PRODUCTION OF BIOETHANOL FROM CAP TIKUS BY REDISTILLATION

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ABSTRACT

Cap Tikus is a traditional drink made from ethanol, which is derived through the distillation of palm sap. In North Sulawesi, the sap of the sugar palm tree (Arenga pinnata) is made into Cap Tikus. Initially, Cap Tikus was consumed primarily by farmers to warm their bodies. However, over time, it has become one of the popular alcoholic beverages in North Sulawesi, particularly among the Minahasa people. It has spread to other regions such as Papua, Kalimantan, and Java. The sap is produced from the sugar palm tree. The people of North Sulawesi, especially the Minahasa tribe, have long recognized this sap as the main ingredient for producing Cap Tikus. The process begins with tapping the enau tree to collect the sap. After collection, the sap undergoes fermentation, resulting in a sour liquid known locally as saguer or, more generally, tuck. After approximately ten hours of fermentation, the saguer is distilled to produce Cap Tikus. During distillation, ethanol is obtained from the Cap Tikus. This ethanol is formed through a hydration reaction involving hydrocarbons, where water molecules are added in the presence of an acid catalyst (H^+). This process leads to a new secondary metabolite compound derived from naphthalene 1,2' - oxirane, accompanied by a reduction in double bonds or additional reactions.

Keywords: *cap tikus, ethanol, distillation, hydration, fermentation.*

INTRODUCTION

Substances that do not change their properties and maintain their original identity are mixtures of two or more substances [1]. Mixtures can be homogeneous or heterogeneous. If the boundary is not visible between the two phases, it is called a homogeneous mixture. Meanwhile, a heterogeneous mixture is one in which the boundary between the two phases is still visible [2].

Separation of mixtures is carried out to obtain good product quality. It is carried out by considering the physical and chemical properties of the mixture. In solving a mixture, it is necessary to consider information about the physical properties of the mixture, namely the freezing point, boiling point, and texture of the mixture. Separation of mixtures physically consists of filtration, sublimation, crystallization, and chromatography. Separation of mixtures chemically considers the chemical reactions that occur. Coagulation is an example of a mixture separation [3].

Users who live outside Java, especially in the Sulawesi area, feel the problem of obtaining ethanol solvents. This is because ethanol solvents must be ordered from Java, which takes a long time and requires much money. To obtain ethanol, we, as ethanol users, students and researchers, conduct research using distilled ethanol from cap tikus. The results from the ethanol extract, separation process, and identification of compounds are very satisfying.

Ethanol separation in palm sap using the distillation method [4]. Ethanol is characterised by being transparent, colourless and flammable. Ethanol burns at a boiling point of 78.5°C. The names for ethanol are alcohol, ethyl alcohol, or grain spirit. Ethanol can also be drunk and is non-toxic [5]. The enau tree (Arenga pinnata) is a palm plant that produces fruit, sap, starch, or flour in the trunk. Enau trees often grow well in tropical areas and are often cultivated by farmers, especially farmers [6]. Palm (Arenga pinnata) is a versatile palm species that can produce various foods and beverages, wood commodities, fibres, biopolymers, and biocomposites [7, 8]. The results of the sugar palm tree, such as sugar palm sap, are fermented into bioethanol to be used as an environmentally friendly renewable energy source [9, 10].

Cap Tikus is a traditional Minahasa liquor which was initially called sopi [11]. Before 1829, the Minahasa people changed the name of sopi to Cap Tikus. They found sopi in a blue bottle with a picture of a rat's tail. Cap Tikus is produced from palm sap with an alcohol content of around 30 - 38 % [12]. This liquor is made from the fermentation and distillation of palm sap [6]. The mixing technique can be used to process the sap.

Sap is a liquid obtained by tapping the male flowers of the aren tree, which contains relatively high sugar. Because it contains sugar, the sap can be processed into alcoholic soft drinks, vinegar, and data. In North Sulawesi, some farmers process sap into a light alcoholic drink called cap tikus. The length of fermentation can increase the alcohol content. The results obtained during the fermentation process of aren sap into cap tikus for 10 h are from 0.33 - 8.33 % [13].

Distillation is a process of separating a mixture using boiling point and relative volatility. The mixture is to be separated at a specific temperature. Substances with high relative volatility will evaporate and then condense to obtain distillate. The process of evaporation and condensation causes the temperature to remain stable and increases the concentration of the distillate. An azeotrope is a mixture of two or more substances with a fixed boiling point and composition. Because ethanol forms an azeotrope, it becomes a challenge when the air limit of ethanol is exceeded. By weight, the ethanol content in water is 95.6 %. The distillation method is one of the methods that shows the azeotrope of air and ethanol. Thus, this study aims to make ethanol from fermented palm sap (*Arenga pinnata*). This study is expected to utilize natural resources in North Sulawesi.

EXPERIMENTAL

The materials used in this study were sago palm obtained from Keroid Village, South Minahasa Regency, North Sulawesi, Indonesia. Meanwhile, the equipment used in the study was a series of distillation tools for making cap tikus traditionally, such as drums for sap, bamboo as a condenser, a heater (stove) using firewood, a macerator, a blender thermometer, and a set of cap tikus distillation tools (round bottom flask, condenser, Erlenmeyer flask heating mantle, water pump, and evaporator).

Taking sap begins by tapping the flower stalk from the tree's base towards the flower cluster. This process is carried out for one month or until the flowers fall. Starting with a period in the first week, which is twice a week. After that, it is continued once a week until the flower clusters fall. This stage is continued to relax the pores or channels of the sap that will come out. After that, tapping is carried out, taking sap from the tree. The time the results of the sap collection is every 24 h in the morning. The sap obtained is then fermented to be processed into an alcoholic beