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Synthesis and Characterization of Polyeugenol from Clove Leaf Oil (Syzygium aromaticum)-Derived Eugenol Using a Nitric Acid Catalyst

Andi Bulqiah Nur[1]*, Satria Putra Jaya Negara[2], Ahmad Fudhail[3], Muharram[4]
[1,2,3,4]Department of Chemistry, Faculty of Mathematics and Natural Sciences, State University of Makassar
Corresponding Author: Andi Bulqiah Nur, andi.bulqiah@unm.ac.id

A R T I C L E I N F O

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A B S T R A C T

Eugenol from clove leaf oil has the potential to be used as a raw material for bio-based polymers. This study evaluated the effect of varying NaOH mass on the composition of the eugenol fraction obtained from purification, as well as the characteristics of the polymerization product using HNO_3. Clove leaf oil was purified via acid–base extraction using 3–7 g of NaOH in 100 mL of solution at 50 °C for 30 minutes. The resulting fractions were analyzed using GC-MS and then reacted with 65% HNO_3. The products were characterized using FTIR and solubility tests. The highest eugenol area percentage, 94.18%, was obtained with 5 g of NaOH. The polymerization process produced a blackish-brown gel that was soluble in ethanol, slightly soluble in chloroform, and insoluble in n-hexane. The FTIR spectrum showed functional groups associated with eugenol, but the formation of polymer chains still requires further characterization

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INTRODUCTION

Eugenol is a natural phenolic compound with potential as a raw material in the development of bio-based polymer materials. This potential is supported by its chemical structure, which contains phenolic hydroxyl, methoxy, and allyl groups, thereby enabling various modification reactions to produce derivative compounds with different characteristics. The presence of these reactive groups has been leveraged in the development of monomers and polymer materials through various synthetic approaches (Molina-Gutiérrez et al., 2019; Morales-Cerrada et al., 2021). The use of eugenol as a precursor is also continuing to expand, including in the synthesis of polycarbonates derived from renewable resources (Sengoden et al., 2024). The diversity of these synthetic pathways makes eugenol an attractive natural material for further development, particularly in the production of materials with chemical and physical characteristics that can be tailored through reaction conditions.

Clove leaf oil (Syzygium aromaticum) is a natural source of eugenol that contains various accompanying components, particularly sesquiterpene compounds such as caryophyllene. The presence of these components necessitates a purification process to obtain an eugenol fraction that can be used as a starting material in chemical synthesis. One separation method that can be applied is acid–base extraction, which takes advantage of the weak acidity of eugenol's phenolic hydroxyl group. The addition of sodium hydroxide (NaOH) allows the formation of sodium eugenolate, which is more soluble in the aqueous phase, while acidification converts it back to eugenol in its neutral form. The effectiveness of the separation is influenced by process conditions, including the amount of base used and the separation method applied. The use of various types of bases in the isolation of eugenol has been studied and has yielded fractions with different compositions (Handayani et al., 2023). Differences in extraction and distillation conditions are known to produce eugenol fractions with varying compositions and yields (Sugiharto et al., 2024). Therefore, optimizing purification conditions is a critical aspect in obtaining eugenol fractions as starting materials for subsequent synthesis processes.

The conversion of eugenol into polymeric materials can be achieved through various reaction pathways using different catalysts. The polymerization of eugenol using concentrated sulfuric acid has been studied using starting materials obtained through redistillation, extraction, and fractionation (Sudarlin & Haryadi, 2017). The use of BF_3 as a catalyst has also been developed to produce polyeugenol, which is subsequently utilized as a component in graphene-based anticorrosion coating materials (Prasetya et al., 2023). In addition to these two catalysts, concentrated nitric acid (HNO_3) has been used in eugenol reactions, yielding a gel-like product that is blackish-brown in color. However, the presence of absorption bands associated with unsaturated groups in the FTIR spectrum indicates that the transformation of reactive groups has not yet been fully completed (Pakaya & Abram, 2020). This suggests that the formation of products with different physical characteristics does not always indicate complete polymerization. Furthermore, HNO_3 is both a strong acid and an oxidizing agent; therefore, the possibility of other reactions occurring during the synthesis process

must be taken into account when evaluating the structure of the resulting product.

Previous research has laid the groundwork for the use of NaOH in the purification of eugenol and the application of various acid catalysts in the synthesis of eugenol-based materials. However, these two stages present distinct analytical challenges. In the purification process, changes in the amount of NaOH must be evaluated based on the composition of the fractions obtained, whereas in the reaction stage, product formation must be assessed through its chemical and physical characteristics. Research on reactive extraction has primarily focused on evaluating the conditions for the separation and recovery of eugenol, while studies on polymerization have centered on synthesis conditions and the characteristics of the resulting products (Handayani et al., 2023; Pakaya & Abram, 2020; Sugiharto et al., 2024). In this context, this study examines changes in the compositional profile of the eugenol fraction as the amount of NaOH used in the purification process varies, and then evaluates the initial characteristics of the product resulting from HNO_3 treatment. This approach allows both stages to be analyzed based on their respective experimental results, so that changes in the composition of the starting material are not automatically considered evidence of successful polymer formation.

