Physicochemical Characterization of Soxhlet-Extracted Sunflower (*Helianthus Annuus* L.) Seed Oil and its Application in Soap Production: A Comparative Evaluation of Formulated and Commercial Soaps

[1]Agbedo Ifesinachi Sabbath, [1]Amalunweze Adaude Euphemia & [2]Valentine Chukwuma Ezenwa*

[1]Department of Biochemistry,
Federal Polytechnic Oko, Anambra State Nigeria
[2]Department of Applied Biochemistry,
Enugu State University of Science and Technology,
Enugu State, Nigeria.
AIS: agbedoifesinachi@gmail.com +2348138539357
AAE: amalunweze.adaude@federalpolyoko.edu.ng +2348032287552
VCE: valentine.ezenwa2024060000086@esut.edu.ng +2348134842060
*Corresponding author e-mail: valentine.ezenwa2024060000086@esut.edu.ng

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Abstract

Background: *Vegetable oils are potential renewable raw materials for soap production, but their suitability depends on their physicochemical characteristics and fatty acid composition.*

Aim: *This study investigated the physicochemical properties and fatty acid composition of Soxhlet-extracted sunflower (Helianthus annuus L.) seed oil and evaluated its application in soap production through comparative assessment of the formulated and commercial bathing soaps.*

Methods: *Sunflower seed oil was extracted using n-hexane in a Soxhlet apparatus and characterised using standard physicochemical procedures. Fatty acid composition was determined by gas chromatography–flame ionisation detection (GC-FID). Three independent cold-process soap batches were formulated using the experimentally determined saponification value. The formulated soap was compared with commercial Viva bathing soap based on physicochemical and functional properties. Changes in selected characteristics were monitored over six weeks of curing. Data were analysed using Welch's independent-samples t-test with Holm adjustment and the Friedman repeated-measures test.*

Results: *The oil extraction yield was 42.80 ± 0.30%, with a saponification value of 189.34 ± 0.25 mg KOH/g and an iodine value of 128.73 ± 0.35 g I_2/100 g. Linoleic acid (62.10%) and oleic acid (25.40%) were the predominant fatty acids. The formulated soap had a total fatty matter content of 76.17 ± 0.35%, moisture content of 14.00 ± 0.20% and free caustic alkali content of 0.043 ± 0.006%. Significant differences (Holm-adjusted $p < 0.05$) occurred between the formulated and commercial soaps across all evaluated physicochemical and functional parameters. The formulated soap exhibited lower initial foam height (6.90 versus 9.10 cm) and cleansing efficiency (73% versus 78%), but greater dissolution-related mass loss (18.50% versus 15.20%). During curing, moisture content decreased from 13.8% to 10.2%, while relative bar mass declined by 9.8%. Changes in pH were not statistically significant ($p = 0.140$).*

Conclusion: *Soxhlet-extracted sunflower seed oil demonstrated technical feasibility as a lipid feedstock for solid soap production. However, differences in functional performance indicate the need for further formulation optimisation, particularly regarding moisture management, residual free caustic alkali and fatty acid balance.*

Keywords: *Helianthus annuus; sunflower seed oil; Soxhlet extraction; fatty acid composition; soap formulation; physicochemical properties; curing.*

1. Introduction

Vegetable oils are important renewable lipid resources used in the manufacture of soaps and other industrial products. Their suitability for soap production is largely determined by their chemical composition and physicochemical characteristics, which influence alkali requirements and the properties of the finished product. The increasing interest in plant-derived raw materials has sustained research into alternative vegetable oils for soap formulation, particularly those whose fatty acid composition may provide desirable product characteristics. However, the suitability of an oil for soap manufacture cannot be established solely by its availability or extraction yield; its quality indices and saponification characteristics must also be considered (Oyekunle et al., 2021; Nova et al., 2025). Sunflower (*Helianthus annuus* L.) is an important oilseed crop whose seeds contain appreciable quantities of edible oil, predominantly composed of unsaturated fatty acids. Linoleic and oleic acids are generally the major constituents, although their relative proportions vary with cultivar, geographical origin, growing conditions and processing method. Previous investigations have demonstrated considerable variation in the fatty acid composition and physicochemical properties of sunflower seed oil. For instance, Khairuddin et al. (2024) and Hosni et al. (2022) reported differences in the relative proportions of linoleic, oleic and saturated fatty acids among sunflower oil samples. Such compositional variations are relevant to soap production because the fatty acid profile contributes to the chemical characteristics and functional behaviour of the resulting soap. Consequently, the evaluation of sunflower seed oil from different sources remains important when considering its potential as a soap-making material.

The conversion of vegetable oils into soap occurs through saponification, a chemical reaction in which triglycerides react with an alkali to produce glycerol and fatty acid salts. The quantity of alkali required is influenced by the saponification value of the oil, making its experimental determination important for appropriate formulation. In addition, the degree of fatty acid unsaturation and the proportions of individual fatty acids can influence properties such as lather formation, foam stability and the physical characteristics of soap. Beyond formulation, the quality of finished soap is assessed through physicochemical and functional parameters, including pH, moisture content, total fatty matter, free caustic alkali, chloride content, insoluble matter, foaming behaviour and cleansing performance. Previous studies have demonstrated substantial variation in these properties among formulated and commercially available soaps (Abdul et al., 2024; Nova et al., 2025; Maduelosi et al., 2026). Curing is also relevant to solid soap manufacture because moisture loss during this period can influence the physical condition and stability of the product (Imani et al., 2025). Although previous studies have investigated the physicochemical characteristics and fatty acid composition of sunflower seed oil, as well as the formulation and quality assessment of vegetable-oil-based soaps, these aspects have frequently been examined as separate areas of investigation. There remains value in an integrated assessment that connects the characteristics of a particular oil sample with its application in soap production and the quality of the resulting product. The contribution of the present study lies in combining Soxhlet extraction, physicochemical and fatty acid characterisation of sunflower seed oil, cold-process soap formulation based on the

experimentally determined saponification value, and comparative evaluation of the formulated soap against a commercial bathing soap. The assessment of selected functional properties and changes during curing further extends the investigation beyond basic oil characterisation and soap formulation to consider the quality attributes and behaviour of the finished product.
Accordingly, this study investigated the physicochemical characteristics and fatty acid composition of Soxhlet-extracted sunflower (*Helianthus annuus* L.) seed oil and evaluated its application in cold-process soap production. It further assessed the physicochemical and selected functional properties of the formulated soap in comparison with a commercial bathing soap and examined changes in selected characteristics during curing. The study provides an integrated evaluation of the relationship between sunflower seed oil characteristics, its suitability for soap formulation and the quality properties of the resulting soap.

2. Materials and Methods

2.1 Research Design

The study adopted an experimental laboratory-based design to investigate the physicochemical properties and fatty acid composition of sunflower (*Helianthus annuus* L.) seed oil extracted by Soxhlet extraction and evaluate its suitability for cold-process soap production. The investigation was conducted in five principal stages: collection and preparation of sunflower seeds; extraction and physicochemical characterization of the seed oil; determination of the fatty acid composition of the extracted oil using gas chromatography–flame ionization detection (GC-FID); production of soap using the extracted oil as the sole lipid source; and comparative evaluation of the physicochemical and selected functional properties of the formulated soap against commercially available Viva bathing soap.
Three independent seed-oil extraction batches were prepared to assess the reproducibility of the extraction process. Each extraction batch subsequently provided the oil required for the preparation of a corresponding independent soap batch. The formulated soap samples were evaluated alongside commercial Viva bathing soap under identical analytical conditions wherever the methods were applicable. Laboratory determinations were performed in triplicate, while the independent extraction and soap-production batches constituted the principal experimental units for statistical analysis.

2.2 Study Area and Source of Plant Materials

Sunflower seeds were obtained from a local farm in Oko, Orumba North Local Government Area, Anambra State, Nigeria. The seeds were collected on 27 November 2025. Collection was restricted to mature, apparently healthy seeds without visible evidence of fungal growth, insect infestation, mechanical damage or deterioration. The collected seeds were placed in clean, dry and appropriately labelled sample containers and transported to the laboratory for processing.
The seeds were identified as *Helianthus annuus* L. and authenticated by Dr. S. I. Okeke of the Department of Biology, Federal Polytechnic Oko, Anambra State, Nigeria. The authentication reference number was FPO/25/04/178.

2.3 Materials, Reagents and Equipment

The principal materials used for the study included mature sunflower seeds, analytical-grade n-hexane, sodium hydroxide pellets, ethanol, methanol, potassium hydroxide, hydrochloric acid, glacial acetic acid, chloroform, potassium iodide, sodium thiosulphate, starch indicator, phenolphthalein indicator, Wijs reagent and other analytical reagents required for the determination of the physicochemical properties of the oil and soap. Analytical-grade reagents and distilled or deionised water were used throughout the investigation.

The major equipment used included a Soxhlet extraction apparatus fitted with an appropriate extraction flask and condenser, heating mantle, rotary evaporator, analytical weighing balance, laboratory oven, desiccator, grinder, sieve, thermometer, pH meter, burettes, pipettes, conical flasks, volumetric flasks, measuring cylinders, beakers, glass rods and other standard laboratory glassware. Soap production was carried out using a heat-resistant mixing vessel, a suitable mechanical or immersion mixer, soap moulds, a thermometer and a ventilated curing rack. Fatty acid composition was determined using a gas chromatograph equipped with a flame ionization detector and a suitable capillary column. The GC-FID analysis was performed using an Agilent J&W HP-88 capillary column, manufacturer part number 112-8867, with a length of 60 m, internal diameter of 0.25 mm and stationery-phase film thickness of 0.20 µm. The HP-88 stationary phase is a highly polar cyanopropyl-containing phase designed for the separation of fatty acid methyl esters, including cis/trans fatty acid isomers. The column specification was reported separately from the GC instrument model and detector configuration.

