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Epoxidation of Palm Cooking Oil to Bio-Plasticizer by Sulfuric Acid Catalyzed

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Abstract

The increasing environmental and health concerns associated with conventional phthalate-based plasticizers have driven research toward sustainable alternatives. This study investigates the epoxidation of palm cooking oil using sulfuric acid as a catalyst to produce epoxidized palm cooking oil as a bio-based plasticizer. The epoxidation process was conducted at various temperatures and catalyst concentrations to evaluate their effects on iodine value, double bond conversion, and viscosity. Optimal epoxidation was achieved at 50 °C and 1 wt% sulfuric acid, resulting in an iodine value of 1.86 g I_2/100 g and a C=C conversion rate of 65.53%. FTIR spectroscopy confirmed the successful formation of oxirane rings, as evidenced by the appearance of C–O–C stretching peaks. The epoxidized product exhibited increased viscosity and density, indicating successful structural modification. Furthermore, blending tests with PVC showed good miscibility and plasticizing performance, supporting epoxidized palm cooking oil potential application as a renewable, non-toxic plasticizer in polymer processing.

INTRODUCTION

The utilization of compounds derived from renewable resources as alternatives to petroleum-based chemicals is gaining increasing attention, driven by the rising cost of fossil fuels as well as environmental and economic considerations. Among the materials widely explored are polyhydroxyalkanoates, polylactic acid, cellulose-based plastics, thermoplastic starch, and vegetable oils. Among all of these alternatives, vegetable oils emerge as a highly promising raw material due to their biodegradability, cost-effectiveness, widespread availability, and strong social acceptance. The chemically complex composition of vegetable oils, consisting mainly of fatty acids and glycerol esters, offers a high degree of structural flexibility, making them well-suited for a range of industrial applications. Plasticizers, polymers, and composites derived from epoxidized vegetable oils are environmentally friendly, biodegradable, and non-toxic, making them a promising starting point in the advancement of green technology (Tan & Chow, 2010; Thiele, et al., 2019; Marriam, et al., 2023)

To improve the flexibility, durability, and processability of materials, plasticizers are commonly incorporated during the formulation and manufacturing of poly (vinyl chloride) (PVC). Among various types of plasticizers, phthalates remain the most extensively used in industrial applications, accounting for the majority of the global plasticizer market. However, phthalates are known to migrate from plastic products into the human body via ingestion, inhalation, or direct contact, posing potential risks to both human health and the environment. In response to these concerns, epoxidized vegetable oils have been developed as bio-based alternatives. Epoxidized vegetable oils are derived from vegetable oil biomass through the

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epoxidation of unsaturated double bonds in their molecular structure and are suitable for use in food-contact plastic applications. Mechanistically, the epoxy ring in epoxidized vegetable oils can react with hydrogen chloride released during PVC degradation, facilitating the reintroduction of labile chlorine atoms into the polymer backbone. This interaction effectively inhibits further dehydrochlorination, thereby preserving color stability and minimizing the loss of mechanical flexibility in plasticized PVC at elevated temperatures (Bai, et al., 2020).

Various types of vegetable oils have been utilized in the synthesis of plasticizers through the epoxidation process, derived from both edible and non-edible plant oil sources. Examples include cocoa butter (Satwikanitya, et al., 2023), palm oil (Puwanto, et al., 2011; Alamsyah , et al., 2013), castor oil (Sudrajat, et al., 2007), soybean oil, and nyamplung oil (Muniarti, et al., 2022). Additionally, the use of cottonseed oil (Dinda, et al., 2008), mustard oil, linseed oil, karania oil, canola oil, and neem oil (Devansh, et al., 2024) has also been reported. Indonesia ranks as the top global producer of palm oil, exporting approximately 46 million metric tons each year (USDA, 2025; Jenflower, et al., 2024). However, a substantial portion is still traded in crude form, which limits its economic potential. This research aims to increase the value of domestically produced palm oil by developing chemically modified derivatives, particularly through epoxidation, for applications in bio-based materials.

