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SYNTHESIS OF ALPHA TERPINEOL FROM THE ESSENTIAL OIL OF SAPU-SAPU PLANT USING A COMBINATION OF NATURAL ACID CATALYST OF STARFRUIT AND SYNTHETIC ACID

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ABSTRAK

Belimbing wuluh (*Averrhoa bilimbi* L.) adalah tumbuhan yang tidak termasuk kedalam tumbuhan musiman yang banyak tumbuh di Bangka Belitung dan memiliki rasa yang asam. Buah belimbing wuluh memiliki kandungan AHA seperti asam malat, asam askorbat, dan asam sitrat. Potensi rasa asam buah belimbing wuluh dapat digunakan sebagai katalis pada sintesis alfa terpineol dari alfa pinene minyak atsiri tumbuhan sapu-sapu. Alfa terpineol dapat disintesis dengan tanpa kombinasi dan kombinasi katalis. Katalis yang digunakan seperti asam sulfat 15%, asam asetat 99,5%, dan asam buah belimbing wuluh. Tujuan dari penelitian ini adalah dapat mengetahui persen area alfa terpineol dengan variasi komposisi katalis, dan dapat menentukan kadar alfa terpineol dengan variasi komposisi katalis. Persen area alfa terpineol yang dihasilkan tertinggi pada katalis asam sulfat (15%) sebesar 16,76%, katalis kombinasi 1 sebesar 12,13%, katalis kombinasi 2 sebesar 8,38%, katalis asam asetat (99,5%) sebesar 3,22%, dan katalis asam buah belimbing wuluh sebesar 0,42%. Kadar alfa terpineol yang dihasilkan tertinggi pada katalis asam sulfat (15%) sebesar 33,14%, katalis kombinasi 1 sebesar 23,99%, katalis kombinasi 2 sebesar 16,57%, katalis asam asetat (99,5%) sebesar 6,36%, dan katalis asam buah belimbing wuluh sebesar 0,83%.

ABSTRACT

Belimbing wuluh (Averrhoa bilimbi L.) is a plant that is not included in seasonal plants that grow in Bangka Belitung and has a sour taste. Starfruit contains AHA compounds, including malic acid, ascorbic acid, and citric acid. The potential sour taste of starfruit can be used as a catalyst in the synthesis of alpha terpineol from alpha pinene of sapu-sapu essential oil. Alpha terpineol can be synthesized with no catalyst or a combination of catalysts. The catalysts used are sulfuric acid (15%), acetic acid (99.5%), and starfruit acid. The purpose of this study was to determine the percentage area of alpha terpineol with variations in catalyst composition, and to determine the levels of alpha terpineol with variations in catalyst composition. The percentage area of alpha terpineol produced was highest in sulfuric acid catalyst (15%), at 16.76%, combination catalyst one at 12.13%, combination catalyst two at 8.38%, acetic acid catalyst (99.5%) at 3.22%, and starfruit acid catalyst at 0.42%. The highest alpha terpineol content was produced in sulfuric acid catalyst (15%) at 33.14%, combination catalyst one at 23.99%, combination catalyst two at 16.57%, acetic acid catalyst (99.5%) at 6.36%, and starfruit acid catalyst at 0.83%.

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INTRODUCTION

Essential oils are natural plant extracts derived from leaves, flowers, wood, and seeds that are volatile. Essential oils are found in several plants that are export products, including citronella, cloves, patchouli, sandalwood, agarwood, ginger, nutmeg, pepper, cinnamon, and eucalyptus (Eralita et al., 2020). In addition, essential oils can also be produced from the sapu-sapu plant (*Baeckea Frutescens* L). The essential oil in the sapu-sapu plant contains high monoterpene group compounds consisting of alpha pinene 26.95%, beta pinene 21.55% and 1.8 cineol 18.04% (Supandi et al., 2019). Alpha pinene can be further reacted into alpha terpineol.

Alpha terpineol is a volatile alcohol compound from the monoterpene group with low toxicity. Alpha-terpineol is also widely used in shampoos, soaps, household cleaners, and detergents. Alpha terpineol exhibits antimicrobial activity, making it suitable for wound healing and the treatment of insect bites (Pratigto et al., 2015). Alpha terpineol is widely used in the perfume, cosmetics, and traditional medicine industries. Alpha terpineol can also be used as a relaxant due to its fragrant aroma (Daryono, 2015).