2.4 Experimental Replication and Sample Identification

The extraction procedure was conducted using three independently prepared portions of sunflower seed powder, designated E1, E2 and E3. Each extraction was performed separately using freshly prepared sample material under the same general extraction conditions. The resulting oils were stored and analysed independently to assess variation between the extraction batches. Three independent soap batches, designated S1, S2 and S3, were prepared using the oils obtained from extraction batches E1, E2 and E3, respectively. Each soap batch was produced using 250 g of sunflower seed oil, giving a total oil requirement of 750 g for soap production. Additional quantities of the extracted oil were reserved for physicochemical characterization and GC-FID analysis. The sunflower seeds collected provided sufficient oil for the planned soap production and subsequent analytical determinations.

Three independently manufactured batches of commercial Viva bathing soap were purchased from retail outlets and designated V1, V2 and V3. The product variant, manufacturer, declared ingredients, net weight, batch or lot numbers, manufacturing dates, expiry dates and purchase details were documented. Each commercial soap batch was analysed independently alongside the formulated sunflower oil soap samples under the same analytical conditions wherever applicable. The three commercial product lots provided independent experimental units for evaluating variation in the physicochemical and functional properties of the commercial soap.

2.5 Preparation of Sunflower Seeds

The collected sunflower seeds were sorted manually to remove stones, plant debris, damaged seeds, foreign materials and visibly deteriorated specimens. The cleaned seeds were spread in a clean drying tray and dried in a laboratory oven at 50°C to reduce moisture and improve their suitability for solvent extraction. Drying continued until the samples attained a reasonably stable mass. The dried seeds were allowed to cool in a desiccator before weighing.

The dried seeds were ground into a relatively uniform powder using a clean laboratory grinder. The powdered material was passed through a suitable sieve to obtain a more homogeneous particle size and improve solvent contact during extraction. The prepared seed powder was transferred into clean, airtight containers and protected from moisture, excessive heat and direct sunlight until extraction. The moisture content of the prepared sunflower seed powder was determined gravimetrically using an oven-drying procedure. A 5.00 g portion of each seed powder batch (E1, E2 and E3) was accurately weighed into a previously dried and weighed moisture dish and heated in an oven until a constant mass was obtained. After drying, the dishes

were cooled in a desiccator before the final weighing. Moisture content was calculated as the percentage loss in mass relative to the initial sample mass using the following equation:

$$\text{Moisture content (\%)} = \frac{W_i - W_f}{W_i} \times 100$$

where (W_i) represents the initial mass of the seed sample before drying and and (W_f) represents the final mass after drying. The moisture content determination provided the basis for interpreting the extraction yield and expressing the oil yield on a dry-matter basis where necessary. The sunflower seed powder recorded a mean moisture content of 8.80 ± 0.20%.

2.6 Soxhlet Extraction of Sunflower Seed Oil

Sunflower seed oil was extracted using the Soxhlet solvent-extraction method, with analytical-grade n-hexane serving as the extraction solvent. For each extraction batch, 100.00 g of the prepared sunflower seed powder was accurately weighed and transferred into a cellulose extraction thimble. The loaded thimble was placed in the Soxhlet extractor, which was connected to a round-bottom extraction flask containing 100 mL of analytical-grade n-hexane. The apparatus was assembled with a properly fitted condenser, and the solvent was heated using a heating mantle under controlled conditions to facilitate continuous reflux and siphoning during extraction. The extraction was conducted for 6 hours for each batch, ensuring continuous solvent circulation and adequate contact between the solvent and the seed material. The procedure was performed independently for the three sample batches (E1, E2 and E3) under the same experimental conditions. At the end of each extraction, the solvent containing the dissolved oil was recovered from the extraction flask. The n-hexane was removed using a rotary evaporator operated at 40°C under reduced pressure to minimise thermal degradation of the extracted oil. The recovered oil was transferred into a clean, dry, pre-weighed amber glass bottle. The bottle containing the oil was weighed to determine the mass of recovered oil. The extracted oil was subsequently sealed and stored under refrigeration, protected from light and unnecessary exposure to air, until further analysis. All extraction procedures were conducted in a well-ventilated laboratory or fume hood because n-hexane is volatile and highly flammable. The solvent was kept away from naked flames, sparks and other ignition sources.

2.7 Determination of Oil Extraction Yield

The extraction yield was calculated for each independent extraction batch by expressing the mass of recovered oil as a percentage of the initial mass of sunflower seed powder used for extraction. The extraction yield was calculated using the following equation:

$$\text{Oil yield (\%)} = \frac{\text{Weight of oil extracted}}{\text{Initial weight of sunflower seeds}} \times 100$$

To account for differences in the moisture content of the seed powder, the oil yield was also expressed on a dry-matter basis. The dry matter content of each seed powder batch was determined by subtracting the moisture content from the initial sample mass. The dry-basis extraction yield was calculated using the following equation:

$$\text{Dry-basis oil yield (\%)} = \frac{\text{Mass of recovered oil}}{\text{Mass of seed sample} \times (1 - M/100)} \times 100$$

where (M) represents the moisture content of the seed powder expressed as a percentage. The extraction yields were calculated separately for the three independent extraction batches (E1, E2 and E3). The results were expressed as mean ± standard deviation (SD) of the three independent extraction batches.

2.8 Physicochemical Characterization of Sunflower Seed Oil

The extracted sunflower seed oil was subjected to physicochemical analysis to determine its quality and suitability for soap production. The parameters analysed included moisture and volatile matter content, acid value, free fatty acid content, saponification value, iodine value and peroxide value. The analytical procedures followed the relevant International Organization for Standardization (ISO) methods or validated equivalent procedures. All determinations were performed using appropriately prepared reagents, reagent blanks and replicate measurements.

2.8.1 Moisture and Volatile Matter Content

The moisture and volatile matter content of the extracted sunflower seed oil was determined gravimetrically. A clean, dry weighing dish was first heated, cooled in a desiccator and weighed. A 5.00 g portion of the oil sample was then transferred into the dish, and the combined mass was recorded. The dish containing the oil sample was placed in an oven maintained at 103 ± 2 °C and heated for an initial period of 1 hour. After the initial heating, the dish was removed from the oven, cooled in a desiccator and reweighed. Subsequent heating was carried out in 30-minute intervals, with cooling and weighing after each interval, until a constant mass was obtained.

The moisture and volatile matter content was calculated using the following equation:

$$\text{Moisture and volatile matter (\%)} = \frac{W_1 - W_2}{W_1 - W_0} \times 100$$

where (W_0) represented the mass of the empty weighing dish, (W_1) represented the mass of the dish and oil sample before heating, and (W_2) represented the mass of the dish and sample after heating to constant mass. The result was expressed as a percentage of the initial oil sample mass. It was reported as moisture and volatile matter rather than moisture alone because the gravimetric loss during heating could include other volatile components.

2.8.2 Determination of Acid Value

The acid value of the sunflower seed oil was determined by titration in accordance with ISO 660:2020. A 5.00 g portion of the oil sample was weighed into a clean conical flask and dissolved in a neutralised solvent mixture consisting of ethanol and diethyl ether mixed in a 1:1 (v/v) ratio. Phenolphthalein indicator was added, and the resulting solution was titrated with 0.1 M standardised potassium hydroxide (KOH) solution while being continuously mixed. The titration endpoint was indicated by the appearance of a faint pink colour that persisted for 30 seconds. A reagent blank was analysed under the same conditions to account for any acidity contributed by the reagents and solvent mixture.

The acid value was calculated using the following equation:

$$AV = \frac{(V_s - V_b) \times M \times 56.1}{W}$$

where (Vs) represented the volume of KOH solution used for the sample titration (mL), (Vb) represented the volume of KOH solution used for the blank titration (mL), (M) represented the molarity of the KOH solution (mol/L), 56.1 represented the molar mass of potassium hydroxide (g/mol), and (W) represented the mass of the oil sample analysed (g). The acid value was expressed as milligrams of potassium hydroxide required to neutralise the acidic constituents in one gram of oil. It provided an indication of the amount of acidic substances, principally free fatty acids, present in the sunflower seed oil.

2.8.3 Determination of Free Fatty Acid Content

The free fatty acid (FFA) content of the sunflower seed oil was calculated from the experimentally determined acid value and expressed as a percentage of oleic acid equivalent. The calculation was based on the conventional conversion factor of 0.503, as shown below:

$$\text{FFA (\% as oleic acid)} = AV \times 0.503$$

where (AV) represented the acid value of the oil expressed in milligrams of potassium hydroxide per gram of oil (mg KOH/g). The calculated FFA content was reported as a percentage of oleic acid equivalent. This parameter was used alongside the acid value to assess the hydrolytic condition and quality of the extracted sunflower seed oil.

2.8.4 Determination of Saponification Value

The saponification value of the sunflower seed oil was determined according to ISO 3657:2023 with modification. A 5.00 g portion of the oil sample was weighed into a suitable reflux flask and treated with 50 mL of 0.5 M alcoholic potassium hydroxide (KOH) solution. The mixture was heated under reflux for one hour to allow the triglycerides and other saponifiable constituents in the oil to react with the alkali. After refluxing, the remaining unreacted KOH was titrated with 0.5 M hydrochloric acid (HCl) solution using an appropriate indicator. A blank determination containing the same reagents, but without the oil sample, was carried out simultaneously under the same conditions.