Based on these considerations, this study aims to evaluate the effect of varying amounts of NaOH 3, 4, 5, 6, and 7 g per 100 mL of solution on the compositional profile of the eugenol fraction obtained from the purification of clove leaf oil (Syzygium aromaticum) using GC-MS. The eugenol fraction obtained was then used as the starting material in a reaction with concentrated HNO_3, and the reaction products were characterized via FTIR analysis and solubility testing. This study presents a comparison of the relative composition of eugenol under various NaOH treatments as well as the initial characteristics of the products after the reaction with nitric acid. These results provide an experimental basis for evaluating both stages of eugenol processing and serve as a reference for further studies on the formation of eugenol-based polymer materials.

THEORETICAL REVIEW

Principles of Eugenol Separation via Acid–Base Extraction

Eugenol is a weak phenolic acid. Under basic conditions, the phenolic hydroxyl group can undergo deprotonation to form sodium eugenolate, which is more readily distributed into the aqueous phase. Acidification converts this salt back to eugenol in its neutral form. The composition of the final fraction is determined not only by acid–base reactions but is also influenced by phase separation, washing, and processing conditions (Handayani et al., 2023; Sugiharto et al., 2024).

The Reactivity of Eugenol in the Polymerization Process

The allyl group in eugenol has the potential to participate in polymer chain formation reactions, while its aromatic and phenolic groups may also undergo transformations under certain conditions. The polymerization

mechanisms reported for other acid-catalyzed systems do not automatically apply to HNO_3 because nitric acid can trigger other reactions under appropriate conditions. Therefore, product evaluation must consider the functional groups that are retained as well as the possibility of new groups forming (Morales-Cerrada et al., 2021; Pakaya & Abram, 2020).

METHODOLOGY

Tools and Materials

The equipment used in this study included laboratory glassware, a separating funnel, a standard funnel, a thermometer, an analytical balance (Ohaus Adventurer), a stopwatch, a hot plate (WiseStir MSH 20-D), a magnetic stirrer, an oven, aluminum foil, as well as Gas Chromatography–Mass Spectrometry (GC-MS) and Fourier Transform Infrared (FTIR) instruments. The main material used was clove leaf oil (Syzygium aromaticum) obtained from an oil distillery in Banyorang Village, Bantaeng Regency, South Sulawesi, Indonesia. Other chemicals included 65% p.a. nitric acid (HNO_3), sodium hydroxide (NaOH), p.a. sulfuric acid (H_2SO_4), p.a. methanol (CH_3OH), 98% ethanol (C_2H_5OH), chloroform ($CHCl_3$), ethyl acetate ($C_4H_8O_2$), petroleum ether, n-hexane, distilled water, and Whatman filter paper.

Research Procedures

The study was conducted in three main stages: the purification of eugenol from clove leaf oil, the polymerization of eugenol using concentrated nitric acid, and the characterization of the reaction products through solubility testing and FTIR analysis.

1. Purification of Eugenol from Clove Leaf Oil

Eugenol was purified through acid–base extraction using varying amounts of NaOH: 3, 4, 5, 6, and 7 g. Each amount of NaOH was dissolved in distilled water to a final volume of 100 mL using a 100-mL volumetric flask. A total of 20 mL of clove leaf oil was mixed with the NaOH solution for each treatment variation, then stirred using a magnetic stirrer on a hot plate at 50 °C for 30 minutes. The mixture was then transferred to a separatory funnel and allowed to stand for approximately 1 hour until two phases formed. The aqueous phase containing sodium eugenolate was then separated and used in the acidification step.

The acidification process was carried out by adding a 1.5 N H_2SO_4 solution prepared by diluting 4 mL of H_2SO_4 with distilled water to a volume of 100 mL. The mixture was stirred using a magnetic stirrer for 30 minutes, then returned to a separatory funnel and allowed to stand for approximately 4 hours until two phases formed. The resulting eugenol fraction was then washed with distilled water in a 1:1 volume ratio to remove any residual salt. After washing, the eugenol fraction was heated on a hot plate to reduce the water content until a clear liquid was obtained. The purified eugenol was then stored in vials for GC-MS analysis and used as the starting material in the polymerization stage.

2. Polymerization of Eugenol Using Nitric Acid

The purified eugenol was placed in a 50-mL beaker and then reacted with 65% concentrated HNO_3. The HNO_3 was added gradually in 0.25-mL increments every 30 minutes for 2 hours. During the reaction, the mixture underwent a color change and an increase in viscosity until a brick-red, gel-like product formed. After the reaction had proceeded for 2 hours, the process was stopped by adding 1 mL of methanol. The resulting product was washed with petroleum ether, then neutralized with distilled water and dried in an oven at 50 °C. The dried product was then weighed and used for solubility testing and FTIR analysis.

