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Epoxidation of Eugenol from Clove Oil (*Syzygium aromaticum*) Using H_2O_2 Oxidizer and H_3PO_4 Catalyst

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Abstract

Clove oil (Syzygium aromaticum) contains 80–90% eugenol. Eugenol is a pale yellow liquid with several active functional groups, such as hydroxyl, aromatic rings, and allyl groups, which allow it to be modified to produce various eugenol derivative compounds. This study aims to characterize eugenol epoxy resin synthesized from clove oil eugenol using hydrogen peroxide (H_2O_2) and phosphoric acid (H_3PO_4) as a catalyst. The resulting eugenol epoxide was in the form of a yellowish-white gel with a distinctive clove aroma. It was soluble in benzene and chloroform but insoluble in ethanol. FTIR analysis showed that the epoxy compound contained an ether group (C-O-C) at absorption peaks of 1266.07 cm^{-1} to 1018.66 cm^{-1}, a hydroxyl group (—OH) at 3347.08 cm^{-1}, a methyl group (-CH_3) at 1470 cm^{-1} to 1350 cm^{-1}, an alkene group (C=C) at 1637.39 cm^{-1}, and a vinyl group at 910.2 cm^{-1}.

Keywords: Catalyst, clove oil, epoxidation, eugenol

Introduction

Indonesia is rich in various spice plants, widely distributed across different regions, offering significant potential for sourcing raw materials to produce essential oils like clove. Clove has long been used in the health sector, particularly in pharmaceuticals. It is employed in treating coughs, headaches, epilepsy, as a sleep inducer, a blood thinner, and a mental stimulant. Additionally, clove is an active ingredient in several mouthwashes used to relieve toothache pain, and it aids in stimulating blood circulation throughout the body (Ticoalu et al., 2024).

Clove oil can be obtained from clove buds (clove oil), clove stems (clove stem oil), and clove leaves (clove leaf oil). The main component of clove oil is eugenol, which is widely used in medicine and possesses antioxidant, antimicrobial, antiviral, antinociceptive, analgesic, anesthetic, anti-inflammatory, and wound-healing properties. The essential oil content in clove buds reaches up to 21.3%, with eugenol content ranging from 78–95%; from clove stems around 6%, with 89–95% eugenol; and from clove leaves approximately 2–3%, with eugenol levels between 80–85% (Lestari et al., 2023).

Eugenol is a phenolic compound with the molecular formula $C_{10}H_{12}O_2$, chemically known as 4-allyl-2-methoxyphenol. Its aromatic structure consists of a benzene ring containing a hydroxyl group (–OH), a methoxy group (–OCH_3), and an allyl side chain (–CH_2–CH=CH_2), which together contribute to the distinctive clove aroma and its potent biological activities (Matykiewicz & Skórczewska, 2022). Derivative compounds include methyl eugenol, isoeugenol, and synthetic vanilla. Another widely used modification of eugenol is polyeugenol, derived from the eugenol polymerization process.

Polyeugenol is produced by polymerization by addition. Polyeugenol is a derivative compound of eugenol, often used as a carrier in metal separation methods and for various other applications. This increases the need for polyeugenol in industry and research (Rahim, 2022). In addition to polymerization, the synthesis of other eugenol-derived compounds can also be modified by methods such as epoxidation. Epoxidation is a chemical reaction in which organic compounds containing double bonds (alkenes or alkynes) react with epoxy (oxidant or ethoxyne) to form cyclic compounds called epoxides. Epoxidation generally involves oxidative reagents such as peroxide or other oxygen compounds. This process is often used in organic synthesis to produce a variety of compounds, including a useful epoxide.

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According to Standau et al. (2021), epoxy refers to compounds or polymers containing epoxide groups, particularly epoxy resins, which are used as adhesives, coatings, or composites due to their adhesive properties and chemical resistance. In the industrial context, epoxy is a product of the chemical industry, produced by epoxidizing alkenes (such as ethylene oxide from ethylene) for the synthesis of glycols, polyols, or resins. Due to its superior properties, epoxy is one of the most widely used polymer materials in various industries. According to Dong et al. (2021), epoxy possesses powerful chemical bonds resulting from the three-dimensional network structure formed during curing, making it ideal for applications requiring high mechanical strength, such as industrial floor coatings and composite materials. The adhesive properties of epoxy allow it to bond to various substrates, including metals, ceramics, and plastics, with structural bonding strength, which is why it is extensively used in the automotive and construction industries.