The saponification value was calculated using the following equation:

$$SV = \frac{(V_b - V_s) \times M \times 56.1}{W}$$

where (Vb) represented the volume of HCl solution used for the blank titration (mL), (Vs) represented the volume of HCl solution used for the sample titration (mL), (M) represented the molarity of the HCl solution (mol/L), 56.1 represented the molar mass of potassium hydroxide (g/mol), and (W) represented the mass of the oil sample analysed (g). The saponification value was expressed as milligrams of potassium hydroxide required to saponify one gram of oil. The determined value was subsequently used to calculate the theoretical quantity of sodium hydroxide required for the soap production process.

2.8.5 Determination of Iodine Value

The iodine value of the sunflower seed oil was determined using the Wijs method in accordance with ISO 3961:2024. A 5.00 g portion of the oil sample was weighed into a clean, dry iodine flask and dissolved in 20 mL of a solvent mixture consisting of glacial acetic acid and cyclohexane mixed in a 1:1 (v/v) ratio. Subsequently, 50 mL of Wijs reagent was added to the solution, and the flask was stoppered, mixed thoroughly and kept in the dark for 2 hours to allow the reaction to proceed. At the end of the reaction period, 20 mL of 10% potassium iodide (KI) solution was added, followed by distilled water, to liberate iodine from the unreacted halogen reagent. The liberated iodine was titrated with 0.1 N standardised sodium thiosulphate solution, with freshly prepared starch indicator added near the endpoint. The titration was continued until the blue-black colour disappeared. A blank determination containing the same reagents, but without the oil sample, was carried out simultaneously under the same conditions.

The iodine value was calculated using the following equation:

$$IV = \frac{(V_b - V_s) \times N \times 12.69}{W}$$

where (Vb) represented the volume of sodium thiosulphate solution used for the blank titration (mL), (Vs) represented the volume used for the sample titration (mL), (N) represented the normality of the sodium thiosulphate solution, 12.69 represented the iodine value conversion factor, and (W) represented the mass of the oil sample analysed (g). The iodine value was

expressed as grams of iodine absorbed per 100 g of oil and was used to estimate the degree of unsaturation of the sunflower seed oil.

2.8.6 Determination of Peroxide Value

The peroxide value of the sunflower seed oil was determined by an iodometric procedure in accordance with ISO 3960:2017. A 5.00 g portion of the oil sample was weighed into a suitable conical flask and dissolved in 30 mL of a solvent mixture consisting of glacial acetic acid and chloroform mixed in a 3:2 (v/v) ratio. Subsequently, 0.5 mL of saturated potassium iodide (KI) solution was added, and the mixture was allowed to react in the dark for exactly 1 minute. Distilled water was then added, and the liberated iodine was titrated with 0.01 N standardised sodium thiosulphate solution. Freshly prepared starch indicator was introduced near the titration endpoint, and titration was continued until the blue colour disappeared. A blank determination containing the same reagents, but without the oil sample, was performed simultaneously under the same conditions.

The peroxide value was calculated using the following equation:

$$PV = \frac{(V_s - V_b) \times N \times 1000}{W}$$

where (Vs) represented the volume of sodium thiosulphate solution used for the sample titration (mL), (Vb) represented the volume used for the blank titration (mL), (N) represented the normality of the sodium thiosulphate solution, and (W) represented the mass of the oil sample analysed (g). The peroxide value was expressed as milliequivalents of active oxygen per kilogram of oil (meq O_2/kg) and was used to assess the extent of primary lipid oxidation in the extracted sunflower seed oil.

2.9 Preparation of Fatty Acid Methyl Esters and GC-FID Analysis

The fatty acid composition of the extracted sunflower seed oil was determined by gas chromatography–flame ionization detection (GC-FID) following conversion of the fatty acids into their corresponding fatty acid methyl esters (FAMEs). The methylation procedure followed ISO 12966-2:2017. A 50 mg portion of the extracted sunflower seed oil was used for FAME preparation. The oil sample was treated with 2 M methanolic potassium hydroxide (KOH) and heated at 65 °C for 10 minutes to facilitate the conversion of the fatty acids into their corresponding methyl esters. Following methylation, the resulting FAME fraction was extracted using n-hexane and transferred into clean GC vials for chromatographic analysis.

Chromatographic separation was performed using a GC-FID system fitted with an Agilent J&W HP-88 capillary column, part number 112-8867, with dimensions of 60 m × 0.25 mm internal diameter × 0.20 µm film thickness. The HP-88 column contained a highly polar cyanopropyl-containing stationary phase designed for the separation of fatty acid methyl esters, particularly cis/trans isomers. This column specification was distinct from DB-FFAP and CP-Wax 58 FFAP columns, which have different stationary-phase chemistries and specifications.

The GC-FID oven temperature programme began at 125 °C, followed by an increase to 145 °C at a rate of 8 °C/min, where the temperature was maintained for 26 minutes. The oven temperature was subsequently increased to 220 °C at 2 °C/min and maintained for 1 minute. Helium was used as the carrier gas at a constant flow rate of 1.4 mL/min. The injector temperature was maintained at 250 °C, while the detector temperature was set at 260 °C. A 1 µL aliquot of the FAME solution was injected using split injection mode at a split ratio of 50:1. Individual fatty acids were identified by comparing their chromatographic retention times with those of authenticated FAME reference standards analysed under the same chromatographic conditions. A certified multi-component FAME reference mixture was used to support peak identification and retention-time assignment. Quantification was based on the relative peak

areas of the identified fatty acid methyl esters, with appropriate correction factors applied where required by the adopted analytical method. The concentration of each fatty acid was expressed as a percentage of the total identified fatty acids. The fatty acids evaluated included palmitic acid, stearic acid, oleic acid, linoleic acid and other fatty acids detected in the sample. The chromatograms, retention times, peak areas and identification records were retained as part of the analytical documentation.

2.10 Calculation of Sodium Hydroxide Requirement for Soap Production

The quantity of sodium hydroxide (NaOH) required for soap production was calculated from the experimentally determined saponification value of the extracted sunflower seed oil rather than from an assumed or literature-derived value. The calculation was based on the saponification value of 189.34 mg KOH/g oil and an oil mass of 250 g for each soap batch. The theoretical NaOH requirement was calculated using the following equation:

$$NaOH_{\text{theoretical}} = \frac{SV \times W_o \times 40.00}{56.11 \times 1000}$$

where $NaOH_{\text{theoretical}}$ represented the theoretical mass of sodium hydroxide required (g), (SV) represented the experimentally determined saponification value (mg KOH/g oil), (W_o) represented the mass of oil used (g), 40.00 represented the molecular mass of sodium hydroxide (g/mol), and 56.11 represented the molecular mass of potassium hydroxide (g/mol). Based on the measured saponification value and 250 g oil charge, the theoretical NaOH requirement was calculated as approximately 33.75 g per soap batch.

A superfatting level of 5% was applied by reducing the theoretical NaOH requirement by 5%. The adjusted NaOH requirement was calculated using:

$$NaOH_{\text{working}} = NaOH_{\text{theoretical}} \times 0.95$$

This gave an active NaOH requirement of approximately 32.06 g per soap batch. Since the analytical-grade sodium hydroxide reagent had a stated purity of 98%, the actual mass of NaOH pellets required was corrected for reagent purity using:

$$\boxed{m_{\text{NaOH,weighed}} = \frac{m_{\text{NaOH,adjusted}}}{P/100}}$$

where $m_{\text{NaOH,weighed}}$ represented the mass of sodium hydroxide pellets weighed (g), and (P) represented the percentage purity of the reagent. Using a purity of 98%, approximately 32.71 g of NaOH pellets was weighed for each 250 g oil batch.

The NaOH solution was prepared at a nominal concentration of 30% (w/w), based on the active NaOH requirement. The quantity of water required was calculated using:

$$W_{\text{water}} = \frac{m_{\text{NaOH,adjusted}} \times (100 - C)}{C}$$

where W_{water} represented the mass of water required (g), $m_{\text{NaOH,adjusted}}$ represented the active NaOH requirement (g), and (C) represented the target NaOH concentration expressed as a percentage. A recorded quantity of 74.16 g of distilled water was used for each batch. Based on the active NaOH mass and recorded water quantity, the resulting active NaOH concentration was approximately 30.18% (w/w).

The calculations were applied to each of the three independent soap batches (S1, S2 and S3), each containing 250 g of sunflower seed oil. The same formulation quantities were used for the three batches to maintain consistency during soap production.

2.11 Production of Sunflower Oil Soap by the Cold-Process Method

The sunflower oil soap was produced using the cold-process saponification method, with sunflower seed oil serving as the sole lipid source. Three independent soap batches (S1, S2 and

S3) were prepared, each containing 250 g of the corresponding extracted sunflower seed oil. The quantity of sodium hydroxide required for each batch was calculated from the experimentally determined saponification value of 189.34 mg KOH/g oil, applying a 5% superfatting level and correcting for the 98% purity of the sodium hydroxide reagent. For each batch, 32.71 g of sodium hydroxide pellets and 74.16 g of distilled water were used. No additional vegetable oil, animal fat or other lipid was incorporated into the formulation.