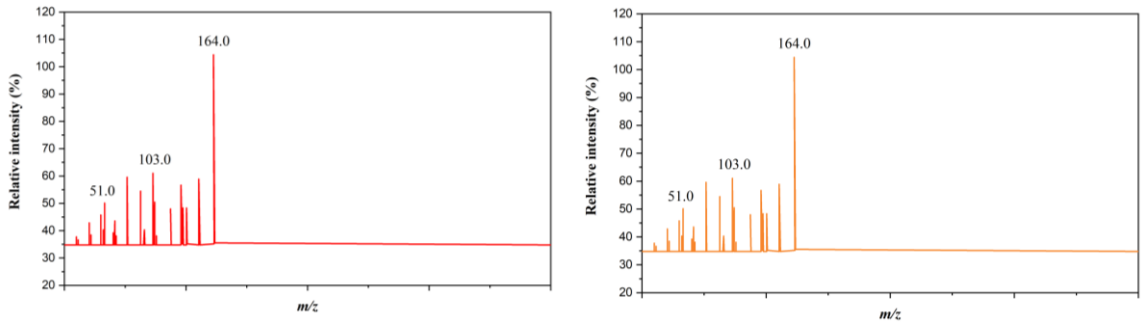
3. Characterization of Polymerization Products

Product characterization was performed through solubility testing and FTIR analysis. Solubility testing was conducted by placing 0.5 g of each product into separate test tubes, to which 5 mL of a solventn-hexane, ethanol, or chloroform was added. The mixtures were gently shaken, and solubility was then assessed visually based on the presence of residual solids and the degree of homogeneity of the mixture. Functional group characterization was performed using an FTIR instrument on products obtained from treatments with 5 g and 6 g of NaOH. The resulting spectra were used to identify the absorption bands of functional groups in the products of the reaction between eugenol and HNO_3.

RESEARCH RESULTS

Results of Eugenol Purification and GC-MS Analysis

Eugenol was purified via acid–base extraction using NaOH masses of 3, 4, 5, 6, and 7 g. The acid–base extraction yielded a fraction containing eugenol that appeared as a yellowish-brown, relatively clear liquid after washing and water removal. GC-MS analysis revealed eugenol along with other components. The reported compound components for each treatment are shown in Tables 1–5, while the mass spectra associated with the eugenol peaks are presented in Figure 1.



(a) (b)

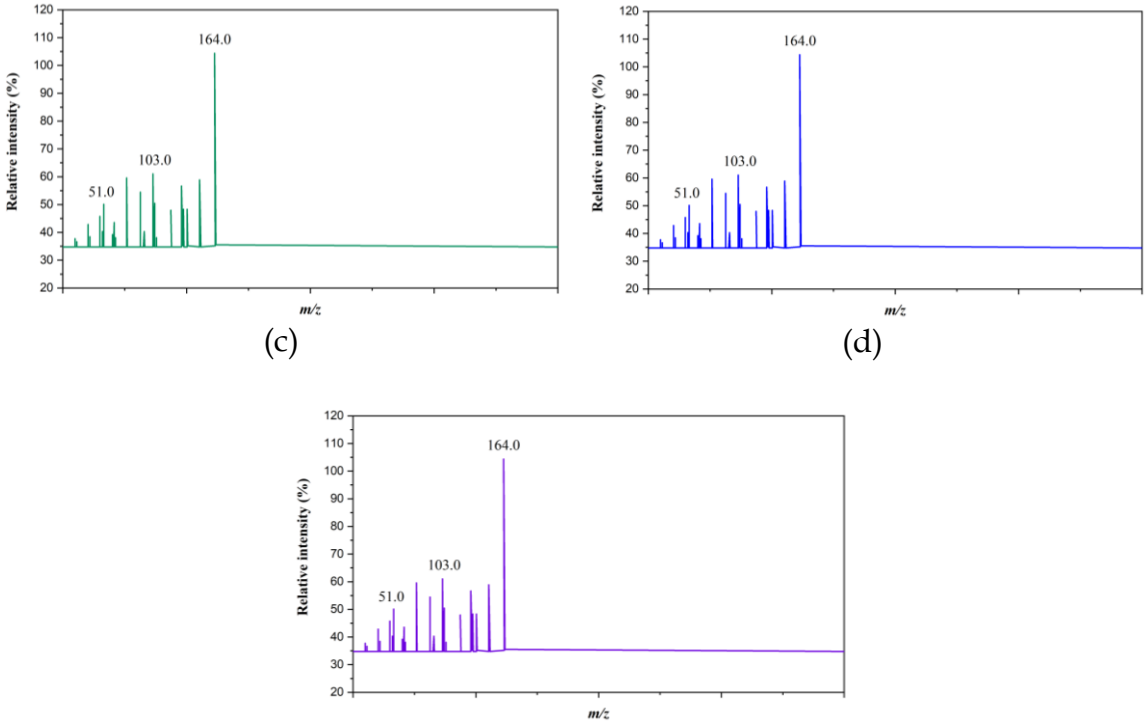


Figure 1. Comparison of the mass spectra of eugenol obtained from the purification of clove leaf oil (Syzygium aromaticum) using varying amounts of NaOH, namely (a) 3 g, (b) 4 g, (c) 5 g, (d) 6 g, and (e) 7 g.