The synthesis of epoxidized palm oil is conducted through a ring opening reaction using formic acid as the oxygen carrier and hydrogen peroxide as the oxygen donor. However, hydrogen peroxide is a relatively weak oxidizing agent and must be converted into a more active species such as performic acid to facilitate efficient epoxidation (Listiana, et al., 2017). Numerous studies have examined the parameters influencing the optimization of vegetable oil epoxidation, including reaction time, temperature, oxygen donor concentration, and the type and amount of catalyst used, such as formic acid and acetic acid (Tan & Chow, 2010; Saremi, et al., 2012; Varghese & Gopalakrishnan, 2023; Aguilera, 2019). Gunawan, et al., (2023) investigated the effects of reaction temperature, the molar ratio of formic acid to hydrogen peroxide, and reaction duration on the epoxidation of terminalia *catappa L.* seed oil, using sulfuric acid as the catalyst. Under optimal conditions of 45°C, five hours of reaction time, and a formic acid to hydrogen peroxide ratio of four to five, the resulting product exhibited an oxirane oxygen content of 3.46 % and a relative oxirane conversion of 78%.

Nor & Salimon (2019) conducted a study on the epoxidation of palm oil using performic acid. The reaction was carried out under optimal conditions with a molar ratio of 5.91:3.60 of formic acid to hydrogen peroxide, at 40 °C for 2.55 hours. The process yielded epoxidized palm oil with a high yield of 86% and oxirane conversion of 99.43%. The influence of formic acid and hydrogen peroxide has become a critical factor in evaluating the epoxidation process. While most existing literature explores the effect of formic acid concentration, there is limited research on the role of sulfuric acid catalyst concentration across different temperatures. In addition, in epoxidation processes, acetic acid is generally considered superior to formic acid due to its higher effectiveness and selectivity. Although formic acid forms the active epoxidizing agent more rapidly, its use often triggers side reactions such as epoxide ring opening. Conversely, acetic acid tends to produce epoxide products in higher yields and fewer side reactions (Harshal and Jyotsna, 2013). Therefore, this research focuses on investigating the effect of reaction temperature and sulfuric acid catalyst concentration on palm oil epoxidation with acetic acid as oxygen carrier.

MATERIALS AND METHOD

Materials

Palm cooking oil (PCO) in this study was purchased from market places. Acetic acid high purity, hydrogen peroxide 30%, sulfuric acid 96%, natrium bicarbonate also was purchased from market places. Kalium iodine and natrium thiosulfat were used in the titration method for determining percent of epoxy.

Method

Apparatus

The experimental setup employed in this study included a range of laboratory equipment such as standard glassware, a beaker constructed from heat-resistant borosilicate glass to ensure thermal stability, a separatory funnel for phase separation, a magnetic stirrer and hot plate for controlled mixing and heating, as well as an alcohol-based thermometer for accurate temperature monitoring throughout the reaction process.

Epoxidation of Palm Cooking Oil

The epoxidation reaction was performed in a 500 mL beaker glass equipped with a magnetic stirrer and placed on a hotplate. The beaker was tightly sealed with aluminum foil, leaving an opening for a thermometer and feed inlet. A total of 100 grams of palm cooking oil, along with predetermined amounts of acetic acid and sulfuric, were added into the reactor and heated to the desired reaction temperature under constant stirring. Subsequently, 30 wt% hydrogen peroxide was slowly added over the course of less than one hour. The reaction mixture with molar ratio oil:acetic acid:hydrogen peroxide = 1:1:1 was allowed to proceed for three hours. As the epoxidation reaction is highly exothermic, the system temperature was carefully monitored and controlled. During the experiment, the mixture was stirred at 400 rpm. At the end of the reaction period, samples were withdrawn and the mixture was separated from the solvent and catalyst using a separatory funnel. The resulting epoxidized palm oil was washed with warm water at 50 °C in eight cycles, followed by the addition of approximately ten drops of sodium bicarbonate solution until a neutral pH was achieved. The final product was dried using a vacuum evaporator at 120°C for 30 minutes.

