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Production and Physicochemical Characterization of Epoxidized Baobab Seed Oil for Green Polymer Applications

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Abstract

Epoxidized vegetable oil (EVO) was synthesized from baobab seed oil (BSO) which is rich in oleic and linoleic acids compared to other vegetable oils, with potential applications in sustainable materials, particularly as a bio-based precursor for epoxy resin production in the polymer industry. The oil was extracted via a cold press method, yielding approximately 55% of oil from the seed biomass. Epoxidation was performed through a homogeneous reaction system using in situ generated performic acid from formic acid and hydrogen peroxide. The epoxidized baobab seed oil (EBSO) obtained had a yield of 81% and exhibited key physicochemical characteristics, such as moisture content (3.2%), oxirane oxygen content (3.6%), iodine value (91.78 g I_2/100 g oil), acid value (4.9 mg KOH/g), and saponification value (181 mg KOH/g). The successful progress of the epoxidation reaction was further validated by Fourier Transform Infrared Spectroscopy (FTIR), which revealed the characteristic oxirane absorption band in the range of 820–850 cm^{-1}.These results highlight the suitability of baobab seed oil for producing bio-based epoxides for green polymer and composite applications.

Keywords: *Baobab seed oil, epoxidation, epoxy resins, renewable resources, green chemistry*

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1.0 INTRODUCTION

Vegetable oils are increasingly valued as sustainable alternatives to petrochemical feedstocks because of their desirable attributes, including high viscosity index, good lubricity, high flash point, low volatility, biodegradability, and low toxicity. These advantages make them attractive for use as lubricant base oils and other industrial applications. However, their practical utility is limited by drawbacks such as poor thermal, oxidative, and hydrolytic stability, as well as weak low-temperature performance (Gner*et al.,* 2006).One important derivative, epoxidized vegetable oils (EVOs), has gained significant attention for its multifunctional role as both a plasticizer and stabilizer in polyvinyl chloride (PVC), and as a matrix material in composites (DeHonor-Marquez *et al.,* 2018). In composites, the polymer matrix often a thermosetting resin reinforced with fibers like E-glass provides mechanical strength and thermal resistance. Thermosetting resins undergo irreversible cross-linking reactions in the presence of hardeners, yielding materials with high robustness and dimensional stability (Habib & Bajpai, 2011; Kulczyski *et al.,* 2019). Among these, epoxy resins are the most widely used, largely due to the reactivity of their epoxide functional groups. The extent of epoxidation strongly influences the thermal, mechanical, and processing characteristics of the resulting polymers (Azhar *et al.,* 2017; Liu et al., 2015; Lligadas *et al.,* 2006).

Although extensive work has been carried out on the epoxidation of soybean and other vegetable oils (Babakura *et al.,* 2019; Azhar *et al.,* 2017; Kiatsimkul *et al.,* 2006), studies on baobab seed oil remain scarce. Epoxides, or oxiranes, are highly reactive three-membered cyclic ethers that serve as valuable intermediates in diverse chemical transformations (Kim & Sharma, 2012; Goud *et al.,* 2016). Epoxidation is typically achieved by adding an oxygen atom to carbon–carbon double bonds within fatty acid chains, using per-carboxylic acids or organic/inorganic peroxides as oxidants (Goud *et al.,* 2010).

The fatty acid profile of vegetable oils strongly affects their suitability for epoxidation. Oils rich in unsaturated fatty acids provide more reactive sites for oxirane formation, and the extent of conversion is generally assessed through oxirane oxygen content and iodine value (Patrovic *et al.,* 2003). Baobab seed oil is compositionally distinct from soybean oil: it is richer in monounsaturated oleic acid (37.7%) (Buhari *et al.,* 2014), while soybean oil contains a higher proportion of polyunsaturated linoleic acid (53%) (Campanella *et al.,* 2004). Baobab oil also has a greater saturated fatty acid content (31%) compared to soybean oil (15%) (Nkafamiya *et al.,* 2007). Since saturated fatty acids are not epoxidizable, this composition could affect both yield and product performance; nevertheless, the predominance of oleic acid suggests promising potential for epoxy resin development.

