. The predicted R^2 and adjusted R^2 values aligned because the discrepancy was smaller than 0.2. The yield of shea nut oil obtained by solvent extraction may be forecasted using this model. The signal-to-noise ratio was measured with enough accuracy. A ratio over four is preferable. The ratio of 58.2197 indicated a robust signal. This paradigm enables navigation inside the design space.

Effect of input variables on shea nut oil yield by solvent extraction method.

The effects of process parameters on shea nut oil yield were analyzed by three-dimensional (3D) surface plots.



(a) (b) (c)

Figure 2: 3D plots shown interaction between (a) Solid to liquid ratio and reaction temperature (b) Solid to liquid ratio and reaction time (c) Reaction temperature and Reaction time.

ISSN No. 2321-2705 | DOI: 10.51244/IJRSI |Volume XIII Issue VII July 2026

The impact of the process factors on the yield of shea nut oil depicted in Figure 2 (a, b, and c). Reaction temperature (A), reaction time (B), and solid-to-liquid ratio (C) resulted in a yield of shea nut oil of 44.39% via solvent extraction. The optimal conditions for the extracted shea nut oil using the solvent method were determined to be a temperature of 75°C, a reaction time of 45 minutes, and a solid-to-liquid ratio of 6, which maintained a yield of 44.39% through optimisation via the Box-Behnken design.

Analysis of the optimal area

The curvature of the response surface indicated a certain optimal area, particularly evident in the interaction between reaction temperature (A) and the solid-to-liquid ratio (AC). This demonstrated that sustaining both parameters within this range was crucial for attaining optimum yield, supporting the conclusions of Khalid Rahal et al. (2025) about the optimisation of extraction conditions in plant-based methods.

Percentage shea nut oil yield by mechanical extraction

$$\text{RUN 1: Percentage shea nut oil yield} = \frac{90}{1000} \times 100 = 9\%$$

$$\text{RUN 2: Percentage shea nut oil yield} = \frac{318}{1000} \times 100 = 31.8\%$$

$$\text{RUN 3: Percentage shea nut oil yield} = \frac{362.3}{1000} \times 100 = 36.23\%$$

Table 6: Mechanical extraction result

S/N	Pressing time (minutes)	Original weight of grinded shea nut seed (grams)	Weight of shea nut cake (grams), after extraction	Weight of shea nut oil extracted (grams)	Yield %
Runs 3	30	1000	637.7	362.3	36.23
Runs 2	20	1000	682	318	31.8
Runs 1	10	1000	910	90	9

Comparison of performance of the RSM via solvent extraction method and mechanical extraction method.

The fundamental distinction observed was in the extraction methodology; mechanical extraction depends only on physical force, whereas solvent extraction uses chemical solubility and mass transfer diffusion. Thus, solvent extraction took much more time (45 minutes) to complete, however it produces substantially greater recovery rates of 44.39%.

Solvent extraction of shea nut oil had a yield efficiency of 44.39%. The extracted shea nut oil included a greater oil yield than that obtained by mechanical extraction (36.23%), making it particularly advantageous for substances with lower oil content. Moreover, solvent extraction shown remarkable versatility as it was used for feedstocks (Singh, et al., 2008). The strong correlation (R^2 value around one) signified that the data aligned seamlessly with the models and offers a dependable estimate of the system's response within the examined ranges. Its scalability makes it an excellent option for industrial applications where optimal shea nut oil recovery is essential.

Nonetheless, the use of n-hexane as a solvent may provide some challenges. This technique requires the careful management of chemical compounds, such as n-hexane, which may pose hazards if improperly handled. N-hexane remains a prevalent solvent for the extraction of natural substances. The regulations in Europe and the United States are unclear concerning the designation "natural," and do not prohibit the use of n-hexane in the manufacturing of natural products. The use of n-hexane as a solvent in the food industry for extracting various products like vegetable oils and additives was common. However, its presence is not typically disclosed on labels, leading to potential traces in final products. N-hexane, has been identified as neurotoxic to humans and

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linked to occupational diseases. To ensure a toxic-free environment and food, it is crucial to gather scientific data on n-hexane through literature reviews, (Christian Cravotto et al., 2022).

A rotary evaporator was used as a purification instrument to eliminate solvent residues, leading to increased operating costs. Moreover, solvent extraction apparatus requires a greater initial capital outlay and ongoing maintenance costs, making it less attainable for smaller-scale companies.

The mechanical extraction procedure yielded pure, chemical-free shea nut oil, (Okoye et al., 2008). This solvent-free approach is often used in the food business, where preserving the original flavour and nutritional properties of oil is crucial. Moreover, mechanical extraction was straightforward when performed, require less advanced apparatus in contrast to solvent-based methods, (Okoye et al., 2008). Its ecological characteristics provide it a feasible solution for the production of sustainable goods.