Alpha terpineol can be obtained through the hydration reaction of alpha pinene with the addition of acid, which produces a complex mixture of monoterpenes, alcohols, and hydrocarbons (Amilia et al., 2015). The use of acid as a catalyst accelerates the synthesis by reducing the activation energy, allowing the reaction to run faster and requiring less energy. Catalysts that can be used include sulfuric acid, hydrochloric acid, acetic acid, oxalic acid, and chloroacetic acid (Muharani et al., 2014). The acetic acid catalyst plays a role in increasing the amount of activated reactants so that it will accelerate the reaction rate (Iryani & Rustamaji, 2016), while the sulfuric acid catalyst plays a role in absorbing water so that the reaction can run to completion, and conversion will increase (Satriadi, 2012). Alpha-hydroxy acid (AHA) can also be used as a catalyst. The content contained in AHA, such as malic acid, glycolic acid, citric acid, and tartaric acid, which are usually found in fruits (Suherman et al., 2009). The use of sulfuric acid as a catalyst can produce alpha-terpineol at yields ranging from 60.21% (Muharani et al., 2014) to 67.79% (Daryono, 2015). Furthermore, using an AHA catalyst with the addition of acetic acid yields alpha terpineol at 43.5% (Meng et al., 2022). To reduce the use of strong acid catalysts that are highly toxic, corrosive, environmentally hazardous, and expensive, a combination of strong acid catalysts with acid catalysts derived from natural materials is necessary. Natural acid catalysts are found in plants that contain AHA, such as starfruit plants (*Averrhoa bilimbi* L.).

Starfruit (*Averrhoa bilimbi* L.) is a plant that is not included in seasonal plants (Aflinda & Armi, 2015). Starfruit contains several chemical compounds that are acidic, including oxalic acid, citric acid, tartaric acid, and succinic acid (Samsudin, 2018). The AHA content in starfruit includes malic acid 1.83%, ascorbic acid 0.093%, and citric acid 2.29% (Suherman et al., 2009). AHA can be combined with other acids to catalyze the synthesis of alpha-terpineol. Therefore, a study was conducted on the synthesis of alpha-terpineol from essential oils of the sapu-sapu plant using a combination of natural acid catalysts from starfruit and synthetic acids.

METHODS

Materials

The materials used in this study were essential oil of sapu-pupa leaves, acetone p.a., starfruit extract, distilled water, 99.5% acetic acid, 15% sulfuric acid, and pH paper.

Tools

The tools used in this study were aluminum foil, sample bottles, three-necked flasks, reverse condensers, 500 mL beakers, Erlenmeyer flasks, hot plates, magnetic stirrers, 100 mL measuring flasks, thermometers, volume pipettes, analytical balances, stands, stopwatches, spatulas, blenders, stirring rods, glass funnels, filter paper, dropping pipettes, separating funnels, a set of reflux and GC-MS equipment, Shimadzu QP 2010 SE brand in the integrated laboratory of the Islamic University of Indonesia.

Procedure

Preparation of Natural Acid Catalyst from Starfruit (*Averrhoa Bilimbi* L.)

The research sample was starfruit from Tuing Hamlet, Mapur Village, Riau Silip District, Bangka Regency. Then the starfruit was blended and squeezed using a clean cloth, and then filtered to obtain clean starfruit water free from dirt and fruit juices. Starfruit water is ready to be used as a catalyst (Ratih, 2021).

pH measurement

The test is carried out by placing universal pH paper into starfruit water. The reading of the results is based on color similarity by matching the standard color (Ratih, 2021).

Preparation of 15% Sulfuric Acid Solution

The preparation of a 15% sulfuric acid solution was carried out by adding 15.30 mL of concentrated sulfuric acid to a 100 mL measuring flask and then adding distilled water to the mark (Amelina et al., 2015).

Synthesis of Alpha Terpineol with Variation of Catalyst Composition

Synthesis of alpha terpineol using essential oil of the sapu-sapu plant with acetic acid (99.5%), sulfuric acid (15%), and natural acid from starfruit water as catalysts.