The degree of epoxidation is particularly important for determining resin viscosity and melting temperature properties that govern processability in applications such as composite manufacturing. For instance, liquid molding methods require resins with viscosities below 500 N·s/m to avoid void formation and ensure efficient processing (Samper *et al.,* 2015; Liu *et al.,* 2015).

In practice, EVOs are usually produced via in situ formation of peracids from hydrogen peroxide and organic acids such as acetic or formic acid. Acetic acid systems require catalysts, which may be homogeneous or heterogeneous (Kiatsimkul *et al.,* 2006). Heterogeneous catalysts, such as ion-exchange resins, are often preferred due to their ease of separation, reusability, and lower environmental impact (Campanella & Baltans, 2015). By contrast, homogeneous catalysts often promote epoxy ring-opening, yielding hydroxylated

by-products. Solvents such as hexane, heptane, benzene, and toluene are sometimes used to suppress side reactions and facilitate purification, though they pose toxicity, recovery, and cost challenges (Rangarajan *et al.,* 2015; Campanella *et al.,* 2015). Ultimately, the efficiency of epoxidation is governed by key variables including reaction temperature, oxidant concentration, oxygen carrier ratio, and catalyst type and loading (Dinda *et al.,* 2017; Goud *et al.,* 2016; Mungroo *et al.,* 2019).

The relative fractional conversion to oxirane, as reported by Goud *et al*. (2016), can be calculated using the oxirane oxygen content values according to the following equations:

Relative conversion to oxirane $(RCO) = \frac{OOc}{OOt}$ ---------- 1

Where

OO_c=Experimental oxygen oxirane

OO_t = Theoretical maximum oxirane oxygen from the expression. (Yelwa and Abdullahi 2019)

$$OO_t = \left[\frac{IV_o/2A_i}{100+(IV_o/2A_i)A_o}\right] A_o \times 100$$ ---------- 2

Where,

Ai (126.9) and *Ao* (16.0) are the atomic weights of iodine and oxygen respectively and *IVo* is the initial iodine value of sample.

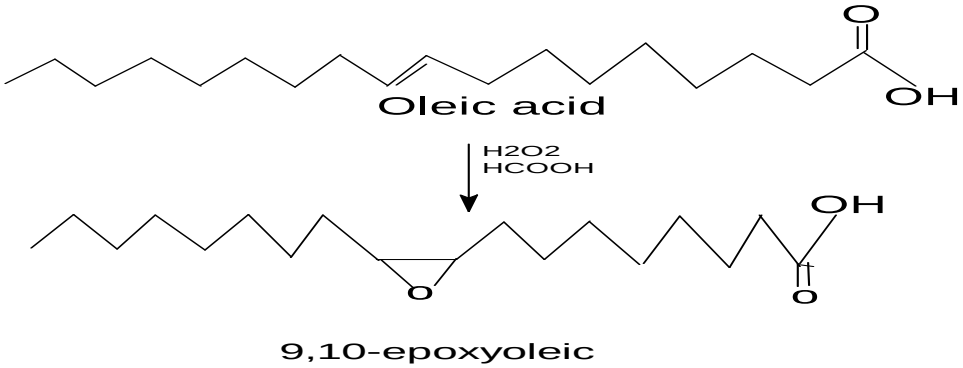


Figure 1: A schematic diagram of epoxidation reaction

To this end the paper presents the results of extraction, epoxidation and characterization of baobab seed oil, because the oil is rich in oleic acid (≈ 37– 40%), a monounsaturated fatty acid. This gives it good stability while still providing sufficient double bonds for epoxidation (Buhari*et al.,* 2014).