In contrast, mechanical extraction produced lesser amounts of shea nut oil (36.23%) than solvent extraction (44.39%), since a portion of the oil remained confined in the residual cake of ground shea nut seeds. This led to increased demands for raw-ground shea nut seeds to meet manufacturing objectives. Another concern was that mechanical extraction may not be suitable for all feedstocks, especially those with tightly bonded oil molecules. The procedure generated heat via friction, which did not compromise the quality of heat-sensitive oils, since the observed temperature was not more than 58°C.

Optimization of shea nut oil yield

The numerical optimisation analysis revealed that the optimal yield of shea nut oil, 44.39%, was achieved through a solvent extraction method utilised a solid-to-liquid ratio of 6 (using n-hexane), a reaction duration of 45 minutes, and a temperature of 75^0C. According to prior research using n-hexane for extraction, the anticipated maximum yield of the oil was 70.2%, attained at the peak extraction temperature of 60^0C and the longest extraction duration of 130 minutes. In a similar vein, this research recorded reaction durations of 45 minutes (almost three times the reaction time of Mbawn et al., 2024) and achieved a yield of shea nut oil at 44.39%, consistent with the results of Enoch Mbawin Alale et al., 2024.

Conversely, a lower yield of 36.23% was attained via a mechanical extraction method and a pressing duration of 30 minutes and a friction-induced pressing temperature of approximately 58°C.

A prior investigation performed by Ajala Elijah Olawale and colleagues in 2019 examined the extraction of shea nut oil using mechanical means, optimising the process using Box-Behnken experimental design within the framework of response surface technique. The examined process parameters, together with their respective ranges, were sample weight (100 – 200 g), temperature (60 – 120°C), and duration of applied pressure (10 – 30 min) to enhance the production of shea nut oil. The properties of shea nut oil were assessed by conventional techniques and Fourier transform infrared spectroscopy (FTIR). A comparative analysis of shea nut oil obtained by mechanical extraction vs other extraction techniques was conducted. The ideal yield of 37% (w/w) of shea nut oil was achieved from a 150 g sample of shea kernel at 90^0C for 20 minutes. The R^2 (0.9957) derived from the analysis of variance indicated that the quadratic model of Box-Behhnken accurately represented the experimental data. The attributes of the shea nut oil from mechanical extraction exhibited unwavering quality, yielding more than the conventional approach but less than the solvent method. This research demonstrated that the techniques of extraction influence both the quantity and quality of shea nut oil and the results obtained in this studied.

Experimental trials were performed in triplicate using the solvent extraction technique. The average results best process was used to validate these ideal conditions. The solvent extraction method yielded an average of 44.197%, demonstrating a significant association with the optimal yield, however the mechanical extraction technique was verified. Experimental trials were performed in triplicate using the mechanical extraction technique. The only process parameter used to assess shea nut oil production was a pressing time of 30 minutes. The mechanical extraction method yielded an average of 36.18%, demonstrating a robust association with the original yield.

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Shea nut oil characterization

Table 7 presents the results of an examination of the physicochemical properties of shea nut oil extracted via both solvent and mechanical methods. In comparison to the standards of ASTM D6751 and EN14214, the qualities of shea nut oil were found to be within the acceptable range.

Table 7: Shown the Comparison of the Literature Value and the Experimental Values of shea nut oil characterization.

S/N	Parameter	Literature Value Range	Experimental Result (Mechanical method)	Experimental Result (Solvent method)	Remarks
1.	Density (g/cm^3)	0.90 – 0.93	0.914	0.9336	This was slightly over the typical range
2.	Viscosity	50-100	94	92	Reflects moderated thickness
3.	Specify gravity	0.91-0.92	0.914	0.9336	This was the same with the density, indicating purity
4.	Acid value (mg KOH/g)	3.5-6.0	Traces	26.367	It shows a High acid value in solvent extraction, indicated a High FFA
5.	FFA mgKOH/g	1.75-3.0	Traces	13.184	High FFA content, in solvent extraction.
6.	Saponification value (mg KOH/g)	160-195	220.2	213.567	Higher than the typical range, this was because the oil has started ranciding
7.	Peroxide value (milli equivalents O_2/kg)	1.0-5.0	169	172	Extremely high, indicating significant oxidation
8.	Iodine value (g I_2/100g)	50-70	116.3	114.21	High unsaturation
9.	Flash point ^{0}C	225-245	235	230	Within range
10	Pour point ^{0}C	12-20	16	15	Moderated pour point
11.	Cloud Point ^{0}C	15-22	18	17	Between the typical range
12.	Water content %	<7	6	5	Within range

13.	FTIR	Matches typical O-H, C=O	Matches typical O-H, C=O	O-H at 3526.01 cm^{-1} C-H at 418.65 cm^{-1}	Confirmed the presence of Fatty acids and triglycerides characteristic of Shea nut.
14.	Shea nut oil yield %		36.23	44.39	Higher shea nut oil yield by solvent method

Table 7 provided essential insights into purity, stability, and deterioration, aiding in the assessment of whether the extracted shea nut oil from both procedures was fresh, rancid, or adulterated with inferior components. Essential metrics include acidity Value, peroxide value, and iodine value (Kofi Duodu et al., 2024).

