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Unlocking the Potential: A Detailed Characterization of n-Hexane Extracted Sweet Orange Seed Oil as a Sustainable Industrial Feedstock

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Abstract
This study conducted an extensive characterization of sweet orange seed oil (SOSO), extracted using the Soxhlet method with n-hexane, to evaluate its potential as a feedstock for industrial applications. The extraction produced 41.64% (w/w) of a light-yellow oil containing 0.249% moisture. The characterization revealed that at 40 °C, the kinematic viscosity was 29.450 mm²/s, the refractive index was 1.437 at 30 °C, and the acid value was 9.24 mgKOH/g, indicating a free fatty acid (FFA) content of 4.67 mgKOH/g. The iodine value was determined to be 107.78 meq/100g, suggesting the oil is highly unsaturated. Further tests showed a saponification value of 243.446 mgKOH/kg, a peroxide value of 0.410 meq/g, a specific gravity of 0.920, and a molecular weight of 1114.68 g/mol. The thermal properties included a flash point of 217 °C, a cloud point of 12.0 °C, and a melting point of 3.5 °C. The ester value was 62.019%. The physicochemical properties closely matched those specified in ASTM D6751f, indicating that SOSO could serve as a non-edible substitute for conventional vegetable oils. Gas Chromatography-Mass Spectrometry (GC-MS) analysis showed that 71.35% of the fatty acids were unsaturated, primarily Oleic (4.34%) and Linoleic acids (67.01%), suggesting excellent cold-flow properties and high reactivity for chemical conversion. Additionally, Fourier Transform Infrared (FTIR) spectroscopy identified characteristic ester functional groups at 1745 cm^{-1}, with the C=O (carbonyl) stretching vibration indicating good structural integrity. This comprehensive study concludes that the oil has favorable properties for long-term use across various industries, particularly in biodiesel and bio-lubricant production.
Keywords: Soxhlet Extraction, sustainable biodiesel, sweet orange, Bio-industrial Feedstock.

1. Introduction

As the world moves towards a circular economy, the need for eco-friendly methods to reuse agro-industrial waste becomes increasingly important. The value of a circular economy is evident. Research shows that the citrus industry is one of the largest producers of organic waste in agriculture (Dubey et al., 2023; Lucia et al., 2022; Maqbool et al., 2023; Mruthunjaya et al., 2025). Globally, citrus fruits are mainly grown for juice, and processing generates millions of tons of waste each year, primarily peels, pulp, and seeds (Dubey et al., 2023; Mruthunjaya et al., 2025; Pratiksha and Adhikary, 2024). In processing regions, especially in southern Nigeria, these leftovers are often discarded in open landfills, harming the environment by releasing greenhouse gases (Maqbool et al., 2023; Dubey et al., 2023). Bioresource engineering research indicates that these wastes, particularly sweet orange (Citrus sinensis) seeds, are not merely garbage but contain high levels of oil with significant industrial potential (Ávila et al., 2025; Prasad, 2024; Warra and Reyna-Patel et al., 2019). Studies indicate that Citrus sinensis seeds are a valuable source of non-edible oils, known as sweet orange seed oils (SOSO), which can serve as a promising alternative raw material for biodiesel and biolubricants. They also help address the important "food vs. fuel" dilemma associated with first-generation feedstocks such as palm or soybean oil (Moreno-Gómez et al., 2020; Pandit et al., 2023). Proper characterization of the oil is essential for evaluating its suitability for applications such as bio-lubricants, alkyd resins, and biodiesel. Soxhlet extraction with n-hexane has proven to be an effective method for extracting seed oil, producing high-quality extracts that preserve the seed's natural chemical composition (AOAC, 2019; Kumar et al., 2025; Mustapha et al., 2024; Ogwuche et al., 2026; Shakya et al., 2025).

Additionally, SOSO is suggested to have a unique fatty acid composition, with high levels of unsaturated fatty acids like oleic and linoleic acids (Al-Okbi et al., 2025; Fadairo et al., 2023; Kamińska et al., 2025), which are important for oxidative stability and flow properties in bio-industrial products such as biodiesel and bio-lubricants (Kudli, 2023; Laverdura et al., 2020). Analytical techniques like Gas Chromatography-Mass Spectrometry (GC-MS) and Fourier Transform Infrared (FTIR) spectroscopy are essential for identifying specific fatty acids and

functional groups (Farag et al., 2025; Nazri, 2025). Developing a detailed fatty acid profile will help create strategies to turn this environmental challenge into valuable industrial resources (Singh et al., 2025; Sirohi et al., 2025; Verma & Dubey, 2024). Comparing these chemical properties, such as iodine value, saponification value, and kinematic viscosity, with international standards, such as ASTM D6751, will help assess its viability (Khalighi, 2024; Nayak et al., 2025; Onyia et al., 2024).

While most studies focus on general citrus seed oils, this research specifically explores the efficiency of n-hexane Soxhlet extraction. It includes detailed analysis using GC-MS and FTIR to determine the structural and lipid makeup of Nigerian varieties. This offers a clear insight into their chemical traits and confirms the composition of sweet orange seed oil from Southern Nigeria's agro-industrial areas. Moreover, this study is among the first to assess the "waste-to-wealth" potential of these local residues under strict standards like ASTM D6751, highlighting their importance in a specialized circular bio-economy.