The required quantity of distilled water was first measured into a heat-resistant container. The calculated sodium hydroxide pellets were gradually added to the water while stirring carefully until completely dissolved. Sodium hydroxide was added to water and not water to sodium hydroxide. The resulting lye solution was allowed to cool before mixing. Separately, 250 g of the corresponding sunflower seed oil was measured into a clean mixing vessel. The temperatures of the oil and lye solution were monitored using a laboratory thermometer before combining the two phases. For batches S1, S2 and S3, respectively, the oil temperatures were 37.0, 36.8 and 37.2 °C, while the corresponding lye solution temperatures were 36.5, 36.7 and 36.9 °C. The cooled sodium hydroxide solution was gradually introduced into the sunflower seed oil while the mixture was continuously stirred. Mixing was continued until the mixture reached trace, indicated by an increase in viscosity and the formation of a visible surface pattern after dripping. The recorded times required to reach trace were 8, 9 and 8 minutes for batches S1, S2 and S3, respectively. The soap mixtures initially exhibited a light cream colour, and no visible separation was observed during mixing. After reaching trace, each mixture was transferred into clean, dry soap moulds and covered appropriately to minimise contamination during the initial setting period. The moulded soap was allowed to remain undisturbed for approximately 24–48 hours or until sufficiently firm for removal. The soap was subsequently removed from the moulds, labelled according to its batch identification and transferred to a clean, ventilated curing rack. The bars were cured for six weeks under dry, well-ventilated conditions, away from direct sunlight, excessive heat and moisture. During curing, the mass, moisture content, pH, colour, appearance and physical condition of the soap bars were monitored at the designated assessment intervals. The bars were examined for changes in firmness, surface condition and physical stability throughout the curing period. At the end of the six-week curing period, representative portions of each soap batch were collected for physicochemical and functional evaluation.

The production procedure was documented by recording the oil mass, sodium hydroxide mass, water mass, initial oil and lye temperatures, time required to reach trace, initial appearance of the soap mixture and observations of physical separation. These records provided the basis for monitoring consistency among the three independently prepared soap batches and evaluating the quality properties of the formulated sunflower oil soap.

2.12 Physicochemical Characterization of the Formulated and Commercial Soaps

The cured sunflower oil soap samples and commercial Viva bathing soap samples were evaluated for selected physicochemical properties, including pH, moisture content, total fatty matter, free caustic alkali, chloride content and matter insoluble in ethanol. The same analytical procedures and sample preparation conditions were applied to both products wherever the methods were suitable. The commercial product's declared formulation was considered when interpreting the results, particularly where the product contained ingredients other than conventional fatty-acid sodium salts. Three independently prepared sunflower oil soap batches (S1, S2 and S3) and three independently manufactured commercial Viva soap batches (V1, V2 and V3) were evaluated.

2.12.1 Determination of Soap pH

The pH of the soap samples was determined using a calibrated digital pH meter. Each soap sample was grated or finely cut to obtain a representative portion. A 1.00 g quantity of the prepared soap was weighed and dissolved in distilled water, and the solution was made up to a final volume of 100 mL to obtain a 1% (w/v) soap solution. The solution was mixed thoroughly and allowed to equilibrate at approximately 25 °C before measurement.

The pH meter was calibrated using standard buffer solutions of pH 4.00, 7.00 and 10.00 in accordance with the manufacturer's instructions. The electrode was rinsed with distilled water between measurements and gently blotted to remove excess water. It was subsequently immersed in the prepared soap solution, and the pH value was recorded after the reading had stabilised. The measurement was performed in triplicate for each independent soap sample. The results were reported as the pH of a 1% (w/v) aqueous soap solution at approximately 25 °C rather than as the pH of the dry soap bar.

2.12.2 Determination of Moisture Content

The moisture content of the soap samples was determined gravimetrically using an oven-drying procedure in accordance with ISO 672:1978. A clean, dry moisture dish was first weighed, after which approximately 5.00 g of finely grated soap was transferred into the dish and accurately weighed. The sample was dried in a laboratory oven temperature of 103 ± 2°. After drying, the dish was transferred into a desiccator, allowed to cool and reweighed. The drying, cooling and weighing process was repeated until a constant mass was obtained.

The moisture content was calculated using the following equation:

$$\text{Moisture content (\%)} = \frac{W_i - W_f}{W_i} \times 100$$

where (W_i) represented the initial mass of the soap sample before drying (g), and (W_f) represented the mass of the soap sample after drying to constant mass (g). The result was expressed as a percentage of the original sample mass. The same analytical procedure and drying conditions were applied to both the formulated sunflower oil soap and the commercial Viva soap samples.

2.12.3 Determination of Total Fatty Matter

The total fatty matter (TFM) content of the soap samples was determined in accordance with ISO 685:2020. A 5.00 g portion of each soap sample was weighed and subjected to treatment with sulphuric acid to decompose the soap and liberate the fatty matter. The liberated fatty matter was subsequently extracted using diethyl ether. The extraction was carried out in a separating funnel to facilitate the separation of the ether layer containing the fatty matter from the aqueous phase. The recovered fatty matter was washed, neutralised where required, and dried according to the prescribed analytical procedure before weighing.

The total fatty matter content was calculated using the procedure and correction factors specified in ISO 685:2020 and expressed as a percentage by mass of the original soap sample. The standard's specified calculation was followed rather than treating the mass of an uncorrected solvent extract as the total fatty matter value. The results were interpreted with consideration of the declared ingredients of the commercial Viva product because the presence of synthetic surfactants or other non-soap constituents could affect the comparability of TFM measurements between the formulated sunflower oil soap and the commercial product.

2.12.4 Determination of Free Caustic Alkali

The free caustic alkali content of the soap samples was determined using the titrimetric procedure specified in ISO 456:1973. A 5.00 g portion of each grated soap sample was weighed

into a suitable conical flask and dissolved in 100 mL of neutralised 95% ethanol. Phenolphthalein indicator was added to the prepared solution, which was subsequently titrated with 0.1 M sulphuric acid (H_2SO_4) while being continuously mixed. The titration endpoint was indicated by the disappearance of the pink colour. A reagent blank was analysed under the same conditions to account for any acidity contributed by the solvent and reagents.

The free caustic alkali content was calculated according to the applicable ISO 456 procedure and expressed as a percentage by mass, reported as sodium hydroxide (NaOH). The result was used to assess the amount of unreacted caustic alkali remaining in the formulated sunflower oil soap and commercial Viva soap. The pH value was not used as a substitute for this determination because pH and free caustic alkali represent different chemical properties.

2.12.5 Determination of Chloride Content

The chloride content of the soap samples was determined using the argentometric method described in ISO 457:1983. A 5.00 g portion of each grated soap sample was dissolved or dispersed in distilled water. Potassium chromate (K_2CrO_4) was added as the indicator, and the resulting solution was titrated with 0.1 N silver nitrate ($AgNO_3$) solution. The titration was continued until the appearance of a permanent reddish-brown (brick-red) precipitate, indicating the endpoint. A blank determination was also carried out under the same conditions.

The sodium chloride (NaCl) content was calculated from the volume of silver nitrate solution consumed by the sample after correction for the blank, using the following equation:

$$\text{NaCl (\%)} = \frac{(V_s - V_b) \times N \times 58.44 \times 100}{1000 \times W}$$

Where:

(V_s) = volume of silver nitrate solution used for the sample (mL);

(V_b) = volume of silver nitrate solution used for the blank (mL);

(N) = normality of silver nitrate solution (0.1 N);

58.44 = molecular weight of sodium chloride (g/mol); and

(W) = mass of soap sample analysed (g).

The results were expressed as percentage sodium chloride by mass. All determinations were performed in triplicate for each independent soap sample, and the results were reported as mean ± standard deviation.

2.12.6 Determination of Matter Insoluble in Ethanol

The matter insoluble in ethanol in the soap samples was determined in accordance with ISO 673:1981. A 5.00 g portion of finely grated soap was treated with hot ethanol under the conditions prescribed by the standard to dissolve the ethanol-soluble constituents. The resulting mixture was filtered through a previously dried and weighed filter medium or suitable filtration apparatus. The insoluble residue was washed with the prescribed quantity of hot ethanol to remove any remaining soluble materials.

The filter medium containing the insoluble residue was dried to constant mass, cooled in a desiccator and weighed. The percentage of matter insoluble in ethanol was calculated from the mass of the recovered residue relative to the initial mass of the soap sample, using the following equation:

Matter insoluble in ethanol (%) = $[(W_2 - W_1) \div W_s] \times 100$

Where:

(W1) = mass of the dried filter medium before filtration (g);

(W2) = mass of the dried filter medium containing the insoluble residue (g); and

(Ws) = initial mass of the soap sample analysed (5.00 g).

The results were expressed as percentage matter insoluble in ethanol by mass. All determinations were performed in triplicate for each independent soap sample, and the results were reported as mean ± standard deviation.

2.13 Evaluation of Selected Functional Properties of the Soaps

In addition to the physicochemical tests, the formulated sunflower oil soap and commercial Viva bathing soap were evaluated for selected functional properties, including foaming ability, foam stability, cleansing efficiency and dissolution behaviour. These tests were conducted under controlled conditions to ensure that differences in sample performance are not caused by inconsistent sample concentration, water volume, temperature or handling.

