



Conversion of α -Pinene into More Valuable Compounds by Using Rahims 3rd Generation Catalyst

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Abstract. The hydration reaction of α -pinene and the isomerization reaction of α -pinene were studied with Rahims 3rd generation catalyst. The aim of this study was to determine the level of terpineol compounds formed in the hydration reaction of α -pinene and the product compounds formed in the isomerization reaction of α -pinene with Rahims 3rd generation catalyst. The results showed that the hydration reaction of α -pinene with the polymer supported catalyst gave terpineol compounds with a concentration of 8.12% and a yield of 5.23%. Although in the isomerization reaction, α -pinene produces several bicyclic and monocyclic compounds, such as camphene with the highest concentration of 8.05% and a yield of 6.83%, D-limonene with a concentration of 6.03% and a yield of 5.12 %, terpinolene with a concentration of 5.20% and yield of 4.41%, 3-carene with a concentration of 2.40% and yield of 2.03% and other compounds formed in the isomerization reaction of α -pinene, namely bicyclo[3.1.0]hex-2-ene; 4-methylene-1-(1-methylethyl); β -pinene; 2,6-dimethyl-1,3,5,7 octatetraene; 1,2-cyclohexadiene, 1-methyl-4-(1-methylethyl); o-cymene; trans-p-mentha-2,8-dienol; α -campholenal; endo-borneol; and terpineol.

Keywords: α -pinene, terpineol, rahims 3rd generation catalyst, hydration reaction, isomerization

1. INTRODUCTION

Apart from the α -pinene hydration reaction, there is also an α -pinene isomerization reaction. The isomerization of α -pinene using solid phase catalyst under acidic conditions is currently being developed because it produces isomeric products that have the potential to become important synthetic products in the pharmaceutical and fragrance production fields [1]. Isomerization of α -pinene can produce bicyclic compounds, monocyclic compounds, or other products. The bicyclic products are camphene and tricyclene, while the monocyclic products are limonene, p-cymene, and terpinolene [2]. One example of the benefits of the isomerization product above is camphene which is used as an ingredient to synthesize taxophene compounds as insecticides [3]. Research that has been carried out regarding the isomerization of α -pinene with an acid catalyst has been carried out by several previous researchers, one of which was Comelli et al. [4], who carried out the isomerization of α -pinene with sulfated zirconia to produce the compounds of camphene, tricyclene, limonene, and terpinolene (m-cymene and p-cymene), at a temperature of 120°C and time of 180 minutes. Solid acid catalysts have also developed among synthesis researchers in homogeneous and heterogeneous reactions. One of the new solid acid catalysts is the result of the reaction of eugenol with a mixture of acetic acid and sulfuric acid where the results can be applied as a catalyst support polymer. Eugenol is an attractive natural compound with a low price containing phenolichydroxyl and allyl functional groups. Apart from its cheap price, eugenol dissolved in this organic solvent also has the advantage of being environmentally friendly [5].

2. EXPERIMENTAL SECTION

Procedures

1. Synthesis of Rahims 3rd Generation Catalyst

Add 10 g eugenol into a 250 mL beaker glass, then add 2.5 mL H₂SO₄ - 5 drops CH₃COOH p.a catalyst mixture into a 4:1 ratio (monomer: catalyst). The addition is

carried out in a drop-by-drop manner and stirred using a magnetic stirrer. The resulting polymer is characterized by the appearance of white smoke and the presence of polymers attached to the walls of the beaker. The polymerization is stopped by adding 1 mL of CH₃OH p.a to the mixture so that the polymer thickens and forms solid.

2. Synthesis of Terpineol from α -pinene

A three-neck flask that had contained 10.8 g of aquadest and 34.0575 g of α -pinene was then assembled together with a condenser, magnetic stirrer, and water heater that had been set at 70°C with constant stirring. When the reaction temperature has been reached, 1.3623 g of Rahim 3rd generation catalyst is introduced into the three-neck flask and heated for 4 hours at that temperature. After the heating is complete, the mixture is transferred to the separating funnel and allowed to stand until two phases are formed. Then the organic phases are separated and washed three times using aquadest. Added anhydrous Na₂SO₄ to bind the leaching residual water. It was further weighed and analyzed using FTIR instruments and gas-chromatography mass spectroscopy.