Despite growing interest in non-edible oils, research on the detailed physicochemical and functional-group makeup of Nigerian sweet orange seed oil (SOSO) remains limited, especially as a sustainable alternative to traditional first-generation feedstocks. Most existing studies focus on the broader citrus family or lack comprehensive instrumental data needed to verify its oxidative stability and flow characteristics for high-value biolubricants and alkyd resins. This study fills that void by presenting a detailed lipid profile that differentiates Nigerian SOSO from Mediterranean or Asian varieties. Consequently, it provides valuable insights into converting an environmental pollutant, which currently adds to greenhouse gas emissions from landfills, into an industrial resource—offering a dual solution for waste management and renewable energy.

2.0 Materials and methods

2.1 Materials and reagents

Orange seeds were collected from the waste bins of fresh fruit vendors at Odegba in New Market, Enugu State, Nigeria. The extracting solvent (n-hexane) and other chemicals used were purchased from Conraws Chemical and Laboratory Equipment Ltd, Enugu, Enugu State, Nigeria. The chemicals were of analytical reagent grade (Sigma-Aldrich) and were used without further purification.

2.2. Sample pre-treatment

The seeds were manually separated from the fruit pulp, washed several times with clean water, and then rinsed repeatedly with distilled water to remove contaminants. The seeds were dried in the sun for six hours each day over five days, then dried in an oven at 110 °C for six hours to ensure a moisture content below 5%. The dried seeds were decorticated, and the kernels were ground into a fine powder using a mechanical grinder. The meal was sifted to achieve a uniform particle size of about 0.5 mm and stored in airtight containers until extraction.

2.3. Oil extraction

Oil was extracted from sweet orange seeds by direct leaching with n-hexane as the solvent, following the AOAC 2003.06 (2006) protocol and the Randall/Soxtec extraction procedure. An isomantle heating mantle on an adjustable platform facilitated easy removal or adjustment, with a three-neck round-bottom flask placed on it. A vertical reflux condenser with water hoses on each arm was fitted securely onto the flask—connected to the water faucet at the lower arm and draining into the sink at the upper arm. The flask vent was sealed to prevent vapor loss, and a thermometer was inserted through a rubber bung fitted tightly into one opening to monitor temperature. Another rubber bung sealed the inlet for adding the solvent and samples. The setup was stabilized with a retort stand, with clamps at the flask's main neck and the condenser. For each sample, 1.5 mL of n-hexane per gram of sample was measured using a measuring cylinder and funnel, then added to the flask. Water was filled into the reflux condenser, which drained into the sink as the heating mantle was turned on. The temperature was maintained at 40 °C and monitored with the thermometer. Once the temperature was stabilized, 2 g of the sample was added to the solvent to initiate oil extraction by leaching. The addition time marked the start, and extraction continued for 50 minutes at 40 °C. Afterward, the heating mantle was turned off, and the apparatus was carefully disassembled. The mixture was filtered under vacuum through a Millipore glass base and funnel, and the resulting solution was poured into a pre-weighed flask. The oil was separated from n-hexane using a rotary vacuum evaporator (Labora 4000), collected in a receiving flask, and weighed and recorded. The oil yield was calculated as per Equation (1).

$$Y = \frac{Wo}{w} \times 100 \quad (1)$$

2.4 Acid Esterification of Sweet Orange Seed Oil

In the transesterification of high FFA (sweet orange seed) oils using a base catalyst, the FFA content must be reduced to below 2 mg KOH/g or 0.1 wt% to prevent soap formation (Ezidinma et al., 2015; Salaheldeen et al., 2021). This was achieved through acid-catalyzed esterification, following a modified method outlined by Ezidinma et al. (2025). The oil sample was dehydrated by heating to 105°C for 20 minutes to remove trace moisture that could interfere with esterification. An acid-methanol mixture was prepared separately by adding a calculated amount of H_2SO_4 (98% assay), corresponding to 1 w/w% of the oil, to the measured alcohol in a 6:1 alcohol-to-oil ratio. This mixture was then added to the oil in a reactor equipped with a reflux condenser. The reaction was maintained at 65°C for 90 minutes with constant stirring at 600 rpm. Afterwards, the mixture was allowed to settle in a separating funnel for 2 hours. The top layer, containing methanol, acid, and water, was separated. Conversely, the bottom layer (pre-treated oil) was withdrawn and washed with warm distilled water at 50 °C to remove residual sulfuric acid. Washing was repeated until the wash water reached a neutral pH. The oil was then dried in an oven set to 105 °C, and its weight was recorded at 10-minute intervals. The oil was considered dry when the weight remained constant. The FFA value of the oil was confirmed to be less than 2 mg KOH/mg (1 wt%). The yield of low-FFA oil was calculated, and the oil was characterized by proximate and instrumental analyses.

2.5. Oil Characterization

2.5.1 Physicochemical characterization

The physicochemical properties of the extracted orange seed oil were analyzed using standard test methods presented in Table 1. In contrast, the proximate composition of the seeds and the extracted oil was determined following the official methods of the Association of Official Analytical Chemists (AOAC, 2019). A Fourier transform infrared (FTIR) spectrometer and gas chromatography-mass spectrometry (GC-MS) were used to determine the functional groups and fatty acid profile, respectively.