2.13.1 Determination of Foaming Ability and Foam Stability

The foaming ability of each soap sample was evaluated using a standardised cylinder-shaking procedure. A 1% (w/v) aqueous solution of each soap was prepared by dissolving 1.00 g of finely grated soap in 100 mL of distilled water. A 50 mL portion of the prepared solution was transferred into a clean 100 mL graduated measuring cylinder. The cylinder was closed and subjected to 20 uniform vertical strokes over approximately 30 seconds. Immediately after shaking, the cylinder was placed upright on a level surface, and the initial foam height was recorded.

Foam stability was evaluated by recording the foam height at 5, 10 and 15 minutes after the initial measurement. The initial foam height was used as an indicator of foaming ability, while the percentage of foam retained at each observation time was used to assess foam stability. Foam retention was calculated using the following equation:

$$\text{Foam retention (\%)} = \frac{H_t}{H_0} \times 100$$

where (H_0) is the initial foam height and (H_t) is the foam height at the specified observation time. The procedure was performed in triplicate for each independent soap sample under the same experimental conditions. The results were expressed as mean ± standard deviation.

2.13.2 Evaluation of Cleansing Efficiency

The cleansing efficiency of the formulated sunflower oil soap and commercial Viva bathing soap was evaluated using a gravimetric artificial-soil removal procedure. Clean, dry and uniformly sized stainless-steel substrates were weighed before the application of an oily soil mixture. Each substrate was subsequently weighed after contamination to determine the initial mass of the applied soil. Aqueous solutions of the formulated and commercial soaps were prepared at a fixed concentration. The artificially contaminated substrates were washed separately using the respective soap solutions under controlled conditions. The washing procedure was maintained consistently across the samples to minimise variations arising from differences in washing time, water volume, temperature and mechanical agitation.

Following washing, the substrates were rinsed with distilled water, dried to constant mass and reweighed. A water-only washing control was included to assess the contribution of water and mechanical washing to soil removal. Unsoiled substrates were also weighed to establish the reference mass of the clean substrates. Cleansing efficiency was calculated as the percentage of the initially applied soil removed during washing, using the following equation:

$$\text{Cleansing efficiency (\%)} = \frac{W_i - W_f}{W_i - W_c} \times 100$$

where (Wi) is the mass of the contaminated substrate before washing, (Wf) is the mass of the substrate after washing and drying, and (Wc) is the mass of the clean substrate. The procedure

was applied consistently to the formulated sunflower oil soap and commercial Viva soap. The results were expressed as mean ± standard deviation.

2.13.3 Determination of Soap Dissolution Behaviour

The dissolution behaviour of the formulated sunflower oil soap and commercial Viva bathing soap was evaluated by determining the percentage mass loss of the soap specimens following exposure to distilled water. Uniformly sized specimens were prepared from each soap sample and dried under controlled conditions to obtain stable initial masses. Each specimen was weighed before immersion, and the initial dry mass was recorded.

The soap specimens were individually immersed in a defined volume of distilled water maintained under controlled temperature conditions. The specimens were exposed to water for a predetermined period under identical experimental conditions to ensure consistency between the two soap products. At the end of the exposure period, the specimens were removed from the water, allowed to drain and dried under the same conditions used for the initial weighing until stable final masses were obtained. A non-immersed drying control was maintained to account for any mass changes resulting from drying alone.

The dissolution-related mass loss was calculated using the following equation:

$$\text{Dissolution-related mass loss (\%)} = \frac{W_i - W_f}{W_i} \times 100$$

where (W_i) is the initial dry mass of the soap specimen and (W_f) is its final dry mass after immersion and redrying. The procedure was applied consistently to all soap samples, and the results were expressed as mean ± standard deviation. The measured mass loss was interpreted as dissolution-related mass loss under the experimental conditions rather than as an absolute measure of the solubility of the soap.

2.14 Assessment of Storage Stability During Curing and Post-Curing

The physical stability of the formulated sunflower oil soap was monitored throughout the six-week curing period. Observations and measurements were recorded at the beginning of curing (week 0) and subsequently at weeks 2, 4 and 6. The assessment involved monitoring changes in the physical appearance, colour, firmness, surface condition and mass of the soap samples.

At each observation interval, the soap bars were examined for visible changes, including cracking, oil separation, deformation and other signs of physical deterioration. The mass of representative soap bars was recorded at the designated intervals to monitor mass loss during curing. Moisture content and pH were also determined using the analytical procedures described in the preceding sections to assess changes in these properties throughout the curing period. The pH measurements were performed on the three independently produced soap batches (S1, S2 and S3) at weeks 0, 2, 4 and 6. The moisture content and relative mass of the soap bars were similarly monitored over the six-week period. All samples were maintained under comparable curing and storage conditions throughout the assessment.

The stability of the formulated soap was evaluated based on the recorded physical observations and changes in mass, moisture content and pH during curing. The measurements were used to assess the progression of soap hardening and moisture loss, as well as the physical condition of the bars at the end of the six-week curing period. Instrumental hardness or penetration measurements were not included in the assessment.

2.15 Statistical Analysis

All experimental data were organised in Microsoft Excel and analysed using R statistical software, version 4.6.1. Results were presented as mean ± standard deviation (SD). The three independent oil extraction batches (E1, E2 and E3), corresponding soap-production batches

(S1, S2 and S3), and three independently sourced commercial Viva soap lots (V1, V2 and V3) were treated as the respective experimental units. Technical replicate measurements were averaged within each independent experimental unit before inferential statistical analysis to avoid pseudoreplication. Differences in the physicochemical and functional properties of the formulated sunflower oil soap and commercial Viva soap were evaluated using Welch's independent-samples t-test. This test was applied to account for potential differences in variance between the two product groups. To control the family-wise error rate arising from multiple comparisons, the resulting p-values were adjusted using the Holm correction procedure. Mean differences, corresponding 95% confidence intervals and adjusted p-values were reported to describe the magnitude, precision and statistical significance of the observed differences. Changes in the pH of the formulated sunflower oil soap during the six-week curing period were evaluated using the Friedman test for repeated measurements. Measurements recorded at weeks 0, 2, 4 and 6 for the three independently produced soap batches were used to assess changes in pH over time. The Friedman chi-square statistic, degrees of freedom, p-value and Kendall's coefficient of concordance (W) were reported to describe the statistical outcome and the magnitude of the observed repeated-measures effect.

The fatty acid composition of the extracted sunflower seed oil was summarised using the relative percentage of each identified fatty acid obtained from GC-FID analysis. Physicochemical characteristics of the extracted oil, soap quality parameters, functional properties and curing-related measurements were presented using appropriate descriptive statistics. Statistical significance was assessed at a probability level of $p < 0.05$. Exact p-values, adjusted p-values, effect estimates and 95% confidence intervals were reported where applicable.

2.16 Ethical, Environmental and Laboratory Safety Considerations

The study involved the use of plant materials and laboratory chemical procedures and did not involve human participants or experimental animals. The sunflower seeds were obtained from a local farm in Oko, Orumba North Local Government Area, Anambra State, Nigeria, with appropriate permission from the source. The experimental procedures were conducted in accordance with laboratory safety requirements and good laboratory practice.

Personal protective equipment (PPE), including laboratory coats, protective gloves and safety goggles, was used during sample preparation, solvent extraction, titrimetric analyses, GC-FID sample preparation and soap production. Particular care was taken when handling n-hexane because of its volatility, flammability and potential health hazards. Solvent extraction procedures involving n-hexane were conducted in a well-ventilated laboratory or fume hood to minimise inhalation exposure and reduce fire risks.

Sodium hydroxide was handled with appropriate precautions because of its corrosive nature and the heat generated during dissolution in water. The sodium hydroxide solution used for soap production was prepared by gradually adding sodium hydroxide pellets to water while allowing the solution to cool. Direct contact with the alkaline solution was avoided, and appropriate protective equipment was maintained throughout soap preparation.

Other laboratory chemicals, including methanol, chloroform, hydrochloric acid, glacial acetic acid and iodine-containing reagents, were handled carefully in accordance with their chemical hazards and relevant laboratory safety procedures. Procedures involving volatile or hazardous solvents were carried out under appropriate ventilation.

Chemical wastes, including organic solvents, acidic and alkaline solutions, and other chemical residues, were collected in appropriately labelled containers for proper disposal in accordance with institutional laboratory waste-management procedures. Organic solvents and other

hazardous chemical residues were not discharged directly into laboratory sinks or the surrounding environment.

All experimental activities were conducted with due consideration for personnel safety, environmental protection, proper chemical handling and the reliability of the research findings.

3. Results

3.1. Moisture content and extraction yield

The moisture content of the sunflower seed powder and the oil extraction yields obtained through Soxhlet extraction are presented in Table 1. The seed powder had a mean moisture content of 8.80 ± 0.20%. The mean oil extraction yield was 42.80 ± 0.30% on an as-received basis and 46.93 ± 0.43% on a dry-matter basis. These results were obtained from three independent extraction batches (n = 3).

Table 1. Moisture content of sunflower seed powder and Soxhlet extraction yield

Parameter	Mean ± SD (%)
Moisture content of seed powder	8.80 ± 0.20
Oil extraction yield (as-received basis)	42.80 ± 0.30
Oil extraction yield (dry-matter basis)	46.93 ± 0.43

Results expressed as mean ± standard deviation (SD), n = 3 independent extraction batches.

