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SYNTHESIS AND CHARACTERIZATION OF ALPHA-TERPINEOL FROM TURPENTINE-DERIVED *PINUS MERKUSII* WASTE USING *p*-TOLUENE SULFONIC ACID CATALYST

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Abstract

Pine tree sap can be distilled to produce gondorukem and turpentine with alpha-pinene as the primary compound (65-85%). The alpha-pinene contained in turpentine can be reacted with alcohol to produce alpha-terpineol which has a higher selling value. The study aimed was to synthesize alpha terpineol from turpentine derived from processing pine tree sap with varying percentages of p- toluenesulfonic acid (p-TSA) catalyst (5, 10, 15%), reaction time (2, 4, 6 hours), and by comparing extracted and commercial turpentine as starting materials. The alpha-terpineol yields for a 10% catalyst were 53,515%; 54.28%; 62.08% and 15% catalyst amounting to 89.24%; 89.68%; 91.95%, as well as 15% catalyst with turpentine extraction of 92.96%. The densities for 10% alpha terpineol catalyst were 0.969 g/mL, 0.971 g/mL, and 0.969g/mL and with 15% catalyst were 0.969 g/mL, 0.972 g/mL, and 0.971 g/mL. The results of FTIR analysis for alpha terpineol samples with 10 and 15% catalyst for 2, 4 and 6 hours showed the presence of O-H and C-O functional groups. GC-MS analysis for alpha terpineol levels was obtained at 25.88% (10% catalyst) and 24.52% (15% catalyst). Several component compounds were obtained, namely 1-Cyclopentene-1-methanol, alpha, 4,5-tetramethyl-, trans.

INTRODUCTION

Indonesia's extensive forest resources include Pinus merkusii, a native pine species essential for ecological conservation and economic activity. This species produces sap that can be distilled into two main products: gondorukem (solid resin) and turpentine (liquid distillate). Turpentine, contains 65–85% α-pinene, serves as valuable raw material for creating high-value derivatives like alpha-terpineol. This monoterpene alcohol is widely used in cosmetics, perfumes, pharmaceuticals, and household products due to its pleasant scent, low toxicity, and antifungal properties [1], [2]. Turpentine, a distillate of pine sap, is a clear liquid, while gondorukem, the distillation residue, is a solid with a yellow to dark yellow hue. Turpentine oil contains several compound components, including α-pinene, β-pinene, 3-carene, and limonene. The 65-85% compound component contains α-pinene, 1-3% β-pinene, 10-18% 3-carene, and 1-3% limonene. Alpha-pinene can be synthesized into a product with high selling value, such as terpineol. Terpineol is a group of terpenoid group compounds that have low toxicity. Terpineol itself has many uses, namely used in the cosmetic field as a fragrance, pharmaceutical field for antifungal, disinfectants, and in personal care products like shampoos, detergents, and soaps due to its pleasant aroma [2][3]. Terpineol, with its lilac-like aroma, is found in various essential oils and widely used in the fragrance industry. In addition to its traditional use, terpineol is extensively utilized in the pharmaceutical industry, for its antioxidant, anti-inflammatory, anti-proliferative, anti-microbial, and analgesic effects [4].

Alpha-terpineol is synthesized from α-pinene using acid catalysts, as demonstrated in previous studies by two step synthesis. Daryono (2015) synthesized alpha-pinene into alpha-terpineol with sulfuric acid catalyst using temperature and volume variations in ethanol. The result of alpha-terpineol was obtained at 57.05% with a percent yield of 67.79% at 70 °C with 135 mL of ethanol [5]. Sekerová et al. treated montmorillonite K10 with several acids (H_2SO_4, HCl, HNO_3, and MCA) to catalyze the hydration of alpha-pinene [6]. Meng Zhong et al (2022) synthesized alpha-pinene into alpha terpineol by using a hydroxy acid catalyst ratio at 70 ℃ and 12-15 hours, alpha terpineol was obtained at 46.9% [7]. Using sulfuric acid as a catalyst poses serious environmental pollution and complicates downstream processes. To address the strong corrosion associated with sulphuric acid in alpha-terpineol synthesis, Román-Aguirre et al. used hydrochloric acid, acetic acid, oxalic acid, and monochloroacetic acid (MCA) as catalysts for the direct synthesis of terpineol from alpha-pinene [8]. The study showed that MCA was the most effective catalyst, with a 90% conversion of alpha-pinene; with a terpineol selectivity of 65%. Meng et al., 2022 prepared a carbon–silicon composite solid acid, via carbonization and sulfonation as the catalyst, using MCA as the promoter for the hydration of alpha-pinene to terpineol [9]. Wijayati et al. prepared a catalyst by impregnating TCA onto Y-zeolite molecular sieve and synthesized a TCA/ZHY catalyst (Si/Al = 3.25) for alpha-pinene hydration [10,11]. But chloroacetic acids, including MCA and TCA, are highly corrosive and harmful to humans (TCA is a class 2B carcinogen) and, thus, it is important to find non-toxic and environmentally friendly organic acids, with similar catalytic activity for alpha-pinene hydration. In this study, the synthesis of alpha-terpineol was carried out directly from turpentine in the distillation of pine tree sap using pTSA catalyst or p-toluenesulfonic acid. The p-toluenesulfonic acid catalyst is a white solid that is soluble in water and has homogeneous properties. The use of p-toluenesulfonic acid catalyst simplifies the neutralization process [12]. The pTSA catalyst can be reused so that it is more environmentally friendly and cost-effective. In addition, ethanol solvent is used due to its affordability.