Table 1: Standard Test Methods for Characterization of Extracted Oils

Oil property	Test methods
Moisture Contents	AOCS CA 2C-25 (2017b)
Specific gravity (S.G)	AOAC 920.212 (2000c)
Refractive index	ASTM D7483-21 (2021a)
Dynamic and kinematic viscosity	AOCS CC7-25 (2009b)
Melting point	ASTM DS5440 (2017a)
Pour point	ASTM D97 (2019c)

2.5.2 Instrumental analysis

2.5.2 .1 GC-MS analysis

GC-MS analysis was used to determine the fatty acid profile of orange seed oil samples, following AOAC 969.33 and .22 (2000d) standards. The analysis employed a Thermo Finnigan Trace GC/Trace DSQ IA1300 (E. 1 Quadrupole) with an SGE-BPX5 MS fused silica capillary column (0.25 µm) for detection, using an electron ionization system at 70 eV. Helium at 10 mL/min was the carrier gas. The injector and transfer line were set to 220 °C and 290 °C, respectively. The oven temperature was programmed to rise at 3 °C/min from 50 °C to 150 °C, hold for 10 min, then increase to 250 °C at 10 °C/min. Diluted samples (1/100, vv, in methylene chloride) of 1.00 µL were manually injected in slitless mode. Components were identified by comparing retention times with authentic standards on an SGE-BPXS capillary column, matching mass spectral peaks with authentic samples or libraries (Wiley 7N and TRLIB), and referencing published data. Then, the molecular weight of the triglyceride in the oils (Mwo) was calculated from the fatty acid profile obtained by gas chromatography analysis, using Equation (3.1)

$$Mw = 3 \times Mav + 38.049. \quad (2)$$

Where Mw = molecular weight of a triglyceride, Mav = average molecular weight of the oil, 3 = number of fatty acid chains in a triglyceride, 38.049 = molecular mass of glycerol in the triglyceride (the glycerol backbone).

2.5.2.2 FTIR Spectroscopy

The FT-IR spectra of orange seed oil samples were obtained using a SHIMADZU FT-IR-8400 instrument with a 45° ZnSe HATR crystal, a DTGS detector, and KBr as the beam splitter. The device was operated with Omnic software (Version 6.0, Thermo Nicolet). To prevent contamination and ensure accuracy, the sample holder was cleaned with acetone and dried with tissue paper. During each measurement, oil samples were directly applied to the HATR surface at a stable room temperature of 20°C, covering the mid-IR range from 650 to 4000 cm^{-1}. Forty scans were averaged at a resolution of 4 cm^{-1}. Each spectrum was recorded in triplicate and averaged. Peak fitting and smoothing were performed using OPUS 2000 software on the Shimadzu 8400S within the range 4000-650 cm^{-1}.

3.0 Result and Discussion

3.1 Oil Yield and Appearance

The Soxhlet extraction of sweet orange seed oil with n- hexane yielded an oil content of 41. 64 w %. This extraction efficiency is significant and notably higher than the 34. 00 w % yield reported by Ezekoye et al. (2019), the 37% yield reported by Sadiq (2016), and the 40 w % yield reported by Ibrahim et al. (2019). The yield falls within the range of 38. 7 wt % to 45. 8 wt % reported for Jatropha curcas seeds (Jonas et al., 2020). Orange seed is classified as an oil- bearing seed because any seed yielding up to 30 w % oil is considered suitable for industrial use (Eryilmaz et al., 2016; Koubaa et al., 2016). Researchers highlight that high oil yields from citrus seeds are crucial for transforming them from environmental waste into valuable industrial feedstock (Agu et al., 2024; Seyyedi-Mansour et al., 2024; Suri et al., 2021). This yield is comparable to those reported for some common biodiesel feedstocks and other non- edible oils, such as neem seed (42. 63%) (Genene, 2024) and jatropha seed (48. 16%) (Subroto et al., 2015). The Soxhlet method, which uses n- hexane as the solvent, is a common and effective technique for extracting oil (AOAC, 2019; Kumar et al., 2025; Mustapha et al., 2024; Ogwuche et al., 2026; Shakya et al., 2025) because n- hexane dissolves lipids well and is easy to recover (El Marji et al., 2026; Kukkohovi, 2020 ; Osuji et al., 2025). The oil' s light- yellow color indicates it has not degraded or absorbed impurities, which is favorable for subsequent steps (Palanisamy, Gevert, 2016). The moisture content of 0. 249% is not sufficiently low to be acceptable. Dehydration of the oil is essential because excess moisture can lead to triglyceride and FFA breakdown, reducing catalyst activity, biolubricant quality, and biodiesel yield during transesterification (Farouk et al., 2024; Norjannah et al., 2016 Salaheldeen et al., 2021). The measured parameters- cloud point of 12. 0 ° C, melting point of 3. 3.5 ° C, and an ester value of 62. 019%, indicating high unsaturation- show that the oil remains liquid at room temperature (28 ± 2 ° C) (Nadjamoeddin et al., 2025; Umeh & Okonkwo, .2025).