3.2. Physicochemical properties of extracted sunflower oil

The physicochemical properties of the Soxhlet-extracted sunflower (*Helianthus annuus* L.) seed oil are presented in Table 2. The oil had a mean moisture and volatile matter content of 0.19 ± 0.01%, an acid value of 0.73 ± 0.02 mg KOH/g, and a free fatty acid content of 0.37 ± 0.01% expressed as oleic acid. The mean saponification value was 189.34 ± 0.25 mg KOH/g, while the iodine value was 128.73 ± 0.35 g I_2/100 g. The peroxide value was 2.20 ± 0.10 meq O_2/kg. All values represent the mean ± standard deviation of three independent extraction batches (n = 3).

Table 2. Physicochemical properties of Soxhlet-extracted sunflower seed oil

Physicochemical parameter	Mean ± SD
Moisture and volatile matter (%)	0.19 ± 0.01
Acid value (mg KOH/g)	0.73 ± 0.02
Free fatty acid (% oleic acid)	0.37 ± 0.01
Saponification value (mg KOH/g)	189.34 ± 0.25
Iodine value (g I_2/100 g)	128.73 ± 0.35
Peroxide value (meq O_2/kg)	2.20 ± 0.10

Results expressed as mean ± standard deviation (SD), n = 3 independent extraction batches.

3.3. Fatty acid composition of sunflower oil

The fatty acid composition of the Soxhlet-extracted sunflower (*Helianthus annuus* L.) seed oil, as determined by gas chromatography–flame ionization detection (GC-FID), is presented in Table 3. Linoleic acid (C18:2) was the predominant fatty acid, accounting for 62.10% of the total composition, followed by oleic acid (C18:1) at 25.40%. Palmitic acid (C16:0) and stearic acid (C18:0) accounted for 6.20% and 3.80%, respectively. Other identified components included linolenic acid (0.20%), arachidic acid (0.30%), behenic acid (0.20%) and other identified fatty acids (1.80%). The total reported fatty acid composition was 100.00%.

Table 3. Fatty acid composition of Soxhlet-extracted sunflower (*Helianthus annuus* L.) seed oil

Fatty acid	Notation	Relative composition (%)
Palmitic acid	C16:0	6.20
Stearic acid	C18:0	3.80
Oleic acid	C18:1	25.40
Linoleic acid	C18:2	62.10
Linolenic acid	C18:3	0.20
Arachidic acid	C20:0	0.30
Behenic acid	C22:0	0.20
Other identified fatty acids	-	1.80
Total reported fatty acid composition	-	100.00

GC-FID results; relative composition expressed as percentage (%).

3.4. Sodium hydroxide formulation calculations

The sodium hydroxide and water quantities calculated and used for the formulation of sunflower oil soap are presented in Table 4. Based on the measured saponification value of 189.34 mg KOH/g oil, the theoretical sodium hydroxide requirement for 250 g of sunflower oil was 33.75 g. Following the application of a 5% superfatting allowance, the active sodium hydroxide requirement was reduced to 32.06 g. Considering the reagent purity of 98%, 32.71 g of commercial sodium hydroxide was weighed for each batch. A water quantity of 74.16 g was used, giving a calculated active sodium hydroxide concentration of 30.18% (w/w).

Table 4. Sodium hydroxide and water quantities used in the formulation of sunflower oil soap

Formulation parameter	Quantity
Sunflower oil per batch (g)	250.00
Measured saponification value (mg KOH/g)	189.34
Theoretical NaOH requirement (g)	33.75
NaOH after 5% superfatting (g)	32.06
NaOH reagent purity (%)	98
Commercial NaOH weighed (g)	32.71
Water actually used (g)	74.16
Calculated active NaOH concentration (% w/w)	30.18

Note: The water quantity represents the actual experimental amount used during soap production. The calculated active NaOH concentration was derived from the recorded quantities.

3.5. Production conditions and observations

The production conditions and physical observations recorded for the three formulated sunflower oil soap batches (S1, S2 and S3) are presented in Table 5. Each batch was produced using 250.00 g of sunflower oil, 32.71 g of commercial sodium hydroxide reagent and 74.16 g of water. The oil temperatures during production ranged from 36.8 to 37.2°C, while the corresponding lye temperatures ranged from 36.5 to 36.9°C. The time required to reach trace was 8 minutes for S1 and S3 and 9 minutes for S2. All three batches exhibited a light cream initial appearance, with no visible separation recorded during production.

Table 5. Production conditions and physical observations of formulated sunflower oil soap

Production parameter	S1	S2	S3
Sunflower oil (g)	250.0	250.0	250.0
NaOH reagent (g)	32.71	32.71	32.71
Water (g)	74.16	74.16	74.16
Oil temperature (°C)	37.0	36.8	37.2
Lye temperature (°C)	36.5	36.7	36.9
Time to trace (min)	8	9	8
Initial appearance	Light cream	Light cream	Light cream
Physical observation	No visible separation	No visible separation	No visible separation

Note: The quantities of sunflower oil, sodium hydroxide reagent and water represent the actual amounts used during soap production. Temperatures and time to trace are the recorded experimental observations.

3.6. Comparative physicochemical properties of formulated and commercial soaps

The comparative physicochemical properties of the formulated sunflower oil soap and commercial Viva soap are presented in Table 6. The formulated sunflower soap recorded a mean pH of 9.76 ± 0.05, compared with 9.51 ± 0.04 for Viva soap, while its moisture content was 14.00 ± 0.20%, compared with 11.00 ± 0.20% for Viva soap. The total fatty matter content was 76.17 ± 0.35% in the formulated soap and 78.70 ± 0.30% in Viva soap. The free caustic alkali content was 0.043 ± 0.006% and 0.023 ± 0.006%, respectively. Chloride content was 0.337 ± 0.015% in the formulated soap and 0.493 ± 0.015% in Viva soap, while matter insoluble in ethanol was 0.433 ± 0.015% and 0.600 ± 0.020%, respectively. Welch's independent-samples t-tests revealed statistically significant differences between the two products across all six physicochemical parameters following Holm adjustment for multiple comparisons (adjusted $p < 0.05$).

Table 6. Comparative physicochemical properties of formulated sunflower oil soap and commercial Viva soap

Physicochemical parameter	Sunflower soap	Viva soap	Mean difference	95% CI	Holm-adjusted p-value
pH	9.76 ± 0.05	9.51 ± 0.04	0.25	0.156–0.344	0.0041
Moisture content (%)	14.00 ± 0.20	11.00 ± 0.20	3.00	2.55–3.45	0.0003
Total fatty matter (%)	76.17 ± 0.35	78.70 ± 0.30	-2.53	-3.28 to -1.79	0.0023
Free caustic alkali (%)	0.043 ± 0.006	0.023 ± 0.006	0.020	0.0069–0.0331	0.0132
Chloride (%)	0.337 ± 0.015	0.493 ± 0.015	-0.157	-0.191 to -0.122	0.0012
Matter insoluble in ethanol (%)	0.433 ± 0.015	0.600 ± 0.020	-0.167	-0.208 to -0.125	0.0019

Values are presented as mean ± standard deviation (SD), n = 3 per product. Differences are calculated as Sunflower - Viva. P-values are adjusted using the Holm method across the six comparisons. The 95% confidence intervals are conventional Welch confidence intervals, while the reported p-values were adjusted for multiple comparisons using the Holm procedure within each group of six parameters.

3.7. Comparative functional properties of the formulated sunflower oil soap and commercial Viva soap

The comparative functional properties of the formulated sunflower oil soap and commercial Viva soap are presented in Table 7. The formulated sunflower soap recorded an initial foam height of 6.90 ± 0.10 cm, compared with 9.10 ± 0.10 cm for Viva soap. Foam retention at 5, 10 and 15 minutes was 82.00 ± 1.00%, 68.00 ± 1.00% and 55.00 ± 1.00%, respectively, for the formulated soap, compared with 88.00 ± 1.00%, 77.00 ± 1.00% and 66.00 ± 1.00% for Viva soap. The cleansing efficiency of the formulated soap was 73.00 ± 1.00%, while that of Viva soap was 78.00 ± 1.00%. Dissolution-related mass loss was 18.50 ± 0.30% for the formulated soap and 15.20 ± 0.20% for Viva soap. Welch's independent-samples t-tests showed statistically significant differences between the two products across all six functional parameters following Holm adjustment for multiple comparisons (adjusted $p < 0.05$).

Table 7. Comparative functional properties of formulated sunflower oil soap and commercial Viva soap

Functional parameter	Sunflower soap	Viva soap	Mean difference	95% CI	Holm-adjusted p-value
Initial foam height (cm)	6.90 ± 0.10	9.10 ± 0.10	-2.20	-2.43 to -1.97	0.000068
Foam retention at 5 min (%)	82.00 ± 1.00	88.00 ± 1.00	-6.00	-8.27 to -3.73	0.00365
Foam retention at 10 min (%)	68.00 ± 1.00	77.00 ± 1.00	-9.00	-11.27 to -6.73	0.00116
Foam retention at 15 min (%)	55.00 ± 1.00	66.00 ± 1.00	-11.00	-13.27 to -8.73	0.00088
Cleansing efficiency (%)	73.00 ± 1.00	78.00 ± 1.00	-5.00	-7.27 to -2.73	0.00365
Dissolution-related mass loss (%)	18.50 ± 0.30	15.20 ± 0.20	3.30	2.69–3.91	0.00090

Values are presented as mean ± standard deviation (SD), n = 3 per product. Mean differences are calculated as Sunflower − Viva. P-values are adjusted using the Holm method across the six comparisons. The 95% confidence intervals are conventional Welch confidence intervals, while the reported p-values were adjusted for multiple comparisons using the Holm procedure within each group of six parameters.