EXPERIMENT

Material

The chemicals used were pine tree sap, aluminum granule, turpentine oil, p-toluenesulfonic acid p.a (Merck KGaA): 5, 10, 15%, Ethanol p.a (Merck), 5% technical NaOH, and distilled water.

Instrumentation

The synthesis results will be characterized using Fourier-Transform Infrared Spectroscopy (Thermo scientific Trace 1310 ISQ LT MS) with ATR method and Gas Chromatography-Mass Spectrometry (Aglient Technologies).

Procedure

Processing Pine Tree Resin into Turpentine

10 g of aluminum granules are placed in a three-neck flask, followed by the addition of 100 g of water and 100 g of pine tree resin. The three-neck flask is then placed in a heating mantle. The distillation process is carried out with the heating mantle, and the temperature is set to 110°C. After that, 100 mL of water is added every 40 minutes until a total of 400 mL is reached. The collected distillate is then separated using a separating funnel. The turpentine obtained is stored in a vial.

Synthesis of Alpha Terpineol

The synthesis of alpha terpineol refers to the research by Daryono (2015) [5]. The first step involves weighing 15 g of turpentine oil, 105 mL of ethanol, and a catalyst of p-toluenesulfonic acid (b/v), which are then poured into a three-neck flask. The reflux process is carried out at a temperature of 60°C with a stirring speed of 370 rpm. The crude oil product is extracted and neutralized to pH 7 using 5% NaOH. Catalyst concentrations of 5%, 10%, and 15% are used, with reaction times of 2, 4, and 6 hours.

Characterization

The characterization of turpentine and alpha terpineol includes testing of physical properties in the form of density and characterization with Spectrophotometer Fourier Transform InfraRed (FTIR) using the ATR and Gas Chromatography-Mass Spectrometry (GC-MS). In this section requires references if the methodology is already used in a previous study. Indicate some information about whether the method has been modified.

RESULT AND DISCUSSION

Processing Pine Tree Resin into Turpentine

The pine tree sap used in this study has a quality of 1B. According to SNI 7837:2016, pine tree sap 1 B has a white to cloudy brown color. Pine tree sap 1B has a moisture content + dirt content (%) of 14 to 18%. Pine tree sap is harvested by tapping on the trunk of a pine tree and can be processed to produce turpentine, which contains essential compounds in the form of terpenoids [2].

The hydrodistillation method is used to extract turpentine from the pine tree sap, employing water as the solvent. In this process, dean stark is added to facilitate the separation of turpentine compounds from water as turpentine is insoluble in water. Aluminum granules (10 g) are added during distillation process to prevent bumping due to increased pressure from rising heating temperatures. From the distillation results, two phases are formed: turpentine (0.865 g/mL) is in the upper phase and the by-product, water (0.996 g/mL), is in the lower phase. These phases are separated using a separating funnel. The collected turpentine, a clear liquid, is stored in a vial [4], [13].

The following results of the percentage yield of turpentine obtained from the pine tree sap distillation process can be seen in **Table 1**.

According to Kurniawan et. al (2018), pine tree sap consists of Gondorukem (60%) and turpentine oil (10-17.5%) [14]. The rest of the pine resin content is water, litter, and fine dirt. The percent yield value increases with the addition of water and distillation time. The longer the time used, the more interaction between the solvent and the raw material. Thus, enhancing the diffusion of active compounds from the sap cells [15] [16]. However, the percent yield of turpentine is not significant with the addition of water. This is due to several factors such as quality, impurities storage and conditions during the distillation process may contribute to the low or inconsistent yield of turpentine.

Tabel 1. Data on percentage yield of turpentine.