3.2 Physicochemical Properties

Table 2: Physicochemical Properties of Orange Seed Oil vs. ASTM/Industry Standards

Property	Characterized Value	Unit	ASTM/Standard Method	Standard Limit (Biodiesel/Biolubricant)
Kinematic Viscosity (@ 40°C)	29.450	mm^2/s	ASTM D445	1.9 – 6.0 (Biodiesel) / 20–50 (Light Lubricant)
Acid Value	9.24	mgKOH/g	ASTM D664	< 0.5 (Biodiesel) / < 2.0 (Lubricant)
Free Fatty Acid (FFA)	4.67	mgKOH/g	—	< 1% (for direct base-catalysis)
Specific Gravity (@ 15°C)	0.920	—	ASTM D1298	0.86 – 0.90 (Biodiesel)
Flash Point	217	°C	ASTM D93	130 min (Biodiesel) / > 200 (Lubricant)
Iodine Value	107.78	$gI_2/100g$	ASTM D1510	< 120 (Standard for stability)
Saponification Value	193.45	mgKOH/g	ASTM D5558	180 – 265 (Typical for seed oils)
Peroxide Value	0.410	meq/kg	ASTM D3584	< 10 (Freshness limit)
Cloud Point	12.00	°C	ASTM D2500	Location-dependent (Report only)

Moisture Content	0.249	%	ASTM D2709	< 0.05% (Biodiesel)
Refractive Index	1.4637	—	ASTM D1218	1.44 – 1.48 (Typical)

Kinematic viscosity is a key property of vegetable oils used as feedstocks for biodiesel and biolubricants because it directly impacts engine performance, fuel atomization, and lubrication. The kinematic viscosity of SOSO at 40°C was measured at 29.450 mm²/s. This value is notably lower than the 46.57 mm²/s earlier reported by Onyinye and Tagbo (2023), but higher than the 1.9 to 6.0 mm²/s range typical for biodiesel (ASTM D6751). However, it aligns with the 29.22 mm²/s reported for cottonseed oil by Ezidinma et al. (2015). Ayetor et al. (2015) reported that the kinematic viscosity of coconut oil decreased from 27 to 2.4 mm²/s after transesterification. Nevertheless, biodiesel made from this oil would have lower viscosity, which fits within the specified ranges, because larger fatty acid molecules are broken down into smaller methyl esters (El Marji et al., 2026; Kukkohovi, 2020; Osuji et al., 2025).

The refractive index indicates the oil's quality and purity. SOSO's refractive index of 1.437 at 30°C falls within 1.44–1.48 specified by ASTM D6751, and closely matches the 1.4626 reported for SOSO by Agu, et al., (2024) and the 1.4658 reported by Onyinye and Tagbo (2023) for sweet orange seed oil used in bio-lubricant synthesis.

The acid value and free fatty acid (FFA) content are key properties in biodiesel production. The acid value of sweet orange seed oil (9.24 mgKOH/g), which corresponds to an FFA content of 4.27 mgKOH/g, exceeds the 2 mgKOH/g limit specified by ASTM for direct alkaline catalyzed transesterification. Feedstocks with FFA levels above 1-2% (about 2-4 mgKOH/g) are unsuitable for direct alkaline transesterification because they cause soap formation, emulsions, saponification, reduced ester yield, and catalyst damage (Farouk, et al., 2024; Olatundun et al., 2024; Salaheldeen et al., 2021). Despite this high FFA content, SOSO can still be used for alkaline transesterification by first going through an acid-catalyzed esterification step (section 2.4.2) to reduce FFA levels to below 1.0 mgKOH/g before continuing with alkaline transesterification. This approach helps maximize biodiesel and biolubricant yields and quality (Olatundun et al., 2024; Salaheldeen et al., 2021). This two-step method is often used for oils with high FFA levels (Farouk, et al., 2024; Olatundun et al., 2024; Salaheldeen et al., 2021).

The measured iodine value of 107.78 meq/100g aligns with the ASTM standard (<120 meq/100g) and indicates that SOSO's fatty acid profile has a medium to high level of unsaturation. This value is significantly higher than the 31.47 meq/100g reported by Agu et al. (2024) for SOSO and also exceeds the 56.4047 meq/100g reported for SOSO by Sadiq (2016). The elevated iodine value is reflected in the 67.1% linoleic acid shown by GC-MS analysis. A higher iodine value signifies more double bonds, which can affect properties such as cloud point, pour point, and oxidative stability (Amran & Ruslan, 2022; Kumar et al., 2024; Miyuranga et al., 2022; Pradana et al., 2024). Generally, high unsaturation enhances the low-temperature performance of biodiesel and biolubricants.

The measured cloud point of 12.0°C is similar to the 11°C and 10.2°C reported for SOSO by Agu et al. (2024) and Sadiq (2016), respectively. The cloud point of 12.0°C and the melting point of 3.5°C suggest that biodiesel derived from SOSO would flow well in cold weather and potentially outperform oils with higher saturation, such as palm oil (Hazrat et al., 2020; Leng & Zhou, 2020; Yadav & Chaudhary, 2020; Yusof et al., 2021). However, higher unsaturation also makes the biodiesel more susceptible to oxidation during storage, which can lead to gum formation and engine deposits (Agregán et al., 2017; Çamur & Al-Ani, 2022; Zhang et al., 2021). Research indicates that antioxidants may be required as additives to boost oxidative stability (Agregán et al., 2017; Çamur & Al-Ani, 2022; Chizoo et al., 2023; Hazrat et al., 2021; Zhang et al., 2021).

The saponification value is another key property of triglyceride oils, reflecting the average length of fatty acid chains. A saponification value of 243.446 mgKOH/g falls within the ASTM D6751 standard range of 180 to 265 mgKOH/g for seed oils. Generally, a higher saponification value indicates shorter fatty acid chains, which reduces viscosity and improves combustion in biodiesel and biolubricants (Alsaadi & Ahmad, 2025; Neupane, 2022; Folayan et al., 2019; Giakoumis & Sarakatsanis, 2018).