Regarding chemical resistance, epoxy exhibits resistance to acids, bases, and organic solvents. Moreover, epoxy is an effective electrical insulator, critically important in electronic components and electrical insulation materials (Li et al., 2021). Research on eugenol compounds has been conducted by previous researchers, including Amin et al. (2023), who characterized the essential oil composition of Syzygium aromaticum Linn. (clove) using GC-MS and evaluated its antioxidant activity. The GC-MS analysis of Clove essential oil identified 37 chemical compounds, constituting about 99.49% of the total essential oil. Clove essential oil was found rich in eugenol (59.87%), caryophyllene (23.58%), α-selinene (4.67%), α-terpinyl acetate (4.12%), and humulene (3.74%). The Clove essential oil was found to be a potent antioxidant, with a maximum inhibition of 90.94%, comparable to standard antioxidant compounds such as ascorbic acid (92.94%) and gallic acid (87.80%).

Using response surface methodology, Margareta & Wonorahardjo (2023) researched optimizing eugenol isomerization conditions from clove leaf oil. FTIR analysis of isoeugenol samples showed the absorption band of the OH group at 3498.87 cm-1 (std. 3496.94 cm-1) and the absorption band at 2846.93 cm^{-1} (std. 2846.93 cm^{-1} which indicates the group OCH_3 C-C aryl group (aromatic ring) at 1598,99 cm^{-1} (std. 1598,99 cm^{-1}) and CH_3 group at 2935,66 cm_{-1} (std. 2935,66 cm_{-1}) While the results of the analysis with HNMR indicate the position of chemical shift (3) of methyl group ($-CH_3$) as the identity of isoeugenol compound which is 1,812 ppm (std. 1,814 ppm) in the form of doublets for the 3H from $-CH_3$, The identification results also showed that the isoeugenol obtained was more dominant in the form of trans isoeugenol. FTIR spectra showed that the absorption band of trans-isoeugenol (=CH) was 962.4 cm-1, whereas the cis-isoeugenol was 792,7 cm-1. The results of the HNMR spectrum for the peak of H were located at ppm 3=6.1. and a=5,95.

Guntarti et al. (2024) researched the authenticity of clove leaf oil in products (Syzygium aromaticum (L.) Merr. & L. M. Perry) using GC-MS and FTIR methods combined with chemometrics. The constituents' presence in distilled clove oil from GC-MS analysis was eugenol (49.63%), β-Caryophyllene (28.25%), alpha-Humulene (8.92%), alpha-Copaene (2.15%), delta-Cadinene (1.61%), and Caryophyllene oxide (1.50%). Six concentrations were prepared for FT-IR analysis of a mixture of clove leaf oil and turpentine oil, which was estimated by PLS and PCA chemometrics. Turpentine oil was used as a counterfeit, typically added to clove oil products. The PLS analysis of FTIR obtained optimized wavenumbers, 2960-2860 cm^{-1}. The equation y=0.9998x+0.0096 had an R_2 value of 0.9998 and RMSEC, RMSECV, and RMSEP values of 0.22%, 0.76%, and 1.20%, respectively. The PCA analysis can categorize the oil based on the main component types of distilled clove leaf oil, turpentine oil, market oil, products A, B, and C. The results showed product A was in the same quadrant as distilled clove leaf oil. Moreover, no clove leaf oil product had physical and chemical characteristics similar to turpentine oil.

Based on the background, the researcher is interested in researching the eugenol epoxidation of clove oil (Syzygium aromaticum) using an oxidizer H_2O_2 with an H_3PO_4 catalyst because the researchers have never observed the eugenol epoxidation of clove oil (Syzygium aromaticum) using an oxidizer H_2O_2 with an H_3PO_4 catalyst.