No	Volume of Water (mL)	Sap Mass (g)	Turpentine Mass (g)	Yield (%)	Time (min)
1	400	100.683	10.380	10.31	131.4
2	600	100.158	12.110	12.10	197
3	1200	100.547	14.705	14.62	433

Synthesis of Alpha Terpineol

Alpha terpineol was synthesized using reflux method, with variation of catalyst concentration and time variation which aims to see the optimum alpha terpineol product. The reflux method is considered very effective and efficient for separating the components in the resulting mixture. This research uses a pTSA catalyst which has a white solid form that dissolves in water and has homogeneous properties. The use of *p*TSA acid catalyst offers several advantages, including ease of neutralization, cost-effectiveness, environmental friendliness, and high catalytic activity [7]. In the synthesis process, commercial turpentine and turpentine directly extracted from pine sap were used as the primary raw materials without separating the alpha-pinene content, which constitutes 65–85% of turpentine [2]. This direct approach simplifies the synthesis process.

During the synthesis process using ethanol which acts as a solvent aims to dissolve alpha pinene. The crude product will be neutralized with 5% NaOH which aims to change the acidity of the solution to reach a neutral solution. During the neutralization process there are two phases: the upper phase in the form of alpha terpineol and the lower phase in the form of water. The reaction mechanism begins with the protonation of the *p*TSA catalyst described in **Figure 1** as follows. This mechanism is almost the same as the proposed mechanism by Yang, H., et al 2009 using acetic acid and chloroacetic acid as a catalyst and by Zitova, K 2021. [17,18].

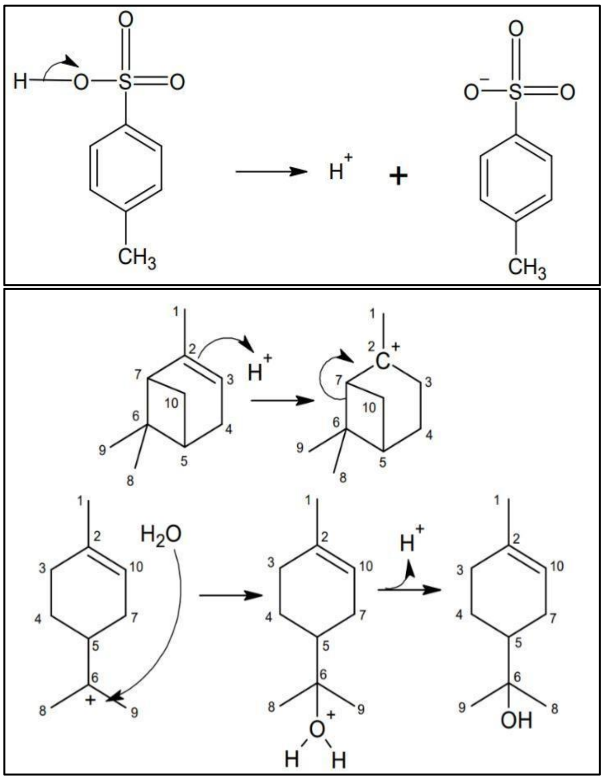


Figure 1. Mechanism synthesis of terpineol.

From the reflux process of alpha terpineol synthesis, the percentage yield for alpha terpineol was obtained during the neutralization process, which can be seen in **Table 2.**

From the results of the synthesis of alpha terpineol, the percent yield obtained during the neutralization process above increased with higher catalyst concentrations and longer reaction times. This indicates that both factors play a significant role in enhancing the reaction efficiency. Additionally, a longer reaction time required a larger volume of neutralizing agent. In addition, at 5% catalyst with variations of 2, 4, and 6 hours no yield or crude product is produced. This is due to the small percentage of catalyst so no reaction has occurred during the synthesis process.

Table 2. Yield % of a-terpineol synthesis results from commercial turpentine and extracted turpentine.

Turpentine	% Catalyst (b/v)	Time (Hours)	%Yield
Commercial	10	2	53.51
		4	54.28
		6	62.08
	15	2	89.24
		4	89.68
		6	91.95
Extract	15	6	92.96

Characterization

Physical Characterization

The results of the synthesis of alpha terpineol were tested for density using a pycnometer. The following data on alpha terpineol density results are described in **Table 3.**

From the results of density data obtained for alpha terpineol on 10% and 15% catalysts with a reaction time of 2, 4, and 6 hours ranged between 0.969-0.971 g/mL. The data results obtained are different from the reference alpha terpineol density of 0.930 g/mL [4]. The higher density of the synthesized product indicates that it is not completely pure or contain other compound components. The turpentine used in the synthesis contains other compounds that do not react with the catalyst or water. When using the increasing amount of ethanol, the density of alpha terpineol produced is getting smaller. This is following the research of Daryono et al, 2015 who reported that increasing ethanol concentrations during synthesis resulted in decreased density. This occurs because the main component in turpentine is alpha-pinene contributes to the reduction in density as its proportion increases in the mixture [4].

Table 3. Alpha terpineol density observation data.