The percentage of the eugenol area when 3 g of NaOH was used was recorded at 37.36% and increased to 91.36% when 4 g of NaOH was used. The highest value was obtained in the 5 g NaOH treatment, at 94.18%. When the amount of NaOH was increased to 6 g, the percentage of eugenol area decreased slightly to 92.79%, then further decreased to 80.70% with 7 g of NaOH. Based on these results, the use of 5 g of NaOH yielded the highest proportion of eugenol area among the five conditions tested. In addition to eugenol, the purified fraction still contained a number of other compounds, primarily caryophyllene, α-humulene, and caryophyllene oxide. In the 5 g NaOH treatment, the percentages of caryophyllene and caryophyllene oxide were recorded at 3.19% and 1.17%, respectively. The presence of these components indicates that the purification process has not yet yielded eugenol as a single compound.

Table 1. Composition of Compounds Obtained from Purification with 3 g of NaOH, as Determined by GC-MS

No.	Identified compounds (tentative)	Area (%)
1	Eugenol	37,36
2	3-Allyl-6-methoxyphenol	19,12
3	Phenol	26,27
4	trans-Caryophyllene	7,28
5	alpha-Humulene	1,61
6	alpha-Caryophyllene	0,75
7	Dihydrojasmone	0,71

No.	Identified compounds (tentative)	Area (%)
8	Globulol	0,70
9	Ferruginol	0,79
10	1,3-Difluoroazulene	2,05
11	5,8-Dimethoxy-4-(2-nitrophenyl)	0,76
12	3,5'-Diallyl-2,2'-dihydroxy-3,3'-dimethpoxybiphenyl	1,55
13	Garciniaxanthone	1,05

Table 2. Composition of Compounds Obtained from Purification with 4 g of NaOH, as Determined by GC-MS

No.	Identified compounds (tentative)	Area (%)
1	Eugenol	91,36
2	Caryophyllene	3,50
3	alpha-Humulene	0,55
4	Caryophyllene oxide	0,90
5	Ferruginol	0,59
6	Dimethyl 1,3,5,6-tetramethyl-	0,77
7	4-Methylene-1-4',6'-dimethylpyrimidin	0,56
8	3,5'-Diallyl-2,2'-dihydroxy-3,3'-dimethpoxybiphenyl	0,76

Table 3. Composition of Compounds Obtained from Purification with 5 g of NaOH, as Determined by GC-MS

No.	Identified compounds (tentative)	Area (%)
1	Eugenol	94,18
2	Caryophyllene	3,19
3	Caryophyllene oxide	1,17
4	Ajmaline	0,70
5	3,5'-Diallyl-2,2'-dihydroxy-3,3'-dimethpoxybiphenyl	0,76

Table 4. Composition of Compounds Obtained from Purification with 6 g of NaOH, as Determined by GC-MS

No.	Identified compounds (tentative)	Area (%)
1	Eugenol	92,79
2	Caryophyllene	3,09
3	Caryophyllene oxide	1,97
4	alpha.-Hydroxycaryophylla 4,8 ,8(13)-diene	0,70
5	. 1-Deuterio-1-tetralol	0,74
6	Presilphiperfolane-9,15-epoxide	0,71

Table 5. Composition of Compounds Obtained from Purification with 7 g of NaOH, Based on GC-MS

No.	Identified compounds (tentative)	Area (%)
1	3-Formyl-N-methyl-9-[phenylethynyl]	0,47
2	Eugenol	80,7
3	Benzene	3,63
4	Caryophyllene	4,46
5	.alpha-Humulene	0,88
6	Cycloheptasiloxane	4,26
7	Caryophyllene oxide	2,28
8	alpha.-Hydroxycaryophylla	0,78
9	Silane	0,92
10	Caryophylenol-II	0,84
11	caryophylla-3,8	0,78

The percentage of eugenol area when using 3 g of NaOH was 37.36%; it then increased to 91.36% in the 4 g NaOH treatment and reached a peak of 94.18% in the 5 g NaOH treatment. This percentage decreased to 92.79% with 6 g of NaOH and to 80.70% with 7 g of NaOH. Thus, the use of 5 g of NaOH yielded the highest proportion of eugenol area among the five treatments tested.