Methods

The tools used in this study were 100 ml beaker (Pyrex), 50 ml beaker (*Pyrex*), 10 ml measuring cup (*Pyrex*), hot plate (Robusta), digital scale (*Sonic Electronic*), FTIR (*Bruker Alpha 2 ECO-ATR*), drip pipette, spray bottle, stirring rod, a thermometer (*Pyrex*), spatula, watch glass, mortar and pestle, test tube and tube rack. Meanwhile, the ingredients used were eugenol, 4 ml of H_2O_2 30%, 85% H_3PO_4, 1 ml, 2.5 ml of benzene, 2.5 ml of ethanol, 2.5 ml of chloroform, 2 ml of glycerin, 2 ml of propylene glycol, 3 grams of CMC-Na, and 30 ml of aqua. The sample used was eugenol and clove oil, obtained from a medical device store in Surabaya.

Eugenol epoxy manufacturing

A total of 2 ml of eugenol is placed into the chemical glass of the test tube, followed by the addition of 4 ml of H_2O_2 as an oxidizer, and the mixture is stirred using a stirring rod. Then, slowly add 1 ml of H_3PO_4 solution while stirring until the solution thickens. Next, the solution mixture lasts 15 minutes until two layers are formed. Separate the two layers of solution (Hikmah et al., 2018).

Eugenol epoxy gel manufacturing

Put 3 grams of CMC-Na, which has been weighed into a lump, into 30 ml of aquades at a temperature of 70 °C. Stir until it forms a gel, then add 1 ml of glycerin and 1 ml of propylene glycol while continuing to stir. Then add the eugenol epoxy solution and stir until homogeneous. The epoxy gel is left to sit for 24 hours at room temperature to allow the solvent to evaporate, and then it is washed twice. After washing, the epoxy gel is transferred to the prepared container and left to sit for 24 hours at room temperature (Purgiyanti & Pratiwi, 2018).

Solubility testing

The solubility test was carried out by dissolving 0.25 grams of epoxide each into 3 test tubes. In each test tube, 2.5 ml of organic solvents —benzene, ethanol, and chloroform —were added successively and then mixed. The solubility of each test tube was observed.

Characteristics of eugenol epoxy using FTIR

Eugenol epoxy analysis was performed using the Fourier Transform InfraRed Spectrophotometer (FT-IR) to examine the functional groups contained in eugenol.

Results and Discussion

This study aims to determine the epoxide character of the epoxidation results using a hydrogen peroxide oxidizer (H_2O_2) and a phosphoric acid catalyst (H_3PO_4). It consists of several stages (Hikmah et al., 2018).

Eugenol epoxidation with hydrogen peroxide oxidizer and phosphoric acid catalyst

Epoxidation is carried out by reacting eugenols, oxidizers, and catalysts in a chemical glass. The oxidizers and catalysts used are hydrogen peroxide and phosphoric acid. Epoxidation is carried out by mixing 2 ml of clove oil and 4 ml of H_2O_2 (hydrogen peroxide) into a beaker while stirring. The addition of hydrogen peroxide is an oxidizer in the eugenol synthesis process. Next, slowly add 1 ml of phosphoric acid (H_3PO_4) while stirring until the solution thickens. The addition of phosphoric acid as a catalyst serves to accelerate the eugenol reaction process. After that, let the eugenol solution mixture sit for 15 minutes until 2 layers are formed, where the top layer is light yellow eugenol epoxy and the bottom layer is water.

Next, in a separate container, place 3 grams of CMC-Na, which has been weighed into a lump, along with 30 ml of aquades at a temperature of 70 °C. Stir until a gel forms, then add 1 ml of glycerin and 1 ml of propylene glycol while continuing to stir. Adding glycerin and propylene glycol aims to maintain the gel's moisture, improve the gel's structure, and prevent drying. Next, a solution of eugenol epoxy is added and stirred until homogeneous. Then the epoxy gel formed is left for 1 x 24 hours at room temperature so that the solvent in the epoxy evaporates, facilitating the washing process. Furthermore, the epoxy gel is washed twice with an aqueous solution until the washing water becomes clear. This indicates that the eugenol epoxy gel is free of any remaining impurities. Then the epoxy gel that has been washed is transferred into the gel container that has been prepared and left to sit for 1 x 24 hours to maintain the gel's moisture, improve the gel's structure, and prevent drying. Next, a solution of eugenol epoxy is added and stirred until homogeneous. Then the epoxy gel formed is left for 1 x 24 hours at room temperature so that the solvent in the epoxy evaporates, facilitating the washing process. Furthermore, the epoxy gel is washed twice with aqueous solution until the washing water becomes clear, indicating that the eugenol epoxy gel is free of any remaining impurities. Then the epoxy gel that has been washed is transferred to the gel container that has been prepared and left for 1 x 24 hours at room temperature so that the epoxy gel is free of moisture content.