Catalyst (%)	Time (hours)					
	2		4		6	
	Mass (g)	Sample (g/mL)	Mass (g)	Sample (g/mL)	Mass (g)	Sample (g/mL)
10	18.247	0.969	18.267	0.971	18.243	0.969
15	18.269	0.969	18.297	0.972	18.270	0.971
15 (Extract)					18.273	0.971

Characterization with FTIR

FTIR spectroscopy uses the ATR method to identify the wavenumber values associated with bond vibrations. The resulting data can be used to determine the functional groups of the tested compounds. The results of processing pine tree sap into turpentine were tested using FTIR. Based on **Figure 2**, it is evident that turpentine oil extracted through commercial methods exhibited similar vibrational peaks. A summary of the functional group analysis present in the extracted and commercial turpentine is provided in **Table 4.**

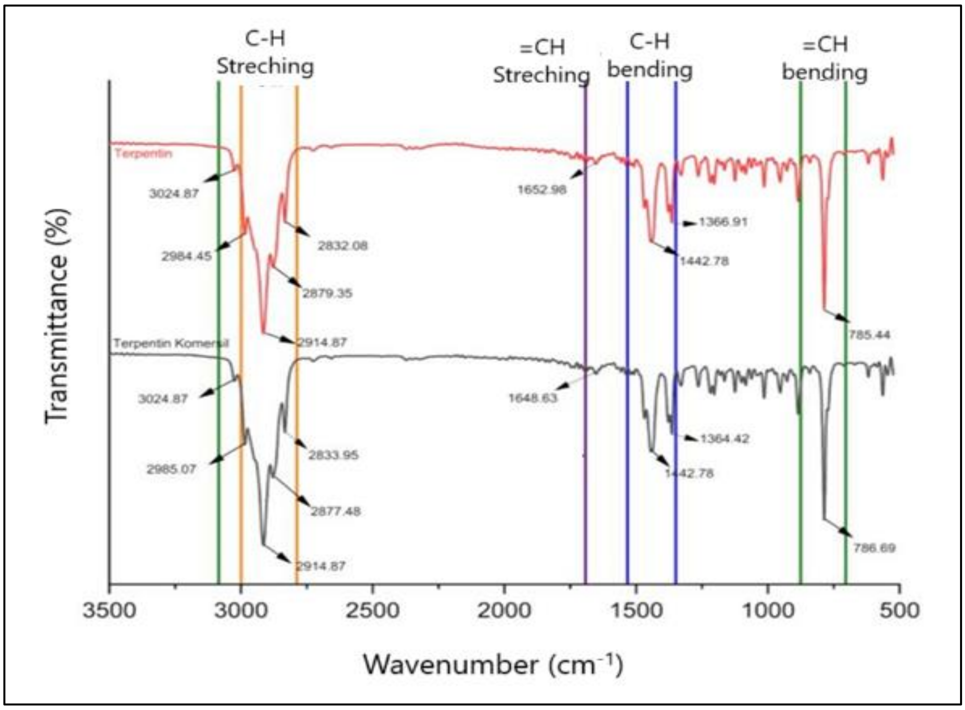


Figure 2. Comparison of FTIR spectrum of turpentine extraction and commercial.

Tabel 4. FTIR spectrum analysis results of turpentine extraction and commercial results.

Vibration of bonds	Types of vibration	Wavenumber (cm^{-1})	
		Turpentine	Commercial Turpentine
C=C Alkene	Stretching	3024	3024
		2984	2985
C-H Aliphatic	Stretching	2879	2877
		2832	2833
C=C alkene	Stretching	1652	1648
C-H Aliphatic	Bending	1442	1442
		1366	1364
C=C alkene	Bending	785	786

From the results of the FTIR analysis, it can be concluded that the turpentine contains the compound alpha-pinene, as indicated by the functional groups in the FTIR test results. The structure of alpha-pinene is shown in **Figure 3**. The presence of bond vibrations in both extracted and commercial turpentine oil indicates the presence of the turpentine compound alpha-pinene, characterized by numerous C-H bonds and the presence of alkene functional groups in the FTIR spectrum. FTIR analysis provides an accurate identification and confirmation of alpha-pinene based on the distinctive vibrational patterns of the chemical bonds involved in the molecule.

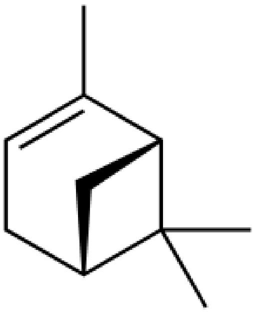


Figure 3. Structure of alpha-pinen.

The comparison of FTIR spectra results for alpha-terpineol with a 10% catalyst at different time intervals (2, 4, 6 hours) is shown in **Figure 4**.