The specific gravity of 0.920 is typical of vegetable oils and close to the 0.86–0.90 range specified for biodiesel (ASTM D6751). With a molecular weight of 1114.68 g/mol and a flash point of 217°C—exceeding safety standards for handling and storage—the oil is considered safe for use. Its flash point ensures suitability for high-temperature applications and surpasses safety requirements for biodiesel and lubricants.

The peroxide value of 0.410 meq/g, far lower than the <10 meq/g ASTM standard, indicates low initial oxidation, signaling good quality and stability (Agregán et al., 2017; Çamur & Al-Ani, 2022; Zhang et al., 2021). This is desirable for industrial feedstock, as higher peroxide values could increase the risk of polymerization during heating and negatively impact catalyst performance and the final product quality (Kim et al., 2018; Meng et al., 2023; Monirul et al., 2015).

3.3 Fatty Acid Profile Analysis (GC-MS)

The GC-MS chromatogram (Fig. 1) revealed several distinct peaks corresponding to the Fatty Acid groups present in the SOSO, while the major identified components are summarized in Table 3.

The GC-MS analysis of the extracted oil (Figure 1) and Table 3 shows that the oil mainly consists of unsaturated fatty acids (UFA), with Linoleic Acid (67.01%) and Oleic Acid (4.34%) making up around 71.35% of the total composition.

The most abundant component is 9,12-Octadecadienoic acid (Z,Z)-, also known as linoleic acid (C18:2), which accounts for 67.01% of the total area percentage. Next is cis-13-Octadecenoic acid (oleic acid, C18:1) at 4.34%. The saturated fatty acid (SFA) fraction, which makes up 31.13% of the total, is mainly composed of n-hexadecanoic acid (palmitic acid, C16:0) at 25.28% and octadecanoic acid (stearic acid, C18:0) at 5.75%.

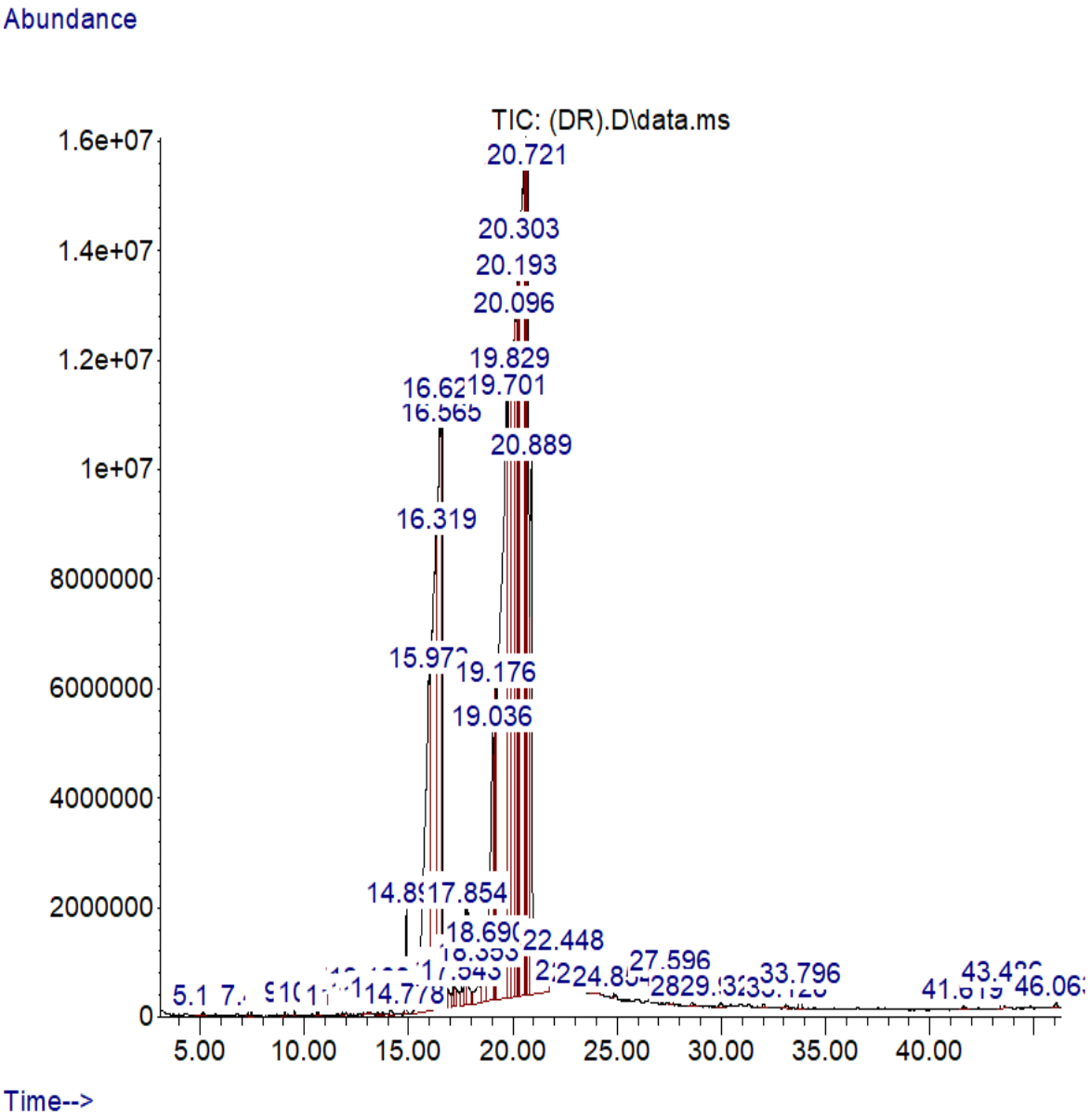

Figure 1: Total Ion Chromatogram of Orange Seed Oil.