Table 7 elucidated critical information regarding purity, stability, and degradation, facilitating the evaluation that the shea nut oil obtained through mechanical extraction was fresher, less rancid, or less contaminated with inferior substances compared to the oil extracted via solvent methods.

A prior investigation disclosed the outcomes of the extraction, refinement, and characterisation of both crude and refined Nigerian shea butter oil. The characterisation results of crude and refined oil indicate a specific gravity of 0.86 and 0.89, moisture content of 2.29% and 0.12%, saponification values of 389.89 mg KOH/g and 162.61 mg KOH/g, acid values of 8.42 mg KOH/g and 3.36 mg KOH/g, peroxide values of 15 meq/kg and 9.40 meq/kg, and refractive indices of 1.472 and 1.467, respectively, all conforming to the requisite standard specifications. The results of Munir et al., 2001 corresponded with the outcomes of this research with slight deviation.

FTIR of shea nut oil extracted via solvent method

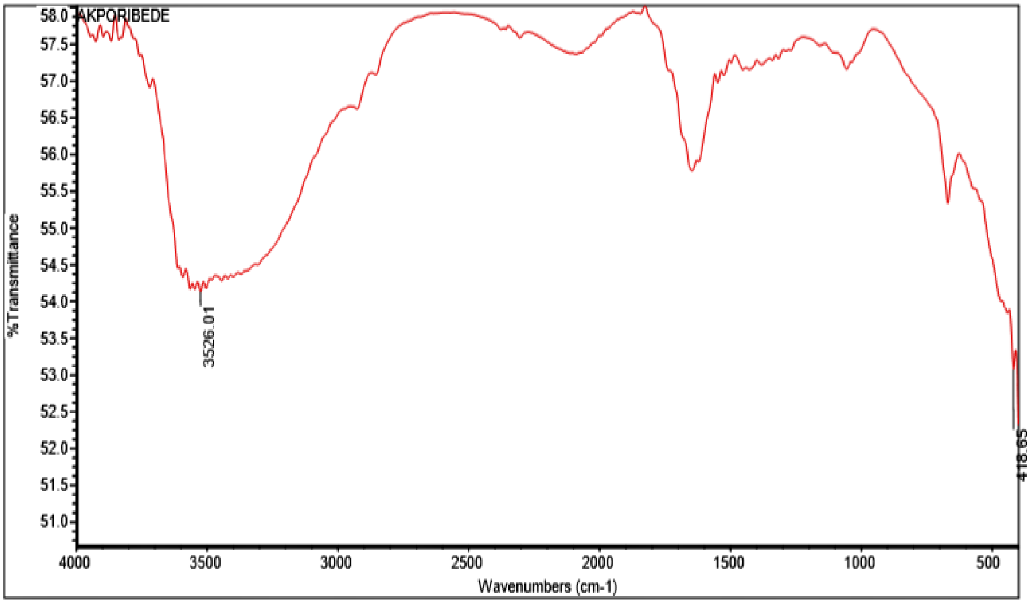

Figure 3: Showing the FTIR of the extracted Shea nut oil

The FTIR spectrum exhibited a peak at 3526.01 cm^{-3}, indicative of O-H stretching in hydroxyl groups, and another peak at 418.65 cm^{-3}, representing C-H bonding (Ashish Soni et al., 2022). The observed peaks aligned with the fatty acids and glycerides present in shea nut oil, so affirmed the existence of functional groups essential for its moisturising effects. The spectral profile verified that the oil's composition renders it appropriate for cosmetics and lotions designed to maintain skin hydration (Mohiddin, A.K., 2019).

ADVANTAGES, LIMITATIONS AND PRACTICAL APPLICATIONS OF BOTH EXTRACTION METHODS (Chin Hong Geow, et al., 2021)

Mechanical Extraction

Advantages

1). Solvent-free: No chemicals used. The product was classified as "organic" / "natural" and is food-grade without further processing.

2). Retains bioactive compounds: Elevated levels of vitamins A, E, triterpenes, and unsaponifiables. Superior for beauty products.

3). More simple apparatus: Reduced initial investment. Can be executed at a localised or collaborative scale.