This study characterized the eugenol polymerization process by observing the solution's thickening and a slow temperature increase, as the reaction was exothermic. In contrast to the research conducted by Hikmah et al. (2018), the polymerization process is characterized by the release of smoke and thickened polymers. The final result of this process is a yellowish-white epoxy compound in gel form that smells like cloves. The results of eugenol epoxidation using hydrogen peroxide oxidizers and phosphoric acid catalysts are shown in **Table 1**.

Table 1. Physical data of eugenol epoxidation results using hydrogen peroxide oxidizer and phosphoric acid catalyst

What is Observed	Eugenol Epoxide
Existed	Gel
Color	Yellowish white
Smell	Clove smell

Solubility testing

Table 2 presents the solubility test results of epoxy compounds obtained from eugenol epoxidation with hydrogen peroxide and phosphoric acid, using benzene, chloroform, and ethanol solutions.

Table 2. Solubility test results

Solution	Solubility
Benzene	Soluble
Chlorophen	Soluble
Ethanol	Insoluble

In this study, the epoxy compounds formed were each weighed to 0.5 grams and then poured into three test tubes of 0.5 grams each. Each test tube is filled with 2.5 mL of benzene, chloroform, and ethanol solvent and shaken until the epoxy is

completely dissolved. After that, the three tubes were observed, and the results from the solubility test showed that polyeugenol compounds were not soluble in ethanol. However, epoxy compounds are highly soluble in chloroform and benzene (Ghumman et al., 2021). Where polyeugenol is not soluble in water, n-hexane, and benzene but in ethanol, ethyl acetate, and chloroform, this is influenced by the polar properties of different solvents, where ethanol and polyeugenol are non-polar, so neither reacts well. At the same time, benzene and chloroform are nonpolar, so that it can dissolve nonpolar epoxy compounds. The factors that affect the solubility of epoxy are the size difference between the epoxy and the solvents, temperature, and volume (Tulegenkyzy et al., 2024).

FTIR characteristic

This study used FTIR (Fourier Transform Infra-Red) spectroscopy to identify the functional groups in the compounds resulting from the eugenol epoxidation reaction, which used hydrogen peroxide as an oxidizer and phosphoric acid as a catalyst. The obtained spectrum showed several distinctive absorption bands that indicated the presence of specific clusters. The results of the eugenol epoxy analysis in this study can be seen in **Figure 1**.