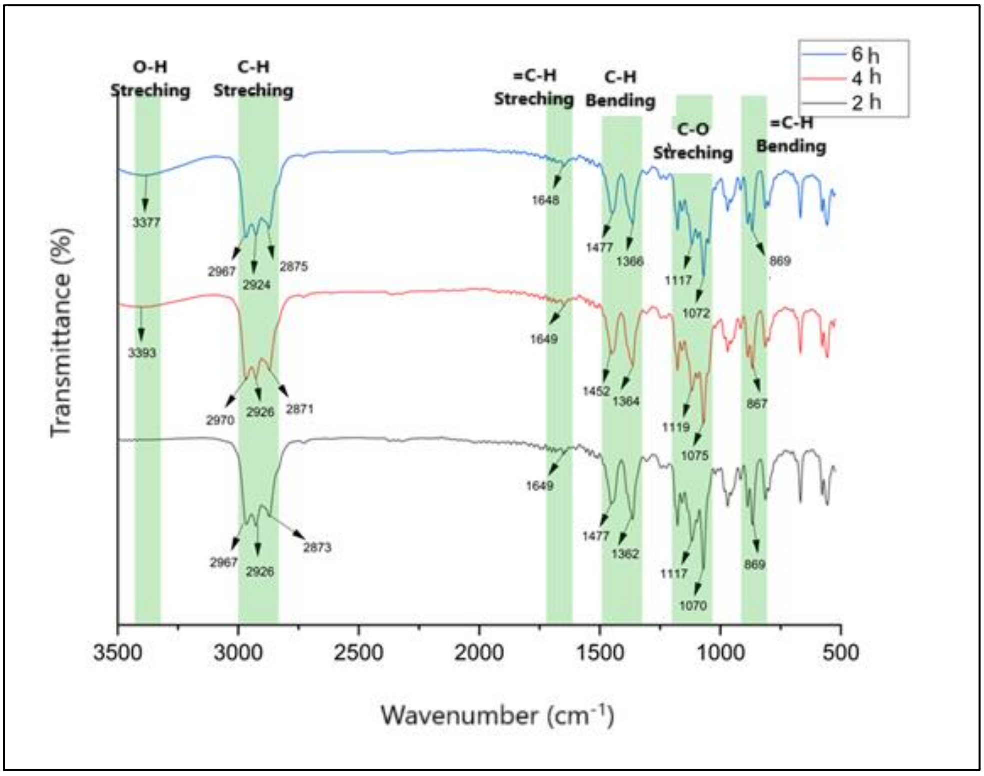


Figure 4. FTIR spectrum of 10% alpha terpineol with time of reaction is 2, 4 and 6 hours.

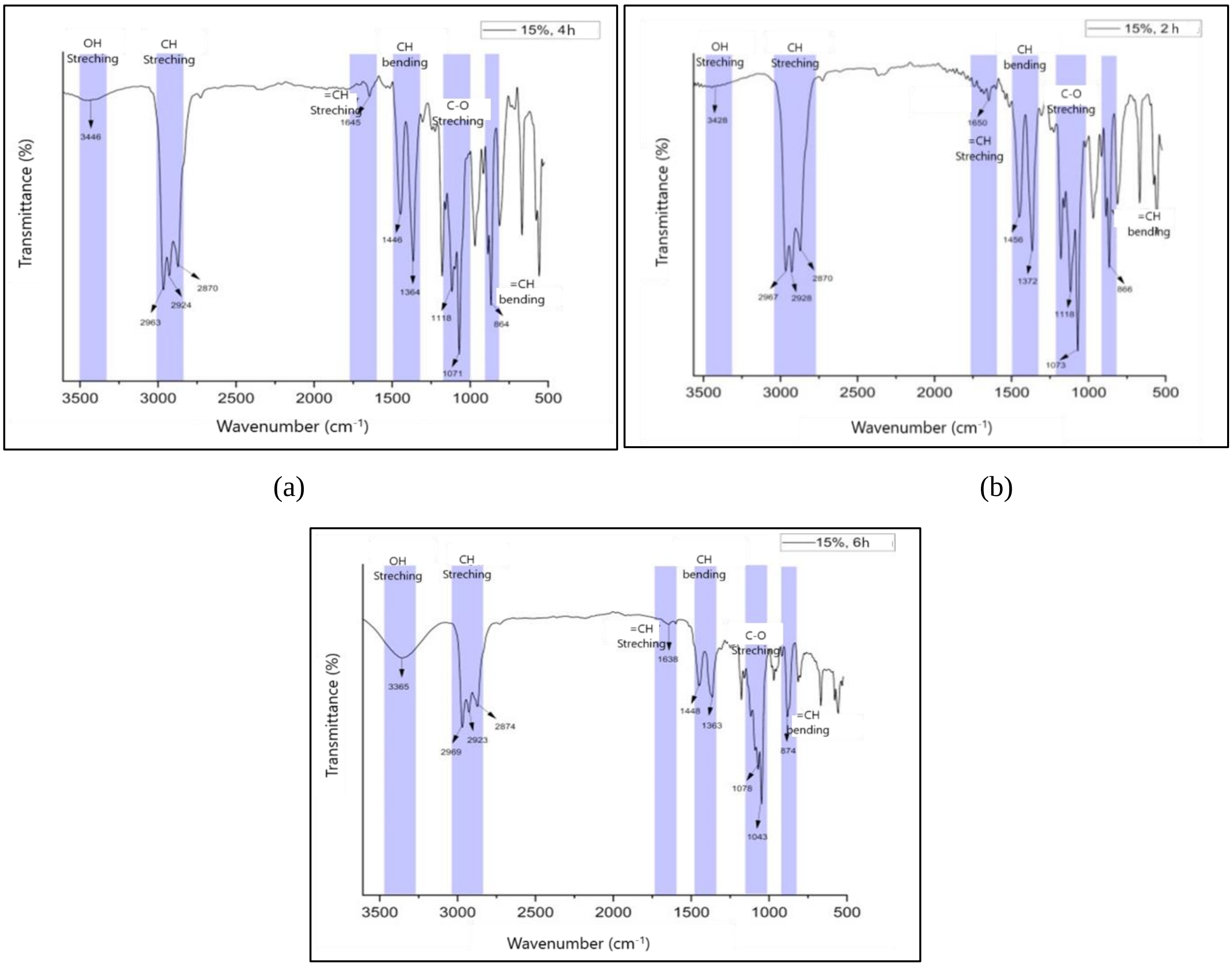
Based on the FTIR spectrum, the results of the analysis of the types of functional groups present in alpha- terpineol with a 10% catalyst can be seen in **Table 5**.