Table 1. Catalyst Composition Variation

		Catalyst (mL)			
Essential oil (mL)	Acetone (mL)	Acetic Acid 99,5% (mL)	Starfruit Sour (mL)	Sulfuric Acid 15% (mL)	Aquades (mL)
10	30	5	0	0	2
10	30	0	0	5	2
10	30	0	5	0	2
10	30	2	2	1	2
10	30	1	3	1	2

The mixture was then stirred until homogeneous using a magnetic stirrer and refluxed at a temperature of 60-70°C for 4 hours. After reflux, the solution was cooled and separated into oil and acetone layers (Muharani et al., 2014).

Alpha Terpineol Analysis Using a GC-MS Instrument

Identification of the alpha pinene compound component into alpha terpineol was carried out using a GC-MS (Gas Chromatography and Mass Spectroscopy) Shimadzu QP 2010 SE. A sample of 1 µL of the reaction was carried out for each experimental treatment. Then the solution mixture was injected into the column with an injector temperature of 280°C, a column temperature of 60-310°C, a sampling time of 1 minute, the carrier gas was helium, a pressure of 13.7 Kpa, a column flow of 0.55 mL/m, a total flow of 58.8 mL/m, and an ionizing type of EL (electron impact) of 70 eV (Muharani et al., 2014). This analysis was conducted to determine the levels of alpha-terpineol formed. The calculation of alpha terpineol levels was carried out using the following equation (Daryono, 2015):

$$\text{Alpha terpineol levels (\%)} = \frac{\text{Alpha terpineol reaction product}}{\text{(Initial alpha pinene+Acetone density)}} \quad \text{(Eq. 1)}$$

RESULT AND DISCUSSION

Preparation of Natural Acid Catalyst from Starfruit *(Averrhoa bilimbi* L.*)*

The starfruit used comes from Tuing Hamlet, Mapur Village, Riau Silip District. The fruit is then cleaned and cut into smaller pieces. The pieces are crushed by pounding or blending. The water is then separated from the remaining flesh using a sieve, resulting in pure, yellow starfruit water.

The acidity level of starfruit juice was measured using pH paper. Starfruit juice as a catalyst has a pH of 1 (one). This pH is highly effective as an acid catalyst in the synthesis of alpha-terpineol from alpha-pinene. The higher the acidity level of the catalyst used, the faster the reaction rate in the synthesis of alpha-terpineol from alpha-pinene (Meng et al., 2022). Starfruit juice contains acids such as malic acid, oxalic acid, and citric acid, which are included in Alpha Hydroxy Acids (AHA) (Suherman et al., 2009).

Synthesis of Alpha Terpineol from Alpha Pinene with Variation of Catalyst Composition

In this study, the synthesis of alpha-terpineol from alpha-pinene, present in the essential oil content of the sapu-sapu plant, will be carried out with and without a catalyst combination. Alpha pinene is the main component of turpentine oil, which is usually obtained by distillation (Wijayati et al., 2014). Alpha terpineol is a volatile alcohol compound from the monoterpene group with low toxicity (Pakdel et al., 2001). This treatment was carried out without a catalyst combination to see the ability of each catalyst used in synthesizing alpha terpineol from alpha pinene essential oil. The treatment, carried out with a combination of catalysts, optimizes chemical reactions, increases reaction rates, alters product selectivity, enhances catalysis, and increases alpha terpineol levels (Meng et al., 2022).

The synthesis of alpha terpineol from alpha pinene was carried out using a reflux system using acetone as a solvent and 15% sulfuric acid catalyst, 99.5% acetic acid, natural starfruit acid, combination 1 (2 mL acetic acid: 2 mL starfruit acid: 1 mL sulfuric acid), and combination 2 (1 mL acetic acid: 3 mL starfruit acid: 1 mL sulfuric acid). The use of natural starfruit acid catalysts can help reduce the use of more hazardous organic catalysts (Prakoso et al., 2020). The AHA content of starfruit, acting as a catalyst, can help increase the synthesis of alpha-pinene into alpha-terpineol through a hydration reaction mechanism. AHA serves as a proton donor, facilitating the formation of new bonds in the alpha-terpineol compound. Starfruit acid can also help control the course of the reaction and prevent the formation of unwanted by-products (Meng et al., 2022). Starfruit acid, in addition to acting as a catalyst, also serves as an acidifying agent to enhance the acidity of reactions that require acidic conditions to operate optimally (Has et al., 2020).