In the fraction obtained from purification using 5 g of NaOH, the percentages of caryophyllene and caryophyllene oxide were 3.19% and 1.17%, respectively. Meanwhile, in the 3 g NaOH treatment, 3-allyl-6-methoxyphenol was reported at 19.12% and phenol at 26.27%. The identity of certain minor components, particularly the names of truncated or uncommon compounds, requires re-examination of the original GC-MS report prior to publication.

Results of Eugenol Polymerization Using HNO_3

The gradual addition of 65% HNO_3 to the eugenol fraction was accompanied by a color change to brick red and an increase in viscosity. After the addition of methanol, washing, and drying, a gel-like product was obtained that was blackish-brown in color, glossy, and had a characteristic clove odor.

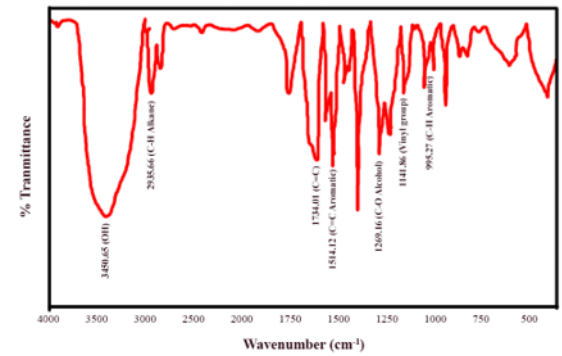
Solubility Test Results

Solubility tests were conducted to determine the behavior of the reaction product of eugenol with HNO_3 in solvents with different characteristics. Based on visual observation, the polymerization product is soluble in ethanol, slightly soluble in chloroform, and insoluble in n-hexane. These observations are the results of qualitative solubility tests, not quantitative measurements.

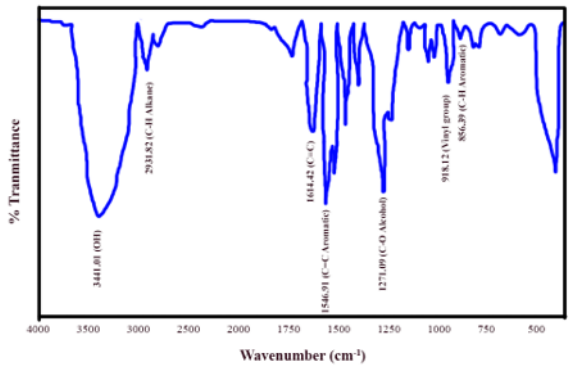
FTIR Characterization Results

FTIR characterization was performed on the eugenol polymerization products from the purification fraction using 5 g and 6 g of NaOH. The FTIR spectrum for the 5 g NaOH treatment showed absorption bands at 3450.65; 2935.66; 1734.01; 1514.12; 1269.16; 1141.86; and 995.27 cm^{-1}, whereas the 6 g NaOH treatment showed absorption bands at 3441.01; 2931.82; 1614.42; 1546.91;

1271.09; 918.12; and 865.30 cm^{-1}. Both spectra show absorption bands associated with O–H, aliphatic C–H, C–O, and aromatic framework groups, as well as bands in regions potentially associated with vinyl groups. A comparison of the FTIR spectra of the two samples is presented in Figure 2.



(a)



(b)

Figure 2. Comparison of the FTIR spectra of the eugenol polymerization products from the fractions obtained through purification using NaOH, namely (a) 5 g and (b) 6 g.

DISCUSSION

Characterization of Clove Leaf Oil as a Raw Material

Clove leaf oil (Syzygium aromaticum) used in this study was obtained from an essential oil distillery in Banyorang Village, Bantaeng Regency, South Sulawesi. Analysis using Gas Chromatography–Mass Spectrometry (GC-MS) identified the presence of eugenol along with several other components, including caryophyllene. This composition is consistent with the general characteristics of clove essential oil, which contains phenylpropanoid and sesquiterpene compounds. Eugenol and β-caryophyllene are two components commonly found in clove essential oil, although their proportions may vary depending on the source and characteristics of the material analyzed (Trifan et al., 2021).

The presence of components other than eugenol is an important consideration before clove leaf oil is used as a starting material in chemical synthesis. Eugenol has a phenolic hydroxyl group that allows for its separation via acid–base reactions, whereas caryophyllene is a sesquiterpene compound that

lacks such a phenolic group. These differences in chemical properties are exploited in the purification process to obtain fractions with a higher proportion of eugenol. However, since the percentage by area of each oil component prior to purification has not been presented quantitatively, the relative increase in eugenol compared to the initial raw material cannot yet be calculated directly.