Table 5. Results of FTIR spectrum analysis of alpha terpineol with 10% catalyst.

Vibration of Bonds	Types of Vibration	Wavenumber (cm^{-1})		
		Terpineol 10%, 2 h	Terpineol 10%, 4 h	Terpineol 10%, 6 h
O-H	Stretching	-	3393	3377
		2967	2970	2967
C-H	Stretching	2873	2926	2924
		2926	2871	2875
C=C-H	Stretching	1649	1649	1648
C-H	Bending	1477	1452	1477
		1362	1364	1366
C-O	Stretching	1177	1119	1117
		1070	1075	1072
C=C	Bending	869	867	869
C=C	Bending	866	864	874

The comparison of FTIR spectra results for alpha-terpineol with a 15% catalyst at different time intervals (2, 4, 6 hours) is shown in **Figure 5**. Based on the FTIR spectrum, the results of the analysis of the types of functional groups present in alpha- terpineol with a 15% catalyst can be seen in **Table 6**.

FTIR results for six samples-using 10% and 15% catalysts with reaction times of 2, 4, and 6 hours show that good results are found in 15% catalyst percent with 6 hours reaction. The more percent of catalyst used and the longer the reaction time, the more effective alpha terpineol products are produced. This is characterized by the formation of O-H stretching vibrations and C-O stretching vibrations. The low percent transmission observed at these wavenumbers suggests higher absorption, further corroborating the effective formation of alpha-terpineol under these conditions.



(a) (b)

(c)

Figure 5. FTIR Spectrum of 15% Alpha Terpineol with Time of Reaction is a) 2 h, b) 4 h and c) 6 h.

Table 6. Results of FTIR spectrum analysis of alpha terpineol with 15% catalyst.

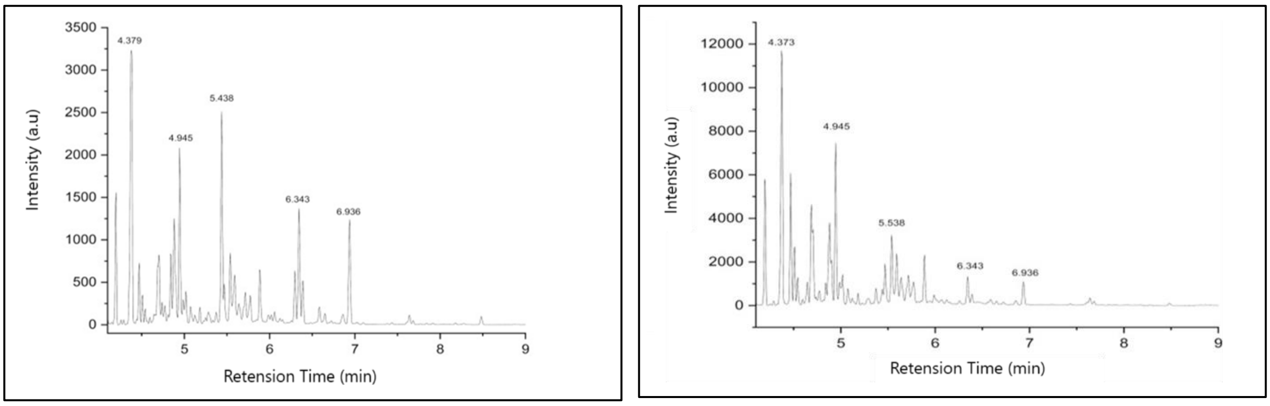
Vibration of Bonds	Types of Vibration	Wavenumber (cm^{-1})			Reference
		Terpineol 15%, 2 h	Terpineol 15%, 4 h	Terpineol 15%, 6 h	[19, 20]
O-H	Stretching	3428	3446	3365	3383
C-H	Stretching	2967	2963	2969	2966
		2870	2924	2923	
		2928	2870	2874	
C=C-H	Stretching	1650	1645	1638	1630
C-H	Bending	1456	1446	1448	1465
		1372	1364	1363	
C-O	Stretching	1118	1118	1078	1120
		1073	1071	1043	