Acetone is used as a solvent because it is a semi-polar compound, allowing it to dissolve organic compounds such as polymers, fats, oils, and resins (Soelistyo et al., 2022). Synthesis carried out using reflux will produce a more optimal reaction, as the reflux system ensures that the reaction between the reactant and the catalyst occurs efficiently. Reflux is also employed to control reaction conditions, extend the reaction time, and accelerate the reaction by heating; however, it does not reduce the amount of substance present (Fatimura, 2014). The results of the synthesis of alpha terpineol from alpha pinene with variations in catalyst composition are presented in the following table.

Table 2. Synthesis Results of Alpha Terpineol from Alpha Pinene with Variations in Catalyst Composition

Catalyst	Product color	Product volume
Sulfuric acid 15%	Yellow	9,5 ml
Acetic acid 99.5%	Yellow-brown	5,8 ml
Starfruit acid	Yellow	9,7 ml
Combination 1	Yellow-brown	9,5 ml
Combination 2	Yellow	5,8 ml

Description:
Combination 1: Acetic Acid: Starfruit Acid: Sulfuric Acid (2:2:1)
Combination 2: Acetic Acid: Starfruit Acid: Sulfuric Acid (1:3:1)

Based on Table 2, the synthesis of alpha-terpineol from alpha-pinene essential oil of the sapu-sapu plant was carried out using a reflux system to obtain products with different colors and volumes. This is due to the difference in catalysts used. The sulfuric acid catalyst can trigger synthesis reactions, resulting in a larger volume due to the formation of purer compounds. The acetic acid catalyst produces a slower reaction and can affect the formation of by-products, resulting in significant color changes and a lower volume. Starfruit acid produces the same color and product as sulfuric acid. This is because starfruit acid can provide almost the same effect as sulfuric acid on the hydration of alpha pinene, so that it can produce a hydration reaction that is quite effective in synthesizing alpha pinene into alpha terpineol. In addition, starfruit acid acts as a source of protons for reactions, eliminating the need for strong acids such as sulfuric acid (Prakoso et al., 2020). The addition of starfruit acid to the combination catalyst can produce a smaller volume of the final product. This is because starfruit contains water, which can react with the solvent in the synthesis mixture, resulting in gas release or water evaporation (Faradika et al., 2019). The refluxed synthesis product was then subjected to GC-MS analysis.

GC-MS analysis was conducted on samples of essential oil from the broom plant and the product of alpha terpineol synthesis from alpha pinene to determine the alpha terpineol content. Based on the results of the GC-MS test on the essential oil of the broom plant and the product of alpha terpineol synthesis from alpha pinene, the essential oil is presented in the following table.

Table 3. GC-MS Results of Essential Oil of Broom Plant and Synthesis Products of Alpha Terpineol from Alpha Pinene Essential Oil

Sample	Area (%)	
	Alpha-pinene	Alpha-terpineol
Sapu-sapu essential oil	49,77	0,17
Product 1 (15% sulfuric acid catalyst)	4,26	16,76
Product 2 (99.5% acetic acid catalyst)	55,12	3,22
Product 3 (starfruit acid catalyst)	76,00	0,42
Product 4 (combination catalyst 1)	53,36	12,13
Product 5 (combination catalyst 2)	55,45	8,38

Description:
Combination 1: Acetic Acid: Starfruit Acid: Sulfuric Acid (2:2:1)
Combination 2: Acetic Acid: Starfruit Acid: Sulfuric Acid (1:3:1)

Based on Table 3, the results of GC-MS analysis on the synthesis of alpha terpineol from alpha pinene essential oil of broom plant show that the percentage area of alpha pinene and alpha terpineol increased and decreased. The synthesis of alpha terpineol from alpha pinene with 15% sulfuric acid catalyst showed that the percentage area of alpha pinene decreased, while the percentage area of alpha terpineol increased. In the synthesis of alpha terpineol from alpha pinene with 99.5% acetic acid catalyst, starfruit acid, combination 1, and combination 2, the percentage area of alpha pinene and alpha terpineol increased. In weak acid catalysts, the percentage area was smaller; for example, the starfruit acid catalyst produced a very low percentage area of alpha terpineol. This is because starfruit acid is a weak acid that contains alpha-hydroxy acid (AHA), which can catalyze the synthesis of alpha-terpineol from alpha-pinene through a hydration reaction mechanism. AHA from starfruit functions as a catalyst that can help break down alpha pinene into alpha terpineol in the alpha pinene hydration reaction, but the synthesis ability tends to be slow if used without a combination with a strong acid catalyst (Meng et al., 2022).