Purification of Eugenol from Clove Leaf Oil

Eugenol was purified through acid–base extraction using NaOH masses of 3, 4, 5, 6, and 7 g in 100 mL of solution, respectively. This process takes advantage of the weak acidity of the phenolic hydroxyl group in eugenol, which reacts with NaOH to form sodium eugenolate. The formation of this salt increases the solubility of eugenol in the aqueous phase, thereby facilitating its separation from the more nonpolar oil components. The principle of reactive extraction using NaOH was also applied in the purification of clove oil by Sugiharto et al. (2024). The reaction for the formation of sodium eugenolate from eugenol and NaOH is shown in Figure 3.

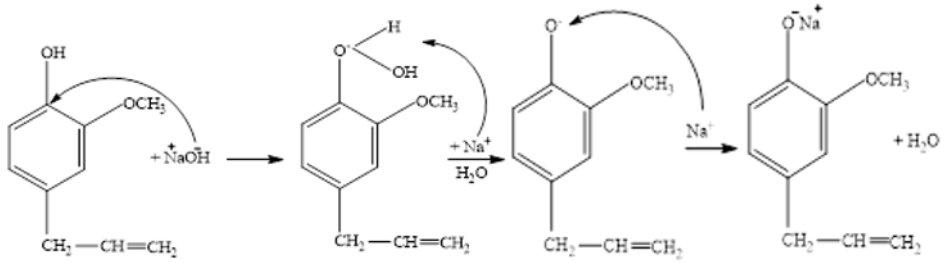


Figure 3. The reaction for the formation of sodium eugenolate from eugenol using NaOH.

Sugiharto et al. (2024) compared the purification of eugenol via reactive extraction using NaOH and fractional distillation. Under extraction conditions using 2 N NaOH, the reported eugenol content reached 75.33% with a product yield of 40%, whereas fractional distillation yielded an eugenol content of 97.02% with a yield of 28.25%. These differences demonstrate that the separation method and conditions play a role in determining the composition and quantity of the product obtained. However, the results of the two methods cannot be directly compared with this study without considering the differences in experimental conditions and the measurement bases used.

GC-MS Analysis of Eugenol from the Purification Process

The increase in the percentage of the eugenol fraction resulting from the use of 3 to 5 g of NaOH can be attributed to extraction conditions that influence the formation of sodium eugenolate and the distribution of eugenol into the aqueous phase. However, increasing the amount of NaOH does not always result in a higher proportion of eugenol in the final fraction. The decrease in the percentage of eugenol area observed with 6 and 7 g of NaOH indicates that increasing the amount of base beyond 5 g does not yield higher results based on the analytical parameters used.

These changes cannot yet be explained by a single specific mechanism. The composition of the purified fractions may be influenced by the formation of sodium eugenolate, the efficiency of phase separation, the acidification step, or the loss of components during the washing process. Furthermore, the GC-MS area percentage is a relative value compared to the total signal of the detected compounds; therefore, a decrease in the eugenol area does not necessarily mean that the absolute amount of eugenol obtained has also decreased. Without yield data, experimental replicates, and quantitative validation, the use of 5 g of NaOH cannot yet be declared as the optimal purification condition across the board.

In addition to eugenol, the purified fraction still contains a number of other compounds, primarily caryophyllene, α-humulene, and caryophyllene oxide. In the 5 g NaOH treatment, the percentage areas of caryophyllene and caryophyllene oxide were recorded at 3.19% and 1.17%, respectively. The presence of these components indicates that the purification process has not yet yielded eugenol as a single compound. Sesquiterpene components have also been found in analyses of clove essential oils from various sources; therefore, their presence in the separation fraction is consistent with the chemical characteristics of the starting material (Kiki, 2023; Neagu et al., 2024).

In the 3 g NaOH treatment, the GC-MS results also indicated 19.12% 3-allyl-6-methoxyphenol and 26.27% phenol. The compound 3-allyl-6-methoxyphenol is known as chavibetol, a positional isomer of eugenol that has the same molecular formula but a different substituent arrangement. The structural similarity between these two compounds must be taken into account because it can result in mass fragmentation patterns that are similar. Therefore, the identification of compounds in the 3 g NaOH treatment needs to be reevaluated based on mass spectra and retention times, as well as retention indices when available. The percentage area of chavibetol cannot be directly combined with that of eugenol without confirming the identity of each peak.

Overall, variations in the amount of NaOH are related to changes in the relative composition of the eugenol fraction obtained. The highest percentage of eugenol area in the 5 g NaOH treatment provides the basis for selecting the fraction for the next reaction stage. However, a quantitative determination of purification efficiency still requires information on the amount of eugenol obtained and its composition prior to the purification process.

Polymerization of eugenol using a nitric acid catalyst

Similar physical characteristics were observed in the study by Pakaya & Abram (2020), which used concentrated HNO_3 in an acetic acid medium for the eugenol polymerization reaction. The resulting product was a glossy, blackish-brown gel that still exhibited absorption bands associated with unsaturated groups in the FTIR analysis. These similarities in physical appearance can serve as an initial point of comparison, but they are not sufficient to conclude that the products obtained have identical chemical structures or molecular weights.