2.0 Materials and Method

2.1 Materials

The following materials were used for the various experiments that were carried out: distilled water, formic acid and hydrogen peroxide. In terms of equipments, we used an analytical balance (Model JA 11003), a hot plate with temperature control and a magnetic stirrer were employed for heating and mixing processes. A thermometer and condenser were used for

temperature monitoring and reflux operations, respectively. Spectroscopic analysis was conducted using an FT-IR spectrometer (Model 530).

2.2 Method:

2.3 Extraction of Baobab Seed Oil

Baobab seed oil was extracted from pulverized seed powder using the cold press method, as described by Jabar *et al*. (2016). The pulverized seed powder was initially mixed with hot water and transferred into a muslin cloth bag. The bag was securely tied and subjected to mechanical pressing until no further oil was released. The crude oil obtained was then purified by heating in a drying oven at 100 °C to remove residual moisture. Characterization of the purified oil was carried out according to AOAC standards.

2.4 Epoxidation Procedure of Baobab Seed Oil.

The epoxidation of baobab seed oil was carried out in a well-equipped laboratory setup designed to ensure both precision and safety throughout the reaction process. A 500 mL three-neck round-bottom flask served as the reaction vessel. The flask was fitted with a mechanical stirrer to ensure uniform mixing, a thermometer for accurate temperature monitoring, and a reflux condenser to prevent the loss of volatile components. The entire assembly was positioned on a thermostatically controlled hot plate to regulate the heating process.

To initiate the reaction, the baobab seed oil was introduced into the flask, and the system was cooled to a temperature of 10 °C. Once the desired temperature was achieved, 46.1 g of 30% hydrogen peroxide was carefully added dropwise into the mixture under continuous stirring. This slow addition, carried out over the course of approximately one hour, ensured controlled reactivity and minimized the risk of side reactions or thermal decomposition.

Following the complete addition of the oxidizing agent, the temperature of the reaction mixture was gradually increased to 70 °C. This temperature was maintained steadily for a period of eight hours, allowing sufficient time for the epoxidation process to proceed to completion.

Upon completion of the reaction, the mixture was allowed to cool naturally to room temperature (approximately 22 °C). The resulting epoxidized oil was extracted using ethyl acetate as the solvent. To purify the product, the organic phase was washed repeatedly with warm water until the washings reached a neutral pH, indicating the removal of residual acidic components. The washed oil was then dried over anhydrous sodium sulfate to eliminate traces of moisture.

The purified epoxidized baobab oil was subsequently characterized to assess the efficiency and extent of epoxidation. Analytical evaluations included Fourier-transform infrared (FTIR) spectroscopy to identify functional group transformations, determination of the iodine value to assess the degree of unsaturation, and measurement of the oxirane oxygen content as an indicator of epoxy group formation. All characterization procedures were conducted in accordance with standard methods established by the Association of Official Analytical Chemists (AOAC).

2.5 Characterization of Epoxidized Baobab Seed Oil (EBSO)

To assess the physicochemical properties and the extent of epoxidation, a series of standard analytical methods were employed. These included spectroscopic analysis and chemical titrations, following validated protocols such as those recommended by the American Oil Chemists' Society (AOCS) and ASTM standards.

2.6 Fourier Transform Infrared (FT-IR) Spectroscopy

Fourier Transform Infrared (FTIR) spectroscopy was employed to monitor functional group transformations associated with epoxidation. Spectral measurements were performed using a Buck Scientific FTIR spectrophotometer (Model 530). Samples were prepared in the form of KBr pellets, and spectra were recorded in the mid-infrared range (4000–500 cm^{-1}). The disappearance of C=C stretching bands and the appearance of characteristic oxirane absorption bands were used as indicators of successful epoxidation.

2.7 Moisture Content Determination

Moisture content was determined by the gravimetric oven-drying method. The weight of a clean, dry crucible was first recorded (Wc). A small quantity of oil (approximately two drops) was added to the crucible, and the combined weight was measured (Wco). The sample was subsequently dried in a laboratory oven at 105 °C for 8 hours to achieve complete removal of moisture. After drying, the crucible was cooled in a desiccator and reweighed (Wo). The percentage moisture content was then determined using the following equation:

$$\%moisture\ content = \frac{(Wco - Wo)}{(Wco - Wc)} \times 100 \text{----------3}$$

The analysis was conducted in triplicate, and the average moisture content was reported to ensure accuracy and reproducibility.