Iodine Value (I)

The iodine value (I) represents the amount of iodine (I_2, in grams) that reacts with 100 g of oil, serving as an indicator of the level of unsaturation (presence of double bonds) in fatty acids. This measurement was conducted using Wijs reagent (ICl), which selectively reacts with carbon–carbon double bonds. The extent of the reaction was quantified via titration with sodium thiosulfate. The I was calculated based on ISO 3961 and expressed using Eq. (1).

$$I = \frac{12.69 \times c \times (V_1 - V_2)}{m} \qquad (1)$$

Where the parameter c denotes the concentration (normality) of sodium thiosulfate solution (0.1 N), while V_1 and V_2 indicate the volumes of sodium thiosulfate required for titrating the blank and the sample, respectively, both expressed in milliliters. The variable m corresponds to the mass of the oil sample (in grams). Furthermore, the percentage of double bond conversion (X_I) can be calculated using Eq. (2).

$$X_I(\%) = \left(\frac{I_0 - I_f}{I_0}\right) \times 100 \qquad (2)$$

Where the parameter of I_0 denotes the initial iodine value measured from the palm cooking oil sample prior to epoxidation, while If represents the final iodine value of the epoxidized palm cooking oil.

FTIR

The characterization of double bond substitution and functional group transformations was performed using Fourier Transform Infrared (FTIR) spectroscopy. FTIR provides a rapid and non-destructive analytical approach to detect molecular structural changes, facilitating the monitoring of epoxidation progression via infrared spectral shifts. Although volumetric titration methods are reported to offer higher quantitative precision (Zheng, 2015). FTIR was employed in this study to qualitatively validate the structural evolution of key functional groups.

FTIR measurements of the dried epoxidized palm cooking oil samples were conducted at Universitas Pendidikan Indonesia, utilizing institutional analytical facilities.

Physical properties

The assessment of specific gravity was performed utilizing a 25 mL calibrated pycnometer, following the ASTM D1963 protocol. All density measurements were conducted at a controlled temperature of 25 °C, with water serving as the reference standard. The specific gravities of both native and epoxidized palm cooking oil were subsequently calculated using Eq. (3).

$$\rho = \frac{M_s - M_e}{M_w - M_e} \qquad (3)$$

where Ms denotes the mass (in grams) of the pycnometer filled with the sample, Me represents the mass of the empty pycnometer, and Mw indicates the mass of the pycnometer when filled with distilled water.

The measurement of dynamic viscosity was performed using two Cannon-Fenske viscometers, in accordance with ASTM D-445 standard procedures. The viscometers were immersed in a thermostatically controlled water bath to ensure precise temperature regulation during the analysis.

RESULTS AND DISCUSSION

Properties of Palm Cooking Oil

Palm cooking oil is characterized by its slightly pale yellow appearance and liquid state under ambient conditions. Yahya, et al., (2018) reported that the density of PCO at 25 °C was measured at 0.904 g/cm³, while its dynamic viscosity reached 40 cP, indicating considerable intermolecular interactions and flow resistance typical of triglyceride-rich vegetable oils. Moreover, the iodine value—an indicator of unsaturation level and a critical parameter in assessing oxidative susceptibility—was measured at 5.4 g-I_2/100g, different from documented by Roslan, et al., (2023). These physicochemical attributes reflect the compositional profile of palm cooking oil, offering insights into its stability, reactivity, and suitability for thermal processing and storage in food applications.

Table 1. Physcochemical properties of palm cooking oil.

Parameter	Value
Colour	Yellow
Water Content (%)	0.07
Impurities Content (%)	1.48 ± 0.01
Density at 20°C (g/mL)	0.9066 ± 0.0002
Molecular Weight (MW) (g/mol)	857.85
Saponification Value (SV) (mg KOH/g oil)	200.59 ± 0.3
Acd Value (AV) (mg KOH/g oil)	4.4039 ± 0.05
Free Fatty Acid (%FFA) (%)	2.01 ± 0.02
Ester Value (mg KOH/g oil)	196.1889

Effect of Temperature and Catalyst Concentration on C=C Conversion of Palm Cooking Oil

Sulfuric acid was applied as a homogeneous catalyst at concentrations of 1 wt%, 2 wt%, and 3 wt% to assess its effect on the epoxidation of palm cooking oil, targeting the conversion of C=C bonds. All reagents were mixed at 60 °C for 3 h under constant stirring at 400 rpm. The iodine values of the raw palm cooking oil and the epoxidized products are reported in Table 1 and shown in Figure 1.