3. α -Pinene Isomerization Reactions

The three-neck flask that had contained 34.0575 g of α -pinene was then assembled together with a condenser, magnetic stirrer, and oil heater that had been set at 120°C with constant stirring. When the reaction temperature has been reached, 1.3623 g of Rahim 3rd generation catalyst is introduced into the three-neck flask and heated for 4 hours at that temperature. After the heating is complete, the mixture is transferred to the separating funnel. Then the organic phase is separated from its catalyst by washing three times using aquadest. Added anhydrous Na₂SO₄ to bind leaching residual water. Further weighed and analysed using FTIR instruments and gas-chromatography mass spectroscopy.

3. RESULTS AND DISCUSSION

1. Synthesis of Rahims 3rd Generation Catalyst

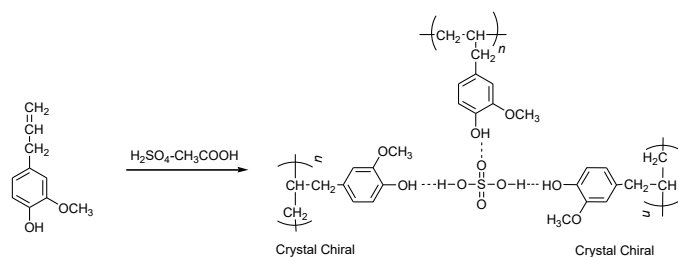


Fig.1. Synthesis of Rahim 3rd Generation Catalyst

2. Synthesis of Terpeneol from α -Pinene

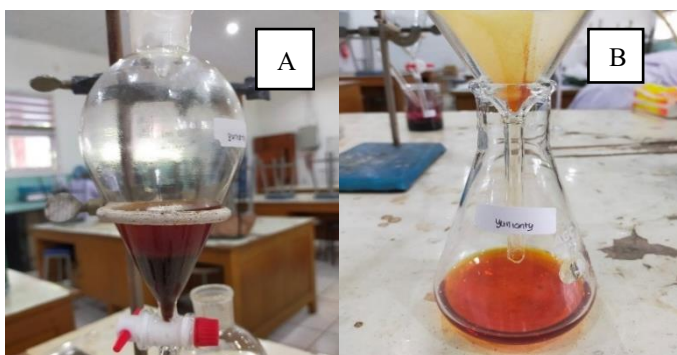


Fig.2. The results of the hydration reaction of α -pinene (a) organic phase before being separated (b) organic phase after separation

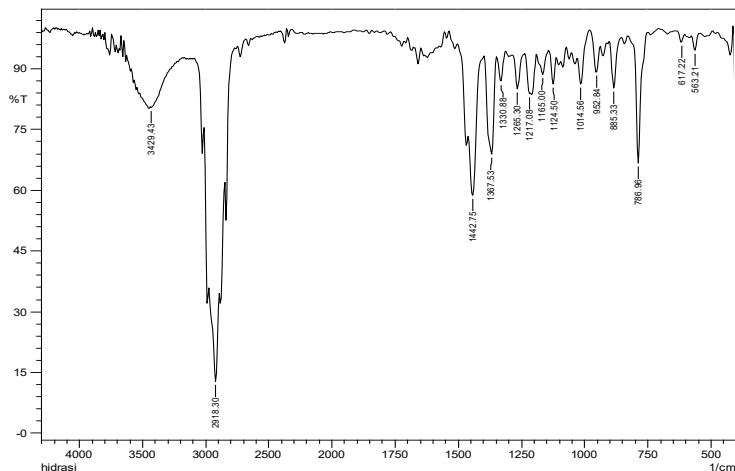


Fig 3. IR spectrum of terpineol compounds

Based on GC MS interpretation it can be estimated that α -pinene compounds that do not react at a retention time of 6.78 min with a content of 29.27% and it can be concluded that terpineol compounds the highest reaction yield products appear at a retention time of 14.21 min with a content of 8.12% and yield yield of 5.23%. In addition, in this reaction there are also other products with lower levels.