Determination of Alpha Terpineol Content from Alpha Pinene Essential Oil

The results of GC-MS analysis on each catalyst were used to calculate the alpha-terpineol content produced from the synthesis process. In the GC-MS analysis of sapu-sapu essential oil, the initial alpha pinene area percentage was 49.77%, while the alpha terpineol produced from the reaction was by the area percentage value of each catalyst from the GC-MS analysis results. In addition, the acetone solvent has a density of 0.79 g/mL. The results of the calculation of alpha terpineol content are presented in the following table.

Table 4. Alpha Terpineol Level Results

Catalyst	Alpha Terpineol Levels (%)
Sulfuric acid 15%	33,14
Acetic acid 99.5%	6,36
Starfruit acid	0,83
Combination 1	23,99
Combination 2	16,57

Description:
Combination 1: Acetic Acid: Starfruit Acid: Sulfuric Acid (2:2:1)
Combination 2: Acetic Acid: Starfruit Acid: Sulfuric Acid (1:3:1)

Based on Table 4, the sulfuric acid catalyst yields a higher alpha terpineol content than the acetic acid catalyst and starfruit acid, which is attributed to the differing acid strengths of each catalyst used. Sulfuric acid is a strong acid that can dissolve in water in all ratios used. The strength of sulfuric acid is evident in its ability to dissociate almost entirely in water, with a pKa value of around -3. This allows it to release hydrogen ions (H+) easily in solution, thereby accelerating the efficiency of chemical synthesis. Acetic acid is a weak acid with a pKa value of 4.76, so that it does not completely dissociate in water and only some of the acetic acid molecules dissociate into acetate ions and hydrogen ions. Starfruit has an AHA content that is not yet pure and does not completely dissociate in water, resulting in a weak acid strength that slows and reduces its synthesis ability (Rahman, 2021). Starfruit acid has acidic properties because the pH of starfruit water is 1-2.2 (Mimi, 2018).

Reaction Mechanism of Alpha Terpineol Formation from Alpha Pinene

The reaction of the formation of alpha terpineol from alpha pinene is by the formation of terpine hydrate, which is continued by the formation of terpineol from terpine hydrate. Terpine hydrate is formed when alpha pinene reacts with acids such as nitric acid, phosphoric acid, and hydrochloric acid (Daryono, 2015). In the synthesis of alpha terpineol from alpha pinene essential oil of the sapu-sapu plant with a catalyst of 15% sulfuric acid, acetic acid, and natural AHA from starfruit water, the reaction mechanism for the formation of alpha terpineol from alpha pinene occurs as follows.