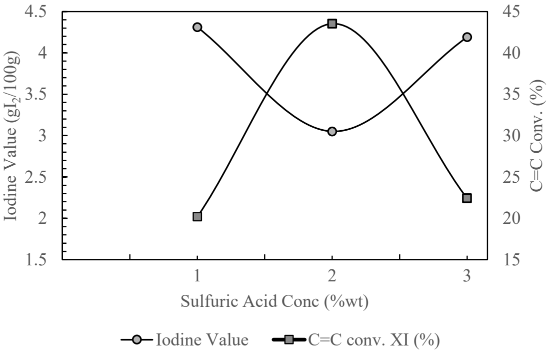

Figure 1. Effect of sulfuric acid concentration on iodine value and C=C conversion of palm cooking oil at 50 °C.

Table 2 indicates that at a catalyst concentration of 1 wt%, the iodine value of the epoxidized palm cooking oil was measured at 1.86, corresponding to a C=C bond conversion of approximately 65.53%. This observation can be attributed to the role of acetic acid and hydrogen peroxide in forming peracetic acid, which subsequently reacts with the double bonds during epoxidation. When the catalyst concentration was increased to 2 wt% and 3 wt% led to a reduction in the iodine value and a decline in the C=C conversion, as depicted in Fig. 1. This result suggests that excess catalyst may shift the peracetic acid formation equilibrium to the left, thereby limiting its availability for effective epoxidation.

Viscosity measurements at 25 °C demonstrated that the epoxidation process with varying concentrations of sulfuric acid catalyst lower than the viscosity of the fresh palm cooking oil used as the feedstock. Among the tested catalyst concentrations, a concentration of 3 wt% yielded epoxidised oil with the highest viscosity. The effect of reaction temperature on iodine value, C=C bond conversion during epoxidation, and the viscosity of the epoxydized oil is summarized in Table 3.

According to the data presented in Table 3, the iodine value of the epoxidized oil increased significantly from 1.86 to 4.31 as the epoxidation temperature rose from 50 °C to 60 °C. Similarly, the conversion of C=C bonds in palm cooking oil decreased from 65.53% to 20.10% due to the

Table 2. The iodine values of the raw palm cooking oil and the epoxidized products at 50°C.

Sulfuric acid Conc. (%wt)	Iodine Value	C=C conv. X_I (%)	Viscosity at 25°C, cP	Density at 25°C, g/cm^3
PCO	5.40		40	0.9040
1	1.86	65.53	3.22	0.9278
2	1.90	64.75	5.69	0.9261
3	2.54	53.00	6.96	0.9268

Table 3. Effect of temperature reaction on iodine value and C=C conversion of palm cooking oil at 1%wt sulfuric acid.

Temperature Reaction (°C)	Iodine Value	C=C conv. X_I (%)	Viscosity at 25°C, cP	Density at 25°C, g/cm^3
PCO	5.40		40	0.9040
50	1.86	65.53	3.22	0.9278
60	4.31	20.10	5.36	0.9229
65	1.95	63.97	5.8	0.9266

epoxidation process. Reaction temperatures above 60 °C are not recommended, as they promote undesirable side reactions such as polymerization and oxirane ring opening by water and/or acetic acid. Sulfuric acid acts as a strong oxidizing agent that accelerates the polymerization process. A temperature of 50 °C was identified as the most suitable condition for this epoxidation. Additionally, Table 3 indicates that the viscosity of the epoxidized oil at 25 °C increased with rising epoxidation temperature. This behavior is likely attributed to the lower degree of unsaturation, as the reduction in C=C bonds tends to increase viscosity.