Table 3: Fatty acid Profile of sweet orange seed oil

Fatty Acid	Common Name	Total Area%	Primary Retention Times (RT)
9,12-Octadecadienoic acid (Z,Z)-	Linoleic Acid	67.01%	19.701, 20.538, 20.096
n-Hexadecanoic acid	Palmitic Acid	25.28%	16.319, 16.519, 15.973
Octadecanoic acid	Stearic Acid	5.75%	20.889
cis-13-Octadecenoic acid	Oleic Acid	4.34%	20.721
Tetradecanoic acid	Myristic Acid	0.10%	12.895

The GC-MS analysis of SOSO in the current study is shown in Table 3, while Table 4 compares GC-MS analysis results of sweet orange seed oils from the current study, seeds collected from the University of Nigeria Nsukka (Ezekoye, et al., 2019), and seeds collected from Fitidi in Oyo State, Nigeria (Nwozo, et al., 2021). These results reveal a significant disparity among them, suggesting that both the fatty acid composition and concentrations vary depending on the seed source location (Özcan et al., 2023). Additionally, small amounts of tetradecanoic acid (myristic acid) were detected at 0.10%. The ratio of unsaturated to saturated fatty acids, 2.29 to 1, indicates that biodiesel produced from this oil will flow well at low temperatures, which is important for its use as a renewable energy source.

Table 4. Comparative analysis with research implications

Fatty Acid	SOSO (Current Study) (%)	Literature (Orange Seed Oil)	
		Nwozo[a]	**Ezekoye[b]**
Linoleic Acid (C18:2	67.01	37.644	----------
Palmitic Acid (C16:0)	25.28	28.051	1.63
Stearic Acid (C18:0)	5.75	5.839	39.42
Oleic Acid (C18:1)	4.34		17.65
Isopropyl linolate	----------	12.699	---------
pentadecanoic acid	----------	9.652	---------
Butyl Linolate	----------	2.867	---------
Lauric acid	----------	---------	7.73
Palmitic acid	----------	---------	1,63
Myristic acid	0.10	---------	33.56

Source: Nwozo, et al., (2021)[a]; Ezekoye, et al., (2019)[b]

Palmitic acid (C16:0) is the main saturated component, providing a good balance of fluidity and oxidative stability (Jadhav et al., 2022; Redondo-Cuevas et al., 2018). As Jadhav et al. (2022) and the National Academies of Sciences, Engineering, and Medicine (2021) have noted, a high linoleic acid content is especially beneficial for the paint and coatings industry because it helps oils dry by linking them together. Additionally, the high level of unsaturation indicates that products made from SOSO would perform well at low temperatures, which is important for biolubricants used in temperate climates (Narayana Sarma and Vinu, 2022; Opia et al., 2023). The increased unsaturation level in triglyceride oils suggests these products will work effectively at low temperatures, which is critical for biolubricants in temperate regions.

3.4 FTIR spectrum of the orange seed oil

Figure 1 displays the FTIR spectrum of SOSO, offering key information about its molecular-level chemical structure. Triacylglycerols (TAGs), the primary components of vegetable oils, show characteristic absorption bands that dominate most of the spectrum. The Carbonyl Region (1740–1745 cm^{-1}): A sharp, intense peak around 1740 cm-1 results from the C=O stretching vibration of the ester carbonyl group in triglyceride molecules. Its high intensity and narrow width indicate many esters are present, which are necessary for converting into biodiesel (Fatty Acid Methyl Esters) or biolubricant base oils (Atadashi et al., 2013). The Aliphatic Hydrocarbon Region (3000–2800 cm^{-1}): The strong bands at 2922 cm-1 and 2853 cm^{-1} are due to the asymmetric and symmetric stretching of aliphatic CH_2 groups. This confirms the presence of long-chain fatty acids, which are essential for

biolubricants to form a strong lubricating film and for fuel applications to achieve high energy density (Bhuiya et al., 2016).