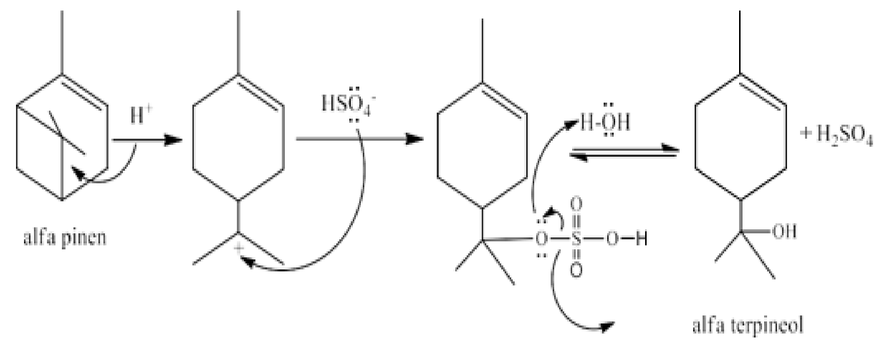

Figure 1. Alpha Terpineol Formation Reaction with 15% Sulfuric Acid Catalyst

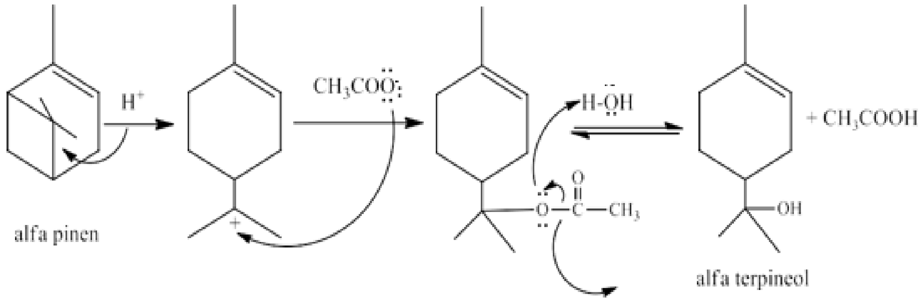

figure 2. Alpha Terpineol Formation Reaction with 99.5% Acetic Acid Catalyst

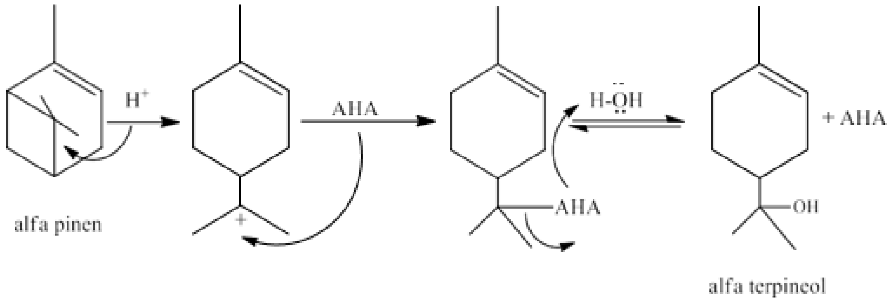

Figure 3. Alpha Terpineol Formation Reaction with AHA Catalyst from Natural Acid of Starfruit

Based on the image above, the reaction of alpha terpineol formation from alpha pinene occurs because the catalysts sulfuric acid, acetic acid, and AHA provide H^+ to attack the double bond in alpha pinene, resulting in the formation of a carboncation that can cause the cyclic or cyclopentane ring to open to achieve higher stability. Then, hydrogen sulfate, acetate ion, and alpha-hydroxy acid will attack the (+) so that a new bond is formed in the structure. Next, a reaction occurs with water, where the carboncation attacks the H^+ in the water, allowing it to form a catalyst again. This is because the catalyst used does not form a product, but rather facilitates the reaction process of alpha-terpineol formation. The reformed catalyst will release from the bond in the structure. In this reaction, water provides OH^- to produce the final product, alpha terpineol.

The catalyst used in the synthesis of alpha-terpineol from alpha-pinene can donate H+ to initiate the reaction mechanism of alpha-terpineol formation from alpha-pinene. Sulfuric acid catalyst, in donating H^+, can be effective in chemical reactions because it is a strong acid. In contrast, acetic acid and AHA catalysts in donating H^+ are less effective because they are weak acids, so they can affect the levels of alpha terpineol produced. The levels of alpha terpineol with strong acid catalysts are greater than the levels of alpha terpineol with weak acid catalysts. This is because strong acids can be used as dehydrating agents to remove water molecules from organic compounds. The ability to donate H^+ to protonate carbon-carbon double bonds in carbon cations is more stable, allowing for increased levels of alpha terpineol (Nurmila et al., 2014).

CONCLUSION

Based on the research data that has been conducted, it can be concluded that the percentage of alpha terpineol area of each catalyst used is the highest in the 5 mL sulfuric acid catalyst of 16.76%, catalyst combination 1 (2 mL acetic acid, 2 mL starfruit acid, 1 mL sulfuric acid) of 12.13%, catalyst combination 2 (1 mL acetic acid, 3 mL starfruit acid, 1 mL sulfuric acid) of 8.38%, 5 mL acetic acid of 3.22%, and 5 mL starfruit acid of 0.42%.

The alpha-terpineol content of each catalyst used was the highest in the 5 mL sulfuric acid catalyst at 33.14%, catalyst combination 1 (2 mL acetic acid, 2 mL starfruit acid, 1 mL sulfuric acid) at 23.99%, catalyst combination 2 (1 mL acetic acid, 3 mL starfruit acid, 1 mL sulfuric acid) at 16.57%, 5 mL acetic acid at 6.36% and 5 ml starfruit acid at 0.83%.

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