3.8. Changes in relative bar mass, moisture and physical characteristics during curing

The changes in relative bar mass, moisture content and physical characteristics of the formulated sunflower oil soap during the six-week curing period are presented in Table 8. The relative bar mass decreased progressively from 100.0% at Week 0 to 94.5% at Week 2, 91.8% at Week 4 and 90.2% at Week 6, representing an overall reduction of 9.8% relative to the initial mass. Similarly, the moisture content decreased from 13.8% at Week 0 to 12.1%, 11.0% and 10.2% at Weeks 2, 4 and 6, respectively. The soap initially appeared light cream and soft at Week 0. It became firmer by Week 2 and remained firm through Weeks 4 and 6. No visible oil separation was recorded during curing, and no visible cracks were observed at Weeks 4 and 6.

Table 8. Changes in relative bar mass, moisture content and physical characteristics of formulated sunflower oil soap during curing

Curing time	Relative bar mass (%)	Moisture content (%)	Colour	Physical observation
Week 0	100.0	13.8	Light cream	Soft; moulded
Week 2	94.5	12.1	Cream	Firming; no oil separation
Week 4	91.8	11.0	Cream	Firm; no visible cracks
Week 6	90.2	10.2	Cream	Firm; stable appearance

Note: Relative bar mass is expressed as a percentage of the initial bar mass recorded at Week 0 (100%). Moisture content is expressed as a percentage. Changes in relative bar mass and moisture content were monitored over the six-week curing period. Physical observations and colour were recorded descriptively at each sampling time. Values represent the recorded observations at each time point.

3.9. Changes in pH during curing

The changes in pH of the formulated sunflower oil soap during the six-week curing period are presented in Table 9 and Figure 1. The mean pH decreased from 10.06 ± 0.33 at Week 0 to 10.02 ± 0.16 at Week 2, 9.84 ± 0.07 at Week 4 and 9.77 ± 0.01 at Week 6. The Friedman repeated-measures test showed that the observed changes in pH across the four curing time points were not statistically significant ($\chi^2 = 5.48$, df = 3, p = 0.140). Kendall's coefficient of concordance was 0.609.

Table 9. Changes in pH of formulated sunflower oil soap during the six-week curing period Values are expressed as mean ± SD for three independently manufactured soap batches (n = 3).

Curing time	S1	S2	S3	Mean ± SD
Week 0	10.42	9.98	9.78	10.06 ± 0.33
Week 2	10.08	9.84	10.14	10.02 ± 0.16
Week 4	9.91	9.78	9.82	9.84 ± 0.07
Week 6	9.76	9.76	9.78	9.77 ± 0.01

Values for S1, S2 and S3 represent individual batch measurements. The Friedman repeated-measures test did not reveal a statistically significant difference in soap pH across the four curing periods ($\chi^2 = 5.48$, df = 3, p = 0.140; Kendall's W = 0.609).

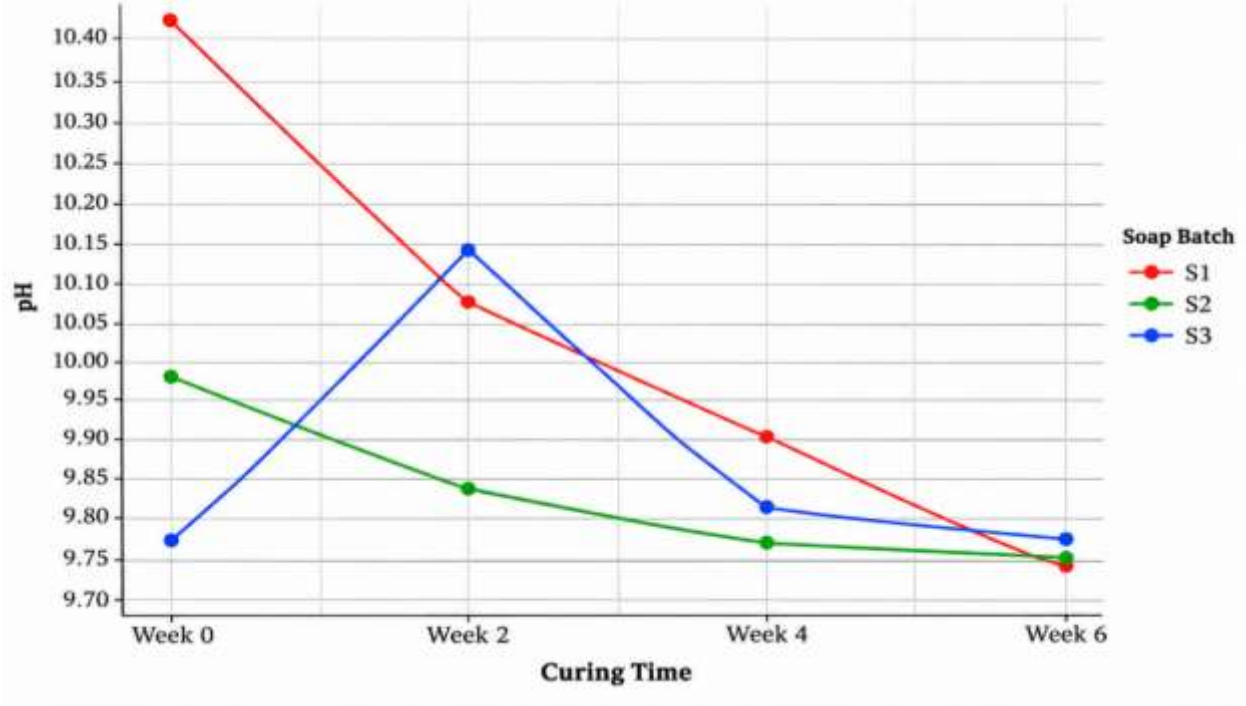


Figure 1. Changes in pH of formulated sunflower oil soap batches during the six-week curing period.

4. Discussion

The findings collectively indicate that the suitability of the extracted sunflower oil for soap production was governed by the interaction between its extraction characteristics, fatty acid composition and chemical quality, rather than by oil yield alone. The comparatively substantial recovery of oil is consistent with the seed-oil variability reported by Hosni et al. (2022), who found oil contents ranging from 35.33 to 59.67% across 22 sunflower accessions, with a mean of 53.2%, and Eseyin et al. (2020), who reported a 49% yield from sunflower seeds sourced in Jos, Nigeria. The lower yield of 26% reported by Machate et al. (2022) following cold-press extraction further illustrates the influence of extraction technology; unlike mechanical pressing, Soxhlet extraction with n-hexane promotes oil recovery through repeated solvent contact and dissolution of the lipid fraction. The fatty acid profile provides a more direct explanation of the oil's behaviour during saponification and the properties of the resulting soap. The predominance of linoleic acid, accompanied by oleic acid and smaller proportions of saturated fatty acids, identifies the oil as a highly unsaturated lipid source. This composition is broadly consistent with the sunflower-oil profiles reported by Khairuddin et al. (2024), who found 51.18% linoleic acid, 33.11% oleic acid, 8.12% palmitic acid and 5.06% stearic acid, and Machate et al. (2022), who reported 54.00% linoleic acid and 37.29% oleic acid. However, the relatively greater linoleic and lower oleic acid proportions in the present oil indicate a compositional difference that may reflect cultivar, seed-growing conditions and genetic variation, as also demonstrated by the accessions examined by Hosni et al. (2022). Mechanistically, the unsaturated fatty acids yield sodium soaps with comparatively flexible hydrocarbon chains, whereas palmitic and stearic acids contribute more saturated soap molecules that can promote tighter molecular packing and greater bar firmness. Consequently, the dominance of linoleate- and oleate-derived soaps provides a plausible basis for the observed functional behaviour of the formulated product, particularly its relatively modest foam generation and greater susceptibility to dissolution-related mass loss when compared with the commercial soap. The oil-quality indices reinforce this interpretation: the relatively low peroxide value suggests limited accumulation of primary lipid oxidation products at the time of analysis, while the acid value and free fatty acid content indicate that some hydrolytic degradation had occurred but that the oil was not extensively hydrolysed. The peroxide value was lower than the 6.300 meq/kg reported by Eseyin et al. (2020), the 9.08–9.48 meq/kg range reported by Katonge (2025), and the 16.16 reported by Machate et al. (2022). Such differences may arise from seed condition, extraction method, oxygen and light exposure, and storage history; they should not be attributed to extraction method alone. Similarly, the saponification value was higher than the 173.230 mg KOH/g reported by Eseyin et al. (2020), the 174.60–180.40 mg KOH/g range reported by Katonge (2025), and the 190.37 mg KOH/g reported by Khairuddin et al. (2024). This parameter was particularly important in the present work because it provided the chemical basis for calculating the alkali requirement, thereby linking oil characterization directly to formulation control. The successful formation of a stable trace without visible separation across the production batches suggests that the calculated alkali-to-oil relationship and processing conditions were adequate to produce a coherent soap mass, although this observation alone does not establish complete conversion of triglycerides.