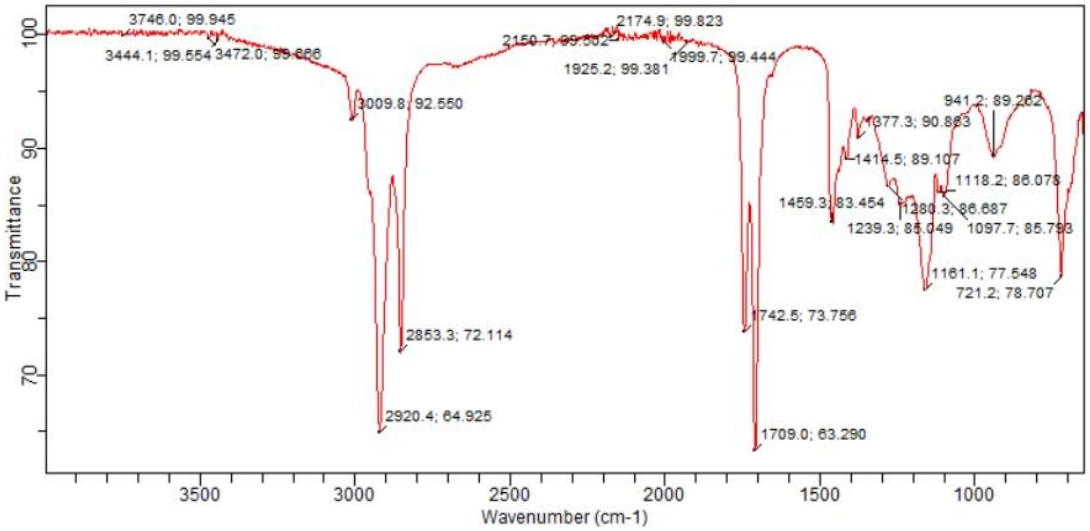

Figure 2: FT-IR spectrum of the orange seed oil

The FTIR spectrum of the Sweet Orange Seed Oil (SOSO) (Figure 2) was examined to identify the functional groups and structural markers that determine its industrial applications. The complete list of peak assignments is shown in Table 5.

Table 5: FTIR absorbance spectra and functional groups of SOSO

Wavenumber (cm−1)	Functional Group	Type of Vibration	Technical Interpretation
3472.0 – 3444.1	O-H	Stretching	Indicates low moisture and trace hydroperoxides.
3009.8	=C-H (cis)	Stretching	Confirms unsaturation; validates 67.01% Linoleic acid.
2920.4	-CH_2	Asymmetric Stretching	Methylene groups of long aliphatic hydrocarbon chains.
2853.3	-CH_2	Symmetric Stretching	Methylene groups of long aliphatic hydrocarbon chains.
2174.9 – 2150.7	C ≡ C (Alkynes)	Stretching	Presence of internal triple bonds/terminal alkyne residues.
1742.5	C=O (Ester)	Stretching	Primary carbonyl group of triglycerides.
1709.0	C=O (Acid)	Stretching	Free fatty acid (FFA) carbonyl; correlates with acid value.
1459.3	-CH_2	Bending (Scissoring)	Aliphatic chain deformation in long-chain fatty acids.
1414.5	-CH_2	Bending (Rocking)	Vibration of methylene groups adjacent to carbonyl/alkene sites.
1377.3	C-H (CH_3)	Symmetric Bending	Methyl group "umbrella" mode.
1280.3	C-O	Stretching	Functional group characteristic of ester linkages.
1239.3	C-O	Stretching	Asymmetric stretching of the triglyceride ester structure.
1161.1	C-O-C	Stretching	Primary ester bond vibration in glycerides.

1097.7	C-O	Stretching	Stretching associated with the ester/glycerol backbone.
941.2	=C-H	Out-of-plane Bending	Indicators of unsaturated fatty acid configurations.
721.2	$(CH_2)_n$	Rocking	Confirms a long hydrocarbon skeleton ($n \geq 4$).

Analysis of the spectrum revealed the presence of esters (C=O), alkenes (C=C), alkynes (C≡C), and alkanes (C–H), as well as carbonyl (C=O) and alcohol (OH) functional groups in the oil. The detection of esters (C=O), alkenes (C=C), alkynes (C≡C), and alkanes (C–H), along with carbonyl (C=O) and alcohol (OH) groups, confirms that the oil is a complex mixture of triacylglycerols with specific indicators of chemical stability and composition (Chen, et al., 2025; Chijioke, et al., 2024; Ufoma, 2023). A notable feature of the spectrum is the dual-peak structure in the carbonyl region. The intense absorption at 1742.5 cm-1 is attributed to the C=O stretching of ester groups in triglycerides, while the peak at 1709.0 cm-1 specifically indicates the C=O stretching of free fatty acids (FFAs) (Agbike, 2023; Gunarathne, et al., 2022; Mekonnen, 2023). This spectroscopic evidence correlates with the high acid value (9.24 mgKOH/g) of the oil (Khairuddin, et al., 2024; Rohman, & Indrayanto, 2023; Uçar, et al., 2024), indicating partial hydrolysis of triglycerides into free acidic components (Cocero, et al., 2024; Menalla, et al., 2024; Vicentini-Polette, et al., 2021).

The peak at 3009.8 cm-1 corresponds to the =C-H stretching of cis-olefinic double bonds. The presence of this peak confirms the high degree of unsaturation (Bathini, et al., 2018; Debabhuti, et al., 2022; Fu, et al., 2021), specifically, the 67.01% Linoleic acid content—identified by GC-MS. The aliphatic nature of this feedstock is verified by the dominant asymmetric and symmetric C-H stretching bands at 2920.4 cm^{-1} and 2853.3 cm^{-1}, which represent the methylene backbones of the fatty acid chains (Gadhiya, 2026; Li, et al., 2025; Soodoo et al., 2023; Srivastava & Mishra, 2026).