2.8 Iodine Value Determination

The iodine value, an indicator of the degree of unsaturation in the oil, was determined using the cyclohexane–acetic acid method following the AOCS Official Method Cd 1d-92. This method involves the addition of Wijs reagent to the sample, followed by titration of the residual iodine with sodium thiosulfate. A reduction in iodine value after epoxidation indicates a successful conversion of double bonds to epoxy groups.

2.9 Saponification Value Determination

Saponification value (SV), which provides insight into the average molecular weight (chain length) of the fatty acids in the oil, was determined following the procedure described by Nkafamiya *et al*. (2007). A known mass of oil (2.0 g) was reacted with an excess of 0.1 M alcoholic potassium hydroxide (KOH) and heated at 60 °C for 5 minutes under constant stirring to complete saponification. The unreacted KOH was back-titrated with 0.5 M hydrochloric acid (HCl) using phenolphthalein as an indicator. The pink endpoint indicated complete neutralization. The saponification value was calculated using the expression:

$$SV = (B - S) \times 5\acute{6}.1\ Wt\ of\ sample \text{----------4}$$

where:

- B = volume of HCl used for blank titration (mL)
- S = volume of HCl used for sample titration (mL)
- W = weight of oil sample (g)
- 56.1 = molecular weight of KOH

Measurements were performed in duplicate, and the average value was reported.

2.10Oxirane Oxygen Content (OO_C) Determination

The oxirane oxygen content, a critical parameter for evaluating the degree of epoxidation, was determined according to AOCS Official Method Cd 9-57 and ASTM D1652-97 (Test Method A). This method involves the titration of epoxy groups with hydrobromic acid (HBr) in glacial acetic acid. The oxirane oxygen content was calculated using the following equation:

$$\%OOc = \frac{(V-B)\times N\times 1.60}{W} \text{----------5}$$

where:

- V = volume of HBr solution used for sample titration (mL)
- B = volume of HBr solution used for blank titration (mL)
- N = normality of HBr solution
- W = weight of sample (g)
- 1.60 = milliequivalent weight of oxygen (g/meq)

This analysis provides a quantitative estimate of the epoxy functionality introduced during the epoxidation process.

3.0 RESULTS AND DISCUSSION

A. Characterization of Baobab Seed Oil

The extraction of baobab seed oil (Adansonia digitata) yielded 55% (v/w), as shown in Table 1. This yield is considered appreciable for non-conventional oilseeds and is higher than that reported by Buhari *et al.* (2012). When compared to other seed oils, such as mahogany seed oils with yields of 48.6% and 54.3% (Dinda *et al.*, 2017; Babakura *et al*., 2019), baobab seed oil demonstrates relatively superior oil content, likely attributable to seed varietal differences, growing conditions, or extraction methodology.

The iodine value, a measure of the degree of unsaturation, was determined to be 124.87 I_2/g. This high value indicates a significant presence of unsaturated fatty acids, making the oil a suitable candidate for epoxidation. The iodine value exceeds those reported by Yelwa*et al.* (2019) and Danbature *et al.* (2015) for sunflower oil, which may result from intrinsic plant genetic differences or geographical factors affecting lipid biosynthesis.

The acid value, an indicator of free fatty acid content and oil quality, was found to be 0.047 mg KOH/g. This is significantly lower than the 5.99 mg KOH/g reported by Abdulhamid *et*

al. (2012), suggesting minimal hydrolytic degradation and highlighting the efficiency of the cold press extraction technique. A low acid value is favourable for base-catalyzed reactions, such as transesterification and epoxidation, as it reduces soap formation and catalyst deactivation.

Moisture content was recorded as 3.74%, which is considerably lower than the 22.27% moisture content of neem oil reported by Wang *et al*. (2014). This low value confirms that the oil is relatively stable and less prone to microbial degradation or hydrolysis during storage.