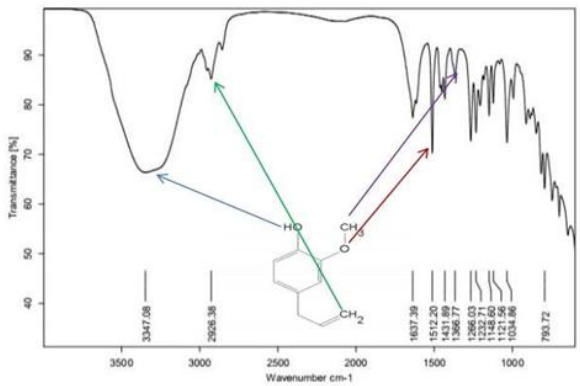

Figure 1. FTIR spectrum

The absorption band at 3347.08 cm^{-1} indicates the presence of a –OH (hydroxyl) group (Dai et al., 2023), which is most likely formed due to the epoxy ring's opening or the alkene group's oxidation. This hydroxyl group is not abundant in pure eugenol, so its presence indicates the occurrence of chemical reactions during the epoxidation process. The band at 2926.98 cm^{-1} absorbs the aliphatic C–H group strain vibrations (Ding et al., 2024), which originate from the methyl and methylene groups in the molecular side chains (Lorenz-Fonfria, 2020). This band is commonly found in organic compounds and indicates the presence of saturated hydrocarbon structures. The strong absorption band at 1637.39 cm^{-1} indicates the presence of an aromatic C=C group, indicating that the benzene ring structure of eugenol is still maintained after the reaction process. In the absorption area of 1470 cm^{-1} - 1350 cm^{-1} is the –CH_3 bend group. The bands in the 1266.07 cm^{-1} to 1018.66 cm^{-1} show vibrations from the C–O–C group, characteristic of both epoxy and ether groups. The presence of this band indicates that the epoxidation process is successfully underway. If the epoxy ring is opened, hydroxyl and ether groups will be formed, showing absorption in this area (Kamairudin et al., 2021). Furthermore, a vinyl group is on the 910.2 cm^{-1} absorption tape.

The FTIR spectrum results showed the success of the epoxidation reaction, which was characterized by the appearance of epoxy groups at wave numbers of 1266.07 cm^{-1} to 1018.66 cm^{-1} and hydroxyl groups at wave numbers of 3347.08 cm^{-1}. The absence of significant changes in the aromatic absorption band suggests that eugenol's main structure remains intact. The reaction, using hydrogen peroxide as an oxidizer and phosphoric acid as a catalyst, does not damage the structure of the aromatic ring. Instead, it adds the desired polar group through epoxidation.

FTIR results comparison

In the analysis of chemical compounds, the FTIR (*Fourier Transform Infrared*) spectrum is an important method for identifying functional groups based on the absorption of infrared waves by molecules (Gong et al., 2024). Each functional group has a typical wavenumber range that can be compared with the literature data to ensure the suitability of the structure of the compound being analyzed. In this section, a comparison was made between the results of the FTIR spectrum of the synthesized eugenol sample and the literature data as a reference. The aim is to validate the existence of the main functional groups present in eugenol structures, such as hydroxyl (–OH) groups, ethers (–OCH_3), alkenes (C=C), and aliphatic groups (–CH_3 and –CH_2–). By comparing the number of waves in the analysis spectrum with those in the literature, conclusions can be drawn about the success of the synthesis process and the purity of the resulting compounds. This comparison is an important step in ensuring that the FTIR results accurately represent the chemical structure of eugenol and are in accordance with scientific standards (Agrawal et al., 2022). A comparison of the wavelengths in the FTIR spectrum of eugenol epoxide is shown in **Table 3**.

Table 3. Comparison of the FTIR spectrum with previous research (Hikmah et al., 2018)

Number of Waves (cm^{-1})		Function Clusters
Hikmah et al. (2018)	Spectrum Results	
3448.72	3347.08	O-H
3518.1	2926.98	C-H stretch
1604.7 - 1512.1	1637.38	C=C aromatic
2908.6 - 2839.2	1470 - 1530	-CH_3 bend
1265.3	1255.07 – 1018.66	C-Eter
910.40 - 995.27	910.2	Vinyl Cluster

Based on the results of the comparative analysis of the FTIR spectrum, the synthesis results, as well as the research conducted by Hikmah et al. (2018), did not show significant differences. Major functional groups such as –OH, C–O ether, and aromatic C=C are detected at comparable wavelengths. For example, the absorption band for the -OH group appears at 3347.08 cm^{-1}, which is comparable to the study conducted at a wave number of 3448.72 cm^{-1}. In addition, the aromatic C=C group was detected at 1737.39 cm^{-1}, similar to the value obtained in the 1604.7 – 1512.1 cm^{-1} range. The C–O ether group was also identified through the absorption of 1255.07 – 1018.66 cm^{-1}, close to the value obtained 1265.3 cm^{-1}. The synthesized compounds, obtained by reacting clove oil with hydrogen peroxide oxidizers (H_2O_2) and using phosphoric acid catalysts (H_3PO_4), exhibit absorption characteristics that represent the functional groups in eugenol.

Conflict of Interest

The authors have confirmed that no competing interests exist concerning the posting of this paper.

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