Characterization and Detection of Epoxidized Products

FTIR spectroscopy was employed to confirm the presence of oxirane functional groups in epoxidized palm oil. Figure 2 presents the FTIR spectral analysis of the epoxidized palm cooking oil sample. The characteristic epoxide absorption band attributed to the asymmetric stretching of the C–O–C group was observed in the range of 1230–1240 cm^{-1}, in agreement with previous findings reported by (Shah & Ahmad, 2012). The disappearance of absorption bands between 1650–1680 cm^{-1}, associated with C=C stretching vibrations, indicates a significant reduction in unsaturation, thereby confirming the successful formation of epoxy rings. Additional signals between 1200–1250 cm^{-1} further support the presence of C–O bonding within the oxirane ring. The absorption bands in the region of 2800–3000 cm^{-1} correspond to C–H stretching vibrations, typically attributed to aliphatic hydrocarbon chains. The broad absorption at 3300–3500 cm^{-1} is indicative of O–H stretching vibrations, likely arising from intermolecular hydrogen bonding in residual H_2O_2. A distinct peak at 850 cm^{-1} may be associated with O–O vibrational stretching within the H_2O_2 molecule. Moreover, the absorption band near 1700 cm^{-1} is attributed to C=O stretching vibrations in carboxylic acid groups (–COOH), while the bands between 1300–1400 cm^{-1} suggest C–O stretching within these same functional groups. Lastly, a peak in the range of 1150–1250 cm^{-1} corresponds to symmetric and asymmetric stretching of the S=O bonds, confirming the presence of residual sulfuric acid used during the epoxidation process.

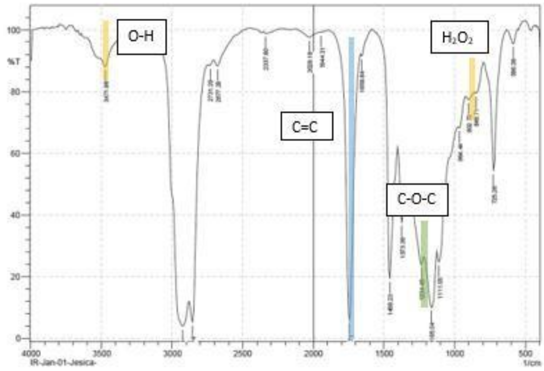

Figure 2. FTIR spectra of epoxydized palm cooking oil.

Miscibility Evaluation of Epoxidized Oil with PVC Matrix

The epoxidized palm oil sample, previously dried under conditions of 1 wt% sulfuric acid and 50 °C, was subsequently blended with dioctyl phthalate and polyvinyl chloride. The mixture consisted of 3 grams of epoxidized palm cooking oil, 1 gram of polyvinyl chloride, and 1 gram of dioctyl phthalate, all combined in a crucible. The blend was then heated at 220 °C and maintained for 15 minutes. The results of the heating process are presented in Figure 3.

CONCLUSION

Silver nanoparticles synthesized from a solution of 1.5 mM $AgNO_3$ with 25% of star fruit (*Averrhoa bilimbi* L.) extract demonstrated stability over a period of 4 days. Characterization of the silver nanoparticles using TEM and PSA revealed a spherical morphology with an average particle size of 14.8 nm, and the particle size distribution fell within the range of less than 100 nm. Additionally, the Sun Protection Factor (SPF) value for the sunscreen activity of silver nanoparticles with star fruit extract as the bio-reductant at a concentration of 1.5 mM $AgNO_3$ and 25% of star fruit extract was determined to be 44.14, categorizing it as ultra protection with a high level of effectiveness.

Figure 3. Visual observation of epoxided palm cooking oil- polyvinyl chloride-dioctyl phthalate mixture after thermal treatment.

Epoxidized palm cooking oil exhibited good compatibility with PVC, supporting the evidence that oxirane groups were successfully formed within the epoxidized structure. Furthermore, the resulting product demonstrated adequate plasticity and tensile resistance, although its mechanical strength characteristics require further investigation.

CONCLUSION

This study successfully demonstrated the epoxidation of palm cooking oil using sulfuric acid as a catalyst to produce epoxidized oil as a potential bio-based plasticizer. The results showed that the optimal epoxidation condition was achieved at a reaction temperature of 50 °C and a catalyst concentration of 1 wt%, yielding an iodine value of $1.86\,g\,I_2/100\,g$ and a C=C bond conversion of 65.53%. FTIR analysis confirmed the formation of oxirane rings through the presence of characteristic C–O–C stretching vibrations. Viscosity and density measurements indicated a correlation between reduced unsaturation and increased physical property values. Additionally, compatibility testing with PVC revealed that epoxidized palm cooking oil demonstrated favorable miscibility and plasticizing behavior, although further investigation is required to assess its mechanical performance comprehensively. Overall, epoxidized palm cooking oil shows promise as a sustainable and safer alternative to conventional phthalate-based plasticizers.

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