Another notable feature in the mid-infrared region is the low-intensity absorption detected between 2174.9 and 2150.7 cm^{-1}. This band is attributed to the C ≡ C stretching vibration, indicating the presence of alkyne functional groups within the lipid matrix (Bakthavatsalam, et al.,2021; Pielesz, et al., 2023; Romei, et al., 2022). Although this feature is less common than alkenes in vegetable oils, these triple-bond residues can occur in specialized non-edible seeds and suggest a unique chemical signature for the oil (Nigerian sweet orange variety) (Adeoye, et al., 2023; Magaji, et al., 2025; Ivchenko and Nifant'ev, 2025), potentially affecting the oil's reactivity during industrial processes such as polymerization or alkyd resin synthesis (Hadzich, et al., 2023; Ifijen, et al., 2022; Zohuriaan-Mehr, and Omidian, 2020). The presence of these alkynes complements the significant unsaturation identified at 3009.8 cm^{-1} (cis-alkene stretching), which also offers spectroscopic confirmation for the 67.01% Linoleic acid detected via GC-MS.

The high transmittance and lack of significant depth in the 3444.1 cm^{-1} region suggest a low concentration of O-H groups associated with moisture or primary oxidation products (Xi, et al., 2021; Zhang, et al., 2024). This confirms that the n-hexane Soxhlet extraction successfully recovered a high-purity oil while maintaining its oxidative integrity. The long-chain nature of the fatty acids (more than four carbon atoms) is further verified by the CH_2 rocking vibration at 721.2 cm^{-1}.

These spectroscopic assignments offered a thorough and consistent chemical profile, aligning the instrumental data with the reported physicochemical results. Additionally, the detailed mapping of C-O and C-H bending modes at 1161.1 cm^{-1} and 1459.3 cm^{-1} confirms the high concentration of stable triglycerides needed for high-yield biodiesel production (Lamb, 2024; Salaheldeen, et al., 2021; Verma, et al., 2024), while the presence of specific bending modes (e.g., -CH2 at 1414.5 cm^{-1}) suggests reactive sites near carbonyl groups, which are essential for the successful synthesis of alkyd resins (Arauz-Solís, et al., 2025; Hadzich, et al., 2023; Ifijen, et al., 2022). The free acid components (C-O), indicated by the 1280.3 cm^{-1} peak, demonstrate that this study provides a non-destructive method to monitor oil degradation and hydrolysis, a crucial requirement for international standards like ASTM D6751.

3.5 **The Industrial "Waste-to-Wealth" Potential of SOSO.**

Orange seeds are highly valuable in industrial applications because of their significant oil content, low acidity, and abundance of healthy fats. Using this seed oil in industry can help fruit-processing businesses to reduce waste disposal costs and create extra income by selling the seed or seed oil to producers of biofuels, cosmetics, and chemicals. This strategy aligns with the views of researchers like Tahulela, et al., (2026), who believe that transforming waste into useful products is a smart way to promote industrial growth in Africa. This method offers an effective way to reduce waste and boost economic development.

4.0. Conclusion

This study analyzed Sweet Orange Seed Oil (SOSO) extracted from Citrus sinensis residues to evaluate its potential as a sustainable industrial raw material. The research was driven by the idea that agro-industrial waste, which causes environmental problems in Nigerian landfills, has a unique chemical structure suitable for high-value industrial applications.

The key findings indicate that Soxhlet extraction with n-hexane yields a high-purity oil with a distinctive fatty acid profile, primarily composed of Linoleic acid (67.01%) and Palmitic acid (25.28%). Analytical techniques such as GC-MS and FTIR confirmed a high concentration of unsaturated fatty acids and identified stable triglyceride ester bonds, which are important for oxidative stability and flow properties. Additionally, the physicochemical properties of the extracted SOSO meet ASTM D6751 standards, confirming its suitability for producing biodiesel, biolubricants, and alkyd resins.

These results are significant for the circular bio-economy, offering a data-driven way to turn "waste" into industrial "wealth" and reduce the environmental impact of citrus processing leftovers, showing that SOSO as a viable non-edible feedstock addresses the "food vs. fuel" dilemma and promotes national industrial independence.

5.0 Recommendation

Future research should focus on the catalytic transesterification of this Nigerian SOSO variety using waste-derived heterogeneous catalysts to improve process sustainability. Additionally, exploring computational modeling tools like Artificial Neural Networks (ANN) and Artificial Neuro-Fuzzy Inference System (ANFIS) can help optimize reaction parameters for industrial-scale applications.

Private investors in the fruit-processing industry should consider setting up integrated oil-recovery units that combine seed collection and processing.

We need to conduct more research on how to extend OSO's lifespan by preventing its oxidation when exposed to air. This would make it suitable for use in premium cosmetics or medicines.

The government in Nigeria should provide special tax incentives to companies that utilize waste-to-wealth technologies, particularly those that use local, non-edible materials like orange seeds. This can help decrease the country's dependence on imported synthetic oils. By doing so, the government can motivate more businesses to adopt these innovative technologies and generate new opportunities for growth and development. It is a simple yet effective way to promote sustainability and bolster the local economy.

To support environmental conservation, local farming officials in Nigeria and other citrus-growing regions should promote small juice producers to adopt methods that prevent waste. This can help lessen the environmental damage caused by discarding seeds and other waste openly. By utilizing all parts of the fruit, these small businesses can reduce their ecological footprint and increase the sustainability of their operationsR

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Nomenclature

FFA= Free Fatty Acid, mgKOH/g;

KOH= Potassium Hydroxide;

SOSO = Sweet Orange Seed Oil;

Y = Oil yield, %;;

Declaration of Generative AI and AI-assisted technologies in the writing process

During the preparation of this work the author(s) used Grammarly in order to correct grammatical errors. After using this tool/service, the author(s) reviewed and edited the content as needed and take(s) full responsibility for the content of the publication.

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