The saponification value, reflecting the average molecular weight (chain length) of the fatty acids in the triglyceride, was determined to be 190.38 mg KOH/g. This is slightly lower than the 210 mg KOH/g reported by Buhari *et al.* (2012), indicating a modest variation in fatty acid composition which could influence the polymerizability and mechanical strength of resins derived from the oil.

Table 1: Physicochemical Properties of Baobab Seed Oil (BSO).

Parameter	**Baobab Seed Oil**
% oil yield (v/wt)	55
% moisture content	3.74
Acid value (mgKOH/g)	0.047
Iodine value (I_2/g)	124.87
Saponification value (mgKOH/g)	190

B. Characterization of Epoxidized Baobab Seed Oil (EBSO)

Epoxidation of unsaturated vegetable oils introduces oxirane rings into their structures, converting double bonds into epoxy functionalities, which are essential for applications in resin and polymer synthesis. The epoxidized baobab seed oil (EBSO) exhibited an oil yield of 81%, indicating high efficiency of the epoxidation reaction.

The oxirane oxygen content (OOC) of the epoxidized baobab seed oil (EBSO) was determined to be 3.6%, indicating a substantial degree of conversion of the available double bonds into epoxy functional groups. Although this value is lower than the 6.1% OOC reported for epoxidized soybean oil (Wang *et al.*, 2012), it still falls within the acceptable range for the synthesis of bio-based thermosetting resins, where moderate oxirane content is sufficient to achieve effective cross-linking. The slightly lower OOC observed for EBSO can be attributed to its fatty acid composition, particularly the higher proportion of saturated fatty acids in baobab oil compared to soybean oil, which reduces the number of reactive sites available for epoxidation.

Furthermore, the reduction in iodine value from 124.87 g I_2/100 g in the native baobab seed oil (BSO) to 91.78 g I_2/100 g in the epoxidized product confirms the partial consumption of

C=C double bonds during the reaction. The incomplete decrease suggests that while a significant portion of unsaturation was converted to oxirane rings, some double bonds remained unreacted. This is consistent with the behavior of oils containing mixed fatty acid profiles, where steric hindrance and uneven reactivity of different unsaturated chains can limit complete epoxidation.

Overall, the balance between oxirane oxygen content and residual iodine value highlights the potential of baobab seed oil as a precursor for bio-based epoxy resins. The achieved OOC provides sufficient epoxide functionality for cross-linking reactions, while the residual unsaturation could impart additional flexibility to the resulting polymer matrix an attribute that may be advantageous in composite and coating applications.

The acid value of the epoxidized baobab seed oil (EBSO) was found to increase to 4.9 mg KOH/g, which can be attributed to side reactions that occur during epoxidation. In particular, epoxy ring-opening in the presence of acidic catalysts and residual water can generate hydroxyl and carboxylic acid functionalities. These secondary reactions not only elevate the acid value but may also influence the reactivity and stability of the resulting resin, since increased acidity can promote further degradation or interfere with curing agents in thermosetting systems.

The moisture content of the oil decreased slightly to 3.21%, reflecting additional dehydration during the heating and purification stages. This reduction is desirable because excess moisture can compromise epoxy stability, promote hydrolytic degradation, and negatively affect resin curing. Thus, the lower moisture level in EBSO suggests improved storage stability compared to the raw baobab seed oil.

The saponification value of EBSO decreased marginally to 181 mg KOH/g, consistent with structural modifications of the triglyceride backbone during epoxidation. Since the saponification value is inversely related to the average molecular weight of fatty acids in triglycerides, a reduction indicates that some ester linkages were altered during the reaction. This may arise from cleavage or rearrangement of ester bonds under the reaction conditions, leading to a slight increase in the average molecular weight of fatty acid chains. Such changes are commonly observed during chemical modifications of vegetable oils and further confirm that the epoxidation process not only transforms double bonds into oxirane rings but also induces subtle alterations in the overall triglyceride structure.