4. α -Pinena Isomerization Reaction

The isomerization reaction of α -pinene with the addition of Rahim 3rd catalyst at 120°C for 4 hours produces an organic phase with physical properties including a liquid state, a dense brown color, and a characteristic odor. In this reaction the catalyst is homogeneous, where the catalyst is in the same phase as the reactants. The catalyst is separated by washing using aquades, directly separating the catalyst from its originally mixed organic phase. One of the advantages of catalyst buffer polymers is that they are heterogeneous and homogeneous so that they can be easily separated from the organic phase. According to Misha and Sergey [6], the development of catalyst polymers that are homogeneous and heterogeneous, keeps the catalyst reactive and easily separated without special treatment. An image of the results of the α -pinene isomerization reaction can be seen in figure 4.

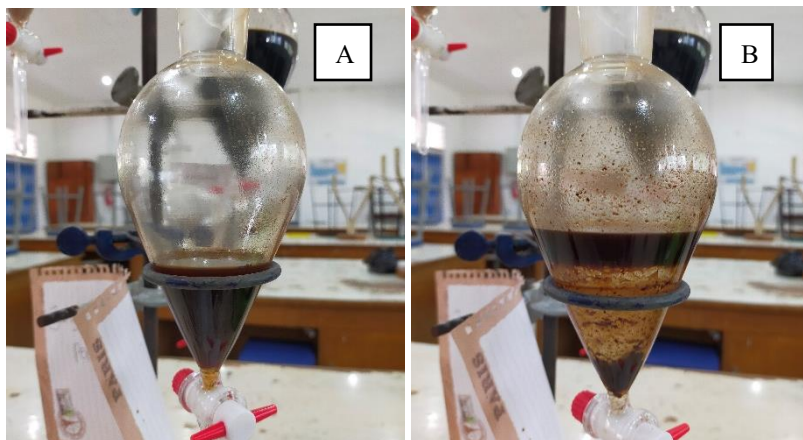


Fig 4 Results of the isomerization reaction of α -pinene (a) reaction mixture before washing with aquades (b) reaction mixture after washing with aquades

Images of α -pinene isomerization products can be seen in figure 5.

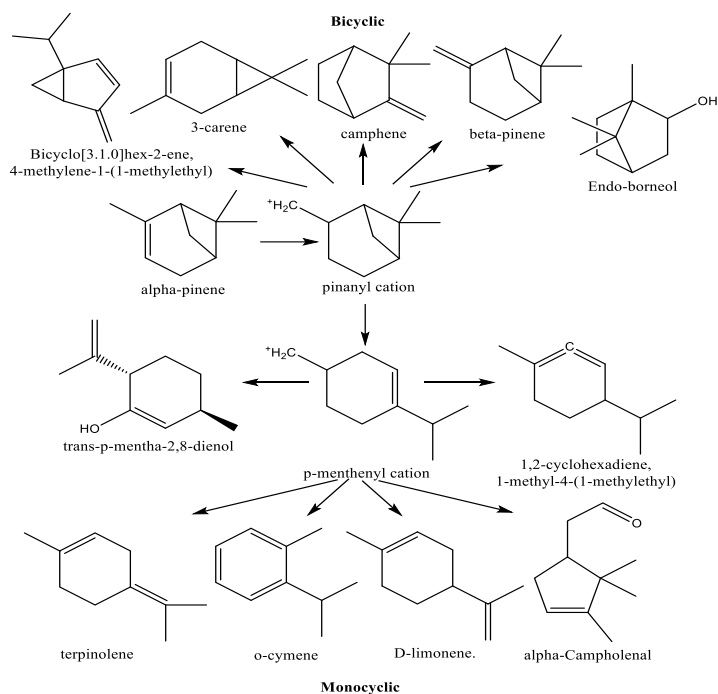


Fig 5. α -Pinene isomerization products

4. ACKNOWLEDGEMENT

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