Characterization with GC-MS

The characterization of turpentine derived from pine tree sap and the synthesized alpha-terpineol was conducted using GC-MS analysis. The GC-MS chromatograms for extracted and commercial turpentine are shown in **Figure 6**.

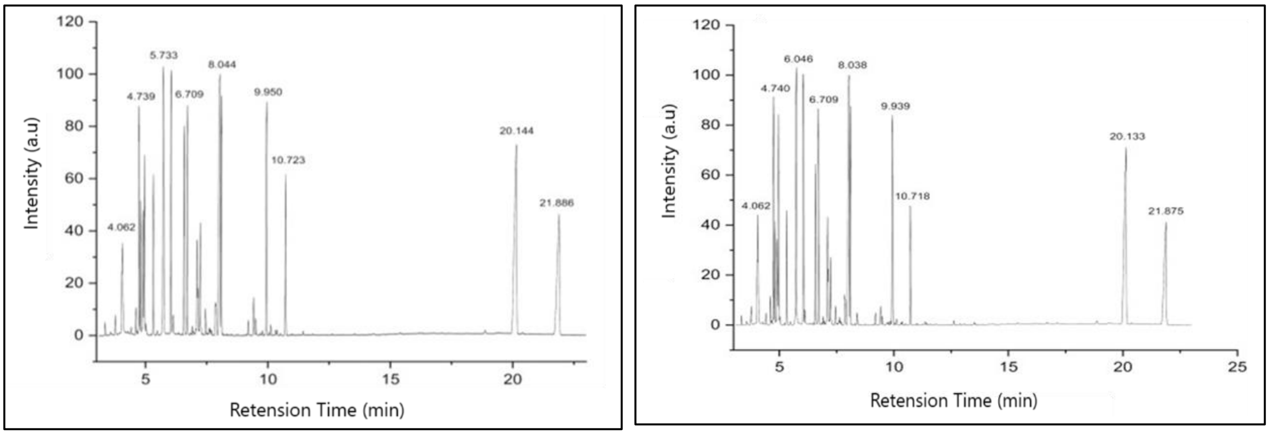
The results show that the compounds have several different peaks with different retention times corresponding to the compounds present in the turpentine. The extracted turpentine exhibited similar chromatographic characteristics to the commercial turpentine, with no significant differences. The most dominant peak with a retention time of 4.379 and 4.373 minutes corresponds to alpha-pinene, constituting 25.88% and 24.52%. The lower alpha-pinene levels can be attributed to factors such as chromatography conditions, including column temperature and the type of column used, which influence the separation of sample components. Alpha terpineol from turpentine synthesis and commercial with *p*-TSA acid catalyst of 15% in 6 hours reaction time is shown in **Figure 7**.

The chromatogram of synthesized using extracted and commercial alpha-terpineol,~~-~~are not much different with the same characteristics reflecting similar compositions, namely alpha-pinene with the largest main component in turpentine. In the chromatogram obtained several peaks, indicating the presence of other compounds and the incomplete purity of the alpha terpineol. From the data, alpha terpineol with turpentine extraction appeared at a retention time of 5,026 minutes. Meanwhile, alpha terpineol with commercial turpentine appeared at a retention time of 5,023 minutes. The peak is the compound 1-Cyclopentene-1-methanol, alpha.,4,5-tetramethyl-, trans.



(a) (b)

Figure 6. Comparation GC chromatogram of turpentine extracted (a) and commercial (b).



(a) (b)

Figure 7. Comparation GC chromatogram of alpha terpineol synthesis from turpentine extract (a) and commercial (b).

The mass spectra of alpha terpineol synthesized using extracted and commercial turpentine (15% catalyst, 6-hours reaction) are presented in **Figure 8**.