Structurally, eugenol contains an allyl group with a double bond that enables various chemical transformation reactions, including those that can potentially lead to polymer chain formation. In addition to the allyl group, the

aromatic ring and the phenolic hydroxyl group can also undergo modification under certain reaction conditions. The diversity of these reactive centers forms the basis for the development of various eugenol-based materials through different synthetic pathways (Morales-Cerrada et al., 2021; Sengoden et al., 2024).

One of the pathways that has been studied in eugenol polymerization is the cationic mechanism involving the formation of active species on the allyl group. Conceptually, cationic polymerization involves the stages of initiation, propagation, and termination, with the formation of new bonds during chain growth. However, the sequence of these stages in the eugenol–HNO_3 system in this study could not be directly determined based on changes in the color and viscosity of the product. Therefore, the polymerization mechanism should be viewed as a theoretical approach that requires support from structural characterization.

The cationic polymerization of eugenol has been studied using a BF_3 catalyst. Prasetya et al. (2023) employed this approach to produce polyeugenol, which was subsequently developed as a component of graphene-based composites for anticorrosive coating applications. Product formation was supported by changes in the FTIR spectrum in the allyl group absorption region as well as by characterization of the resulting material. However, the reaction mechanism using BF_3 cannot be directly applied to explain the entire reaction process using HNO_3 because the two reactants have different chemical characteristics.

In addition to being a strong acid, HNO_3 is an oxidizing agent that facilitates other transformations, including oxidation and nitration reactions under appropriate conditions. Therefore, color changes, increased viscosity, and gel formation cannot yet be used as sole evidence of successful eugenol polymerization. Confirmation of the product's structure requires characterization that can identify changes in functional groups and the formation of molecules with higher molecular weights.

Solubility testing

Solubility tests were conducted to determine the behavior of the product resulting from the reaction of eugenol with HNO_3 in solvents with different characteristics. Based on visual observation, the product is highly soluble in ethanol, slightly soluble in chloroform, and insoluble in n-hexane. These differences indicate variations in the interactions between the product and each solvent.

The basic structure of eugenol contains phenolic hydroxyl and methoxy groups that allow for intermolecular interactions with certain organic solvents. Ethanol is a polar, protic solvent capable of forming hydrogen bonds with the phenolic hydroxyl groups, so these interactions can contribute to better solubility of the product. In contrast, n-hexane is a nonpolar solvent with limited ability to interact with polar groups in the product's structure. This solubility pattern is similar to the findings of Pakaya and Abram (2020), who obtained a reaction product of eugenol using HNO_3 that was soluble in ethanol and slightly soluble in

chloroform. However, this similarity in solubility behavior cannot be used to confirm that the two products have identical molecular structures.

The solubility of a polymer material can also be influenced by its molecular weight, chemical composition, and the presence of cross-links. Research by Lan et al. (2024) on polyeugenol-based nanoparticles shows that the development of materials with cross-linked structures requires a more comprehensive evaluation of their characteristics to understand the properties of the resulting products. Meanwhile, (Ngadiwiyana et al., 2024) used solubility testing along with FTIR characterization and molecular weight analysis in the evaluation of eugenol–allyl eugenol copolymers.

Thus, the solubility test results in this study provide preliminary information regarding the product's affinity for organic solvents. However, visual observation alone is not sufficient to distinguish the polymer product from oligomers, unreacted monomers, or mixtures of other compounds resulting from chemical transformations.

Characterization of Polieugenol Using FTIR

The band at 1734.01 cm^{-1} is one of the absorption peaks that warrants attention because it lies in the carbonyl group (C=O) vibration region. This absorption band may indicate the presence of components with different structures or chemical changes following treatment with HNO_3. One possibility to consider is the formation of compounds containing carbonyl groups as a result of side reactions. However, this interpretation cannot be confirmed without comparison to the initial eugenol spectrum and additional characterization.

The absorption at 995.27 cm^{-1} is also an important factor in evaluating changes in the allyl group. This region may be related to out-of-plane C–H vibrations in the terminal alkene group. In a study (Windarti et al., 2023), the decrease in absorption intensity around 914 and 994 cm^{-1}, accompanied by the disappearance of the double-bond band at approximately 1637 cm^{-1}, was used as an indicator of allyl group transformation during the synthesis of polyeugenol. However, in this study, the presence of a band at 995.27 cm^{-1} did not allow for the determination of the degree of conversion because a quantitative comparison with the pre-reaction eugenol spectrum was not available.

Although both products exhibit a number of similar functional groups, there are differences in several spectral regions. One of these is a band at 918.12 cm^{-1} that may be associated with the vibration of the terminal alkene group. The presence of this band indicates that unsaturated groups may still be present in the reaction products. This finding is similar to the results of a study conducted by Pakaya & Abram (2020), who also detected an absorption band associated with unsaturated groups in the reaction product of eugenol treated with concentrated HNO_3.