4). More secure: Absence of fire or explosion risks and no issues with solvent remnants.

5). By-product: Press cake retains around residual oil and may serve as animal feed or fertiliser.

Constraints

1). Reduced oil extraction: 36.23% of oil retrieved from shea nut oil concentration in the kernel. About 10-15% of oil persists in the cake.

2). High energy and labour: Necessitate the pre-heating or roasting of kernels together with elevated pressure.

3). Reduced throughput: Sluggish performance, unsuitable for substantial volumes.

4). Quality fluctuation: Hue and free fatty acids may differ according on roasting temperature and the proficiency of the operator. But in this study extraction was done without roasting temperature.

5). Usage and upkeep: Screw presses need regular maintenance.

Practical Applications

1). Cosmetics and Shea Butter: The majority, 90%, of "shea butter" used in lotions, balms, and soaps is derived from mechanically extracted oil, since consumers want unrefined, natural butter.

2). Small-scale rural processing: Women's cooperatives in West Africa, namely Nigeria, Ghana, and Burkina Faso.

3). Culinary: Unrefined shea butter used in chocolates as a Cocoa Butter Equivalent (CBE).

Solvent Extraction

Seeds are ground, then oil was leached out via solvent extraction, and n-Hexane was used as solvent. Solvent was later recovered by distillation.

Advantages

1). High oil yield: 44.39%% oil recovery, Residual oil in cake <1%, and Maximizes profit from raw material.

2). High capacity: Continuous process, Can process 100-1000 tons/day and Ideal for large factories.

3). Better quality control: Lower colour, FFA, and odour because refining is built into the process.

4). Lower labour cost: Automated, and less manual handling.

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5). Better for low-grade seeds: Can extract oil from damaged/moldy nuts that would give poor butter mechanically.

Limitations

1). High capital cost: Need extractors, solvent recovery, and explosion-proof plants.

2). Solvent residue risk: Must refine to remove n-hexane traces, and Not "natural" for cosmetics.

3). Loss of bio actives: Refining + heat strips vitamins and unsaponifiables. Oil is less valuable for cosmetics.

4). Safety & Environmental: N-hexane is flammable, needs strict safety and emissions control.

5). By-product: Defatted cake has no oil value but is good for animal feed or biomass.

Practical Applications

1). Edible Oil and Food Industry: Refined shea oil for margarine, and confectionery at large scale.

2). Large-scale oil mills: Used in Nigeria, Ivory Coast for export-grade refined shea oil.

3). Soap and Detergent Industry: Where high volume and low-cost matters more than "natural" label.

Which Extraction Technique Is Superior?

1). For Cosmetics, Pharma, and Export "Shea Butter" → Mechanical Extraction

Reason: The premium cost, bioactive components, and "natural" narrative are more significant than output.

2). For Food, and Industrial Soap at Bulk → Solvent Extraction

Reason: Need maximum yield, consistent quality, and low cost per ton.

CONCLUSIONS

This work used response surface methods with n-hexane as a solvent to improve the extraction of shea nut oil from its seeds. The process variables examined were solid-to-liquid ratio (2–10), duration (30–60 minutes), and reaction temperature (50–100°C). The Box-Behnken design yielded 17 runs, with a maximum output of 44.39% at a solid ratio of 6 w/v, a temperature of 75°C, and a duration of 45 minutes.

This research also used a mechanical extraction technique to get shea nut oil from its seeds. The only process variable examined was pressing time (10 to 30 minutes), with the mass of powdered shea nut seeds held constant. The greatest yield of shea nut oil obtained by mechanical extraction was 36.23% at a pressing duration of 30 minutes and a pressed temperature of 58°C.

Despite several measurements exceeded normal literature guidelines, the solvent extraction and mechanically extracted shea nut oil was deemed compliant with international standards. FTIR analysis confirmed the presence of fatty acids and triglycerides characteristic of shea nut oil.

Furthermore, response surface approach had a strong predictive capability, shown by high R^2 values of 0.9985 for the solvent extraction technique. The mechanical extraction approach did not need R^2 due to suboptimal experimental conditions.

The shea nut oil, extracted using mechanical and solvent methods, complies with international requirements. The mechanical extraction approach produced higher quality oil than the solvent extraction method, making it more advantageous for applications in food, biodiesel, cosmetics, medicine, and pharmaceuticals.

In practice, many large plants in Nigeria use a hybrid: Mechanical pre-press to get 70% of oil as "butter", then solvent extract the press cake to recover the remaining oil as "industrial grade oil". In Nigeria, local and hybrid extraction facilities for palm oil, palm kernel, or vegetable oil are mostly found in key agricultural and industrial areas like Ondo, Ogun, Lagos, Kano, Oyo, and Delta states.

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