Together, these observations an elevated acid value, reduced moisture content, and a slightly lower saponification value highlight the complex interplay of desirable epoxidation and unavoidable side reactions. While the formation of acidic by-products may present challenges for resin formulation, the reduced moisture and modified backbone chemistry suggest that EBSO retains strong potential as a bio-based precursor for polymer and composite applications.

Table 2: Physicochemical Properties of Epoxidized Baobab Seed Oil (EBSO)

Parameter	Quantity
% oil yield (v/wt)	81
% moisture content	3.21
Acid value (mgKOH/g)	4.9
% Oxirane content	3.6
Iodine value (I2 /g)	91.78
Saponification value (mgKOH/g)	181

C. FTIR Spectral Analysis of BSO and EBSO

Fourier Transform Infrared (FTIR) spectroscopy was employed to investigate structural transformations in BSO and EBSO. Figure 2 presents the spectra for both oils, and Table 3 summarizes the corresponding absorption peaks.

In the FTIR spectrum of raw baobab seed oil (BSO), characteristic absorption bands were clearly identified, reflecting its triglyceride composition. The strong peaks observed at 2850–2929 cm^{-1} are attributed to the symmetric and asymmetric stretching vibrations of methylene ($–CH_2$) groups, which dominate the long hydrocarbon chains of fatty acids. The intense band at 1750 cm^{-1} corresponds to the ester carbonyl (C=O) stretching vibrations, a signature feature of triglyceride backbones. Additionally, the presence of unsaturation was confirmed by the =CH bending vibration at 1465 cm^{-1}, associated with olefinic protons, while the absorption at 1174 cm^{-1} is characteristic of C–C–O stretching in ester linkages, further corroborating the glyceride structure of the oil.

Following epoxidation, the spectrum of epoxidized baobab seed oil (EBSO) revealed significant modifications that confirm the chemical transformation of double bonds into epoxy groups. The marked reduction or disappearance of the absorption band around 2850 cm^{-1} indicates the consumption of C=C bonds, consistent with the loss of olefinic unsaturation. More importantly, the appearance of new absorption bands in the region of 830–850 cm^{-1} is diagnostic of oxirane ring (C–O–C) stretching vibrations, which serve as direct evidence of successful epoxidation. This oxirane band is a critical fingerprint, distinguishing the epoxidized derivative from the native oil.

In addition, the appearance of a broad band at 3473 cm^{-1} is attributed to hydroxyl (–OH) groups, which are likely generated through partial ring-opening of oxirane groups or secondary side reactions under the acidic and thermal epoxidation conditions. The presence of hydroxyl functionalities suggests that, alongside epoxidation, competing reactions occurred, introducing polar groups that may influence the reactivity and crosslinking potential of the oil in resin formulations.

Taken together, the FTIR spectra provide compelling evidence of structural transformation: disappearance of olefinic C–H absorptions, emergence of oxirane-specific peaks, and development of hydroxyl groups. These changes not only confirm the efficiency of the epoxidation process but also highlight secondary modifications that can affect downstream applications. Specifically, while the formation of oxirane rings enhances the oil's utility as a precursor for thermosetting resins, the introduction of hydroxyl groups may improve compatibility with hydrophilic curing agents, potentially broadening the application range of EBSO in polymer composites, coatings, and adhesives.

Table 3. FTIR Absorption Bands of BSO and EBSO

Functional Groups	**Absorption Peak (cm^{-1})**	
	BSO	**EBSO**
CH=CH	2850	-
CH stretch	2929, 2858	2923, 2857
C=O	1750	1750
CH scissoring band	1465	1453
C-C-O	1174	1163
C-O-C oxirane group	-	830-850
OH (strong)	-	3473