Interpretation of the region at 918.12 cm^{-1} must be done with caution because the FTIR fingerprint region may contain several closely spaced vibrations. Furthermore, the absorption at 1546.91 cm^{-1} cannot yet be confirmed as originating solely from aromatic ring vibrations. Given that HNO_3 is an oxidizing agent and can participate in nitration reactions, the possible presence of byproducts from side

reactions must be considered. However, identifying such structural changes requires supporting absorption bands and a more comprehensive spectral analysis.

The differences in the FTIR spectra between the two samples reveal variations in the characteristics of the functional groups in the eugenol polymerization products resulting from treatments with 5 g and 6 g of NaOH. These variations are indicated by differences in the positions of absorption bands in several spectral regions, suggesting differences in the chemical characteristics of the resulting products. However, these differences are not sufficient to conclude that variations in the amount of NaOH during the purification stage directly affect the success or degree of eugenol polymerization; therefore, additional characterization is needed to confirm this relationship.

Evaluation of Polieugenol Formation Based on Characterization Results

The formation of polieugenol must be evaluated based on changes in the allyl group and evidence of the formation of larger molecular structures. The disappearance or reduction of absorption bands associated with double bonds may indicate the transformation of reactive groups, but such changes do not necessarily mean that polymer chains have formed. Therefore, FTIR analysis must be supplemented by other characterization techniques capable of providing information on the structure and molecular weight of the product.

A more comprehensive characterization approach has been applied in research on eugenol-based materials. Ngadiwiyana et al. (2022) used FTIR, ^{1}H-NMR, and membrane property analysis to characterize sulfonated eugenol–allyleugenol copolymers. The study by Windarti et al. (2023) also utilized FTIR and molecular weight measurements to support the identification of polyeugenol, while Lan et al. (2024) developed polyeugenol-based nanoparticles using a broader approach to characterizing material structure and properties. The use of these various analytical techniques highlights the importance of complementary evidence in confirming the identity of polymer products.

In addition to structural characterization, the development of polyeugenol into a functional material requires an evaluation of properties relevant to the intended application. Windarti et al. (2023), for example, utilized polyeugenol as a component in calcium phosphate cement composites and evaluated the physicochemical characteristics and biological properties of the resulting material. These developments demonstrate eugenol's potential as a starting material for material development; however, its application potential cannot be directly inferred based solely on synthesis results or the identification of functional groups. Based on the FTIR results of this study, both samples still exhibited functional groups associated with the basic structure of eugenol, along with some differences in absorption bands following HNO_3 treatment.

The presence of bands that may be associated with terminal alkene groups indicates that the transformation of unsaturated groups cannot yet be said to have occurred completely. However, the available data are insufficient to determine the degree of conversion or the formation of polymer chains.

Overall, variations in the amount of NaOH resulted in differences in the relative composition profiles of the eugenol fractions, with the highest percentage by area 94.18% observed in the 5 g NaOH treatment. The reaction using HNO_3 produced a gel-like product with specific solubility characteristics and functional groups. Although these results provide information regarding the purification process and chemical transformation of eugenol, a direct relationship between the composition of the purified fractions and the formation of polyeugenol cannot yet be confirmed.

CONCLUSIONS AND RECOMMENDATIONS

A. Conclusions

Based on the research that has been conducted, it can be concluded that:

1. The purification of clove leaf oil (Syzygium aromaticum) using different amounts of NaOH resulted in variations in eugenol composition, with the highest GC-MS area percentage of 94.18% observed when 5 g of NaOH was used.
2. The reaction of eugenol with concentrated HNO_3 yields a glossy, gel-like product that is blackish-brown in color and has a characteristic clove odor. The product is soluble in ethanol, slightly soluble in chloroform, and insoluble in n-hexane. FTIR characterization revealed the presence of functional groups associated with the structure of eugenol as well as several differences in absorption bands in the reaction product.

B. Recommendation

Based on the research findings, it is recommended that the eugenol purification process be improved by optimizing the extraction and separation conditions to obtain eugenol fractions with a better composition. In addition, the characterization of the polymerization products should be supplemented with additional analytical methods to support the identification of the structure and characteristics of the resulting poly-eugenol.

FURTHER STUDY

Future research could focus on optimizing the eugenol polymerization process by varying the amount of HNO_3 catalyst, reaction time, and synthesis conditions to evaluate their effects on the characteristics of the resulting product. Further characterization using Nuclear Magnetic Resonance (NMR) and Gel Permeation Chromatography (GPC), as well as a comparison of the FTIR spectra of eugenol before and after the reaction, is necessary to gain deeper insight into changes in the chemical structure and molecular weight distribution of the product. Future research could also focus on evaluating the physicochemical properties and potential applications of polyeugenol as a natural-based material.

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