The physicochemical characteristics of the formulated soap demonstrate that the fatty acid composition of the starting oil translated into a product with measurable quality attributes but also reveal trade-offs relative to the commercial comparator. The alkaline pH is consistent with the chemistry of sodium fatty-acid salts: soap hydrolysis in aqueous solution generates a mildly alkaline environment, and the measured pH therefore reflects the combined effects of soap concentration, residual alkali and the equilibrium between fatty-acid salts and water. The pH values are within the 7.01–10.17 range reported across commercial soaps by Nova et al. (2025),

whose selected bar soap had a pH of 9.83 ± 0.02, and within the 9.0–10.0 range reported for bath soaps by Maduelosi et al. (2026). The moisture content also falls within the 9.05–15.69% range reported for cocoa-pod soaps by Oyekunle et al. (2021), the 7.33–15.33% range reported by Abdul et al. (2024), and the 11.2–18.8% range reported by Maduelosi et al. (2026). Moisture is not merely a compositional attribute; it influences bar hardness, physical stability, wear and the rate at which soap dissolves during use. The relatively higher moisture content of the formulated soap compared with Viva may therefore have contributed to its greater dissolution-related mass loss. Its total fatty matter content, however, was within the 75–81% range reported by Maduelosi et al. (2026) and close to the 72.89% reported for a selected commercial bar soap by Nova et al. (2025). This indicates that the formulated product contained a substantial proportion of fatty material, although its lower total fatty matter relative to Viva suggests a smaller proportion of material recovered as fatty matter under the analytical procedure. The free caustic alkali result deserves particular attention because residual sodium hydroxide can influence product harshness and quality. The value was higher than the 0.006–0.020% range reported by Abdul et al. (2024) but remained within the 0.03–0.10% range reported by Maduelosi et al. (2026). This difference may reflect variation in alkali dosing, reagent purity, superfatting, mixing, curing and the analytical characteristics of the respective formulations. Although the use of a calculated superfatting allowance was intended to reduce the likelihood of excess alkali, the detected free caustic alkali indicates that the formulation should not be interpreted as alkali-free. The lower chloride and matter-insoluble-in-ethanol contents relative to Viva further show that the two products differed in their formulation and compositional characteristics. These differences should not automatically be interpreted as evidence of superior or inferior quality because chloride concentration and ethanol-insoluble matter are influenced by raw materials, processing and formulation practices. In particular, the chloride comparison requires caution where studies report chloride on a different chemical basis from the sodium chloride basis used in the present work. The Nigerian soap analysis by Oyekunle et al. (2021), which examined moisture, chloride, free caustic alkali and matter insoluble in ethanol, and the bath-soap assessment by Maduelosi et al. (2026), which reported chloride contents of 0.2–1.0%, provide relevant local context for understanding this compositional variability. Overall, the physicochemical findings suggest that the sunflower-oil soap achieved several characteristics associated with established bathing soaps, but its higher moisture and free caustic alkali relative to the comparator identify formulation and post-production control as important areas for further optimisation.

The functional findings provide a mechanistic connection between the chemical composition of the soap and its performance during use. The lower initial foam formation and progressive reduction in foam retention relative to Viva are plausibly related to the fatty acid distribution of the sunflower oil. Soap molecules derived from unsaturated fatty acids, particularly linoleic and oleic acids, differ in chain packing and interfacial behaviour from those derived from saturated fatty acids. These structural differences can influence adsorption at the air–water interface, the organisation of foam films and resistance to drainage and coalescence. The relatively small saturated fatty acid fraction in the sunflower oil may therefore have contributed to a soap matrix with less favourable foam persistence than a formulation containing a different balance of fatty acid salts. This interpretation is consistent with Merrill and Moffett (1944), who demonstrated that soap-foam stability varies with the composition of the soap and the nature of the fats and oils used. It is also relevant to the findings of Nova et al. (2025), who reported foam heights of 6.83–25.16 cm across 22 commercial soaps and recorded 22.33 ± 0.29 cm for their selected SN3 bar soap, illustrating the substantial variation in foaming performance among formulations. Zayed et al. (2024) reported foam stability of 92.74–94.46% in soaps produced from used cooking oil and organic waste, which was higher than the retention

observed in the present formulation; however, differences in oil composition, additives and test conditions prevent direct attribution of this contrast to fatty acid composition alone. The lower cleansing efficiency relative to Viva may likewise reflect differences in surfactant composition, foam behaviour, moisture and formulation, but foam production should not be treated as a direct measure of detergency. Cleansing depends on the ability of soap molecules to reduce interfacial tension, wet the substrate, emulsify or disperse oily soil and maintain the soil in suspension during rinsing. Stănescu et al. (2025) reported cleansing capacities of 80.88–92.16% for vegetable-oil-based laundry soaps, higher than the result obtained in the present study; nevertheless, their textile-washing and greasy-soil removal procedure differed from the artificial-soil test conducted on stainless-steel substrates here, so the numerical difference is indicative rather than directly equivalent. The greater dissolution-related mass loss of the formulated soap is consistent with the combined influence of its moisture content and fatty acid composition. A relatively unsaturated soap matrix may exhibit different solid-state packing and water interaction from one containing a greater proportion of saturated fatty acid salts, while retained water can facilitate hydration and dissolution of the bar during use. Zayed et al. (2024), who reported 100% solubility for their selected formulations, further demonstrate that dissolution behaviour can vary substantially with formulation and test conditions. The curing observations provide additional evidence of the importance of water removal to the physical development of the bar. Progressive mass and moisture loss, accompanied by increasing firmness and the absence of visible cracking or oil separation, are consistent with the dehydration and structural consolidation expected during soap maturation. Imani et al. (2025) similarly reported decreasing moisture, increasing hardness, stabilisation of pH, reduced free fatty acid content and improved foam quality and durability over curing periods of two to six weeks. In the present study, the pH showed a downward descriptive trend but the repeated-measures analysis did not establish a statistically significant change across the curing period; consequently, it would be inappropriate to claim that curing significantly reduced alkalinity. Rather, the more defensible interpretation is that the principal observable curing changes involved moisture loss and physical firming, while pH remained statistically unchanged under the conditions examined. Taken together, the results show that Soxhlet-extracted sunflower oil can serve as the lipid feedstock for producing a coherent bathing-soap bar with physicochemical characteristics broadly comparable to published soap formulations. However, the functional comparison indicates that acceptable chemical composition does not necessarily translate into equivalent foam persistence, cleansing performance or resistance to dissolution. The relatively high proportion of unsaturated fatty acids offers a plausible mechanistic explanation for some of these performance differences, while moisture retention and residual alkali identify additional formulation variables requiring control. These interpretations should be regarded as evidence-based explanations rather than proof of individual molecular mechanisms, particularly because the comparison involved three independent batches or lots per product and did not include direct measurements of fatty-acid soap speciation, micellar properties, instrumental hardness or interfacial behaviour.

4.2 Limitations of the Study

Although this study provides useful evidence on the suitability of Soxhlet-extracted sunflower seed oil for soap production, certain limitations should be considered when interpreting the findings. First, the investigation was conducted using sunflower seeds obtained from a single geographical source, and the extraction and soap-production experiments involved three independent batches. Consequently, the findings provide evidence of reproducibility under the conditions employed but may not fully represent the variability associated with different sunflower cultivars, geographical locations, growing conditions and harvesting seasons.

Second, the comparative evaluation involved one commercial soap brand, Viva, represented by three independently purchased lots. The findings therefore provide a product-specific comparison and should not be generalised to all commercially available bathing soaps. Third, although GC-FID established the relative fatty acid composition of the extracted oil, the study did not determine the individual sodium fatty-acid soap species formed during saponification or directly investigate their molecular organisation, micellar behaviour and interfacial properties. Thus, the proposed relationships between fatty acid composition and the functional behaviour of the soap are mechanistic interpretations rather than directly demonstrated molecular processes. Furthermore, the functional evaluation was limited to selected laboratory-based tests of foam formation and retention, cleansing efficiency and dissolution-related mass loss. Instrumental hardness, long-term storage stability, skin compatibility, antimicrobial performance and biodegradability were not investigated. Finally, the relatively small number of independent experimental units limits the precision and generalisability of statistical inferences, while the absence of instrumental measurements of bar hardness restricts quantitative interpretation of the physical changes observed during curing. These limitations define the scope of the conclusions and provide direction for subsequent investigations.

4.3 Conclusion

This study demonstrated the potential of Soxhlet-extracted sunflower (*Helianthus annuus* L.) seed oil as a lipid raw material for the production of solid bathing soap. The extracted oil exhibited physicochemical characteristics suitable for saponification and was predominantly composed of unsaturated fatty acids, particularly linoleic and oleic acids. Its experimentally determined saponification value provided a reliable basis for calculating the sodium hydroxide requirement, while the successful production of three independent soap batches without visible phase separation demonstrated the practical feasibility of the cold-process formulation. The formulated soap exhibited physicochemical properties within ranges reported for several previously investigated bathing and toilet soaps, particularly in relation to pH, total fatty matter and moisture content. Its comparative performance, however, revealed differences from commercial Viva soap, particularly in foam generation and retention, cleansing efficiency and dissolution-related mass loss. These differences suggest that the fatty acid composition of the starting oil, together with moisture content and formulation characteristics, plays an important role in determining the functional behaviour of the finished soap. The curing observations further demonstrated that moisture loss was accompanied by progressive firming and improved physical stability, although a statistically significant change in pH over the curing period was not established. Overall, sunflower seed oil represents a technically feasible feedstock for producing solid soap, but achieving functional performance comparable to established commercial products will require further optimisation of the formulation, particularly with respect to moisture management, residual free caustic alkali and the balance of fatty acid components. The findings provide an experimental basis for further development of sunflower-oil-based soap formulations and more detailed investigations of the relationships between lipid composition, soap structure and functional performance.

Declaration of Competing Interests

We declare that there are no competing interests associated with this research that could have influenced its conduct, findings or reporting.

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