The molecular mass of alpha terpineol is 154. From the results of the mass spectrum obtained, the retention time results obtained for alpha terpineol products derived from commercial turpentine and extraction using 15% catalyst percent for a reaction time of 6 hours have the same characteristics with retention times of 5.026 and 5.023 minutes which are known to have a molecular ion (m/z) of 154. The peak at a molecule of 154 produced is low or small. This MS spectra has a fragmentation pattern of alpha terpineol with m/z 154. Fragmentation of the alpha terpineol molecule produces a fragment with m/z 136, which is a fraction of the alcohol. Alpha terpineol releases a water molecule from molecular ion m/z 154, resulting in a fragment with m/z 136. The m/z 136 molecular ion releases CH_3, forming a new fragment with m/z 121, which will then release CH_2 to form m/z 107. After that, the release of C_2H_2 produces a fraction with m/z 81. The fraction with m/z 59 $((CH3)_2C^{+}OH)$ results from the release of molecular ions at 95. The base peak of m/z 41 resulted from a fraction of m/z 136. The m/z 136 ion releases molecule 93 and forms an ion with m/z 43, which then releases H_2 to produce m/z 41. This fragmentation had the same results as those in the Pineda-Rios study in 2021 [21] and Vila, M, 2018 [22].

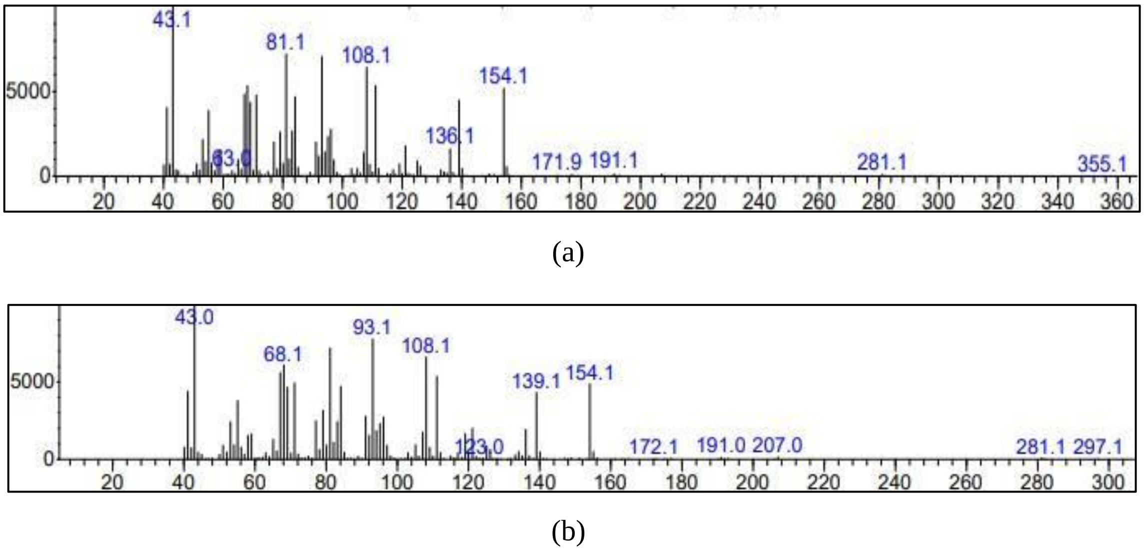


Figure 8. (a) Mass spectrum of alpha terpineol synthesis from turpentine commercial and (b) mass spectrum of alpha terpineol synthesis from turpentine extracted.

CONCLUSION

The synthesis of alpha terpineol using ethanol as a solvent and *p*- toluenesulfonic acid as catalysts was successfully conducted with 10% and 15% catalysts concentration. The percent yield for 10% catalyst at 2, 4, 6 hours were 53.51%; 54.28%; 62.08% while for 15% catalyst were 89.24%; 89.68%; 91.95%, and with 15% catalyst using turpentine extraction for 6 hours obtained 92.96%. Density measurements on alpha terpineol with a catalyst of 10% for 2, 4, 6 hours obtained successively 0.969 g/mL; 0.971 g/mL; 0.969 g/mL, for a catalyst of 15% were 0.969 g/mL, 0.972 g/mL, 0.971 g/mL, and the density of alpha terpineol catalyst 15% with turpentine extraction for 6 hours obtained 0.971 g/mL. FTIR analysis revealed with a catalyst of 10% for 4 and 6 hours showed the presence of O-H stretching vibrations and C-O stretching vibrations, for 2 hours there were only C-O stretching vibrations. Meanwhile, for the 15% catalyst samples at all reaction times, consistent O-H and C-O stretching vibrations were observed, indicating the successful formation of alpha-terpineol. GC-MS characterization confirmed the synthesis of alpha-terpineol, with retention times of 5.026 minutes for extracted turpentine and 5.023 minutes for commercial turpentine, both using 15% catalyst for 6 hours.

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