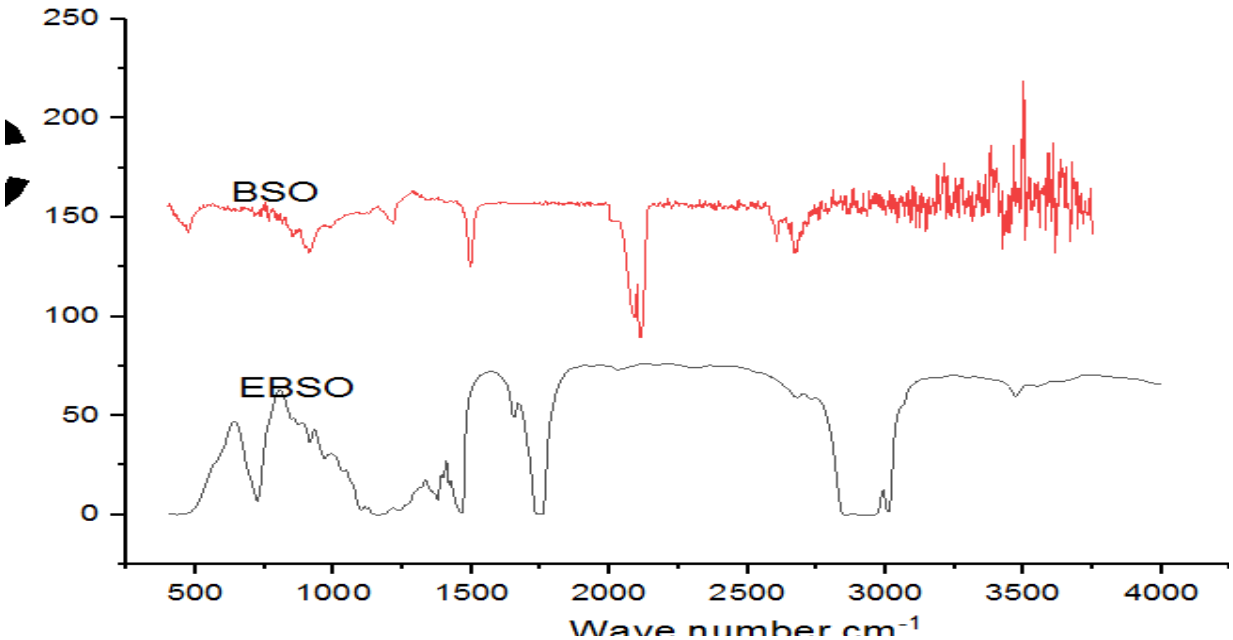


Figure 2: FTIR Spectrum of baobab seed oil (BSO) and epoxidized baobab seed oil (EBSO).

The spectral transformation confirms the successful epoxidation of baobab seed oil, making it a potential bio-based precursor for the synthesis of epoxy resins and other polymeric materials.

4.0. Conclusion

The epoxidation of baobab seed oil (BSO) was successfully carried out using a performic acid system derived in situ from formic acid and hydrogen peroxide. The process yielded approximately 81% epoxidized baobab seed oil (EBSO), indicating a relatively efficient conversion under the adopted reaction conditions.

The maximum oxirane oxygen content obtained was 3.6%, which is moderate when compared to epoxidized products from conventional oils such as soybean or linseed oil. This relatively low oxirane content may be attributed to the limited degree of unsaturation (as reflected in the initial iodine value of 124.87 I_2/g) inherent in baobab seed oil, which constrains the formation of epoxy rings.

The decrease in iodine value post-epoxidation and the emergence of characteristic oxirane ring absorption bands in the FTIR spectra confirm the successful incorporation of epoxide functionalities into the oil structure. These findings demonstrate the technical viability of baobab seed oil as a renewable feedstock for the synthesis of bio-based epoxides, although further optimization or modification may be required to enhance the oxirane content for industrial applications such as resin or plasticizer synthesis.

In conclusion, baobab seed oil represents a promising non-edible, underutilized resource for the production of functionalized bio-based chemicals, contributing to the development of sustainable materials and circular bio-economy.

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Conflict of Interest

The authors in the course of the study express no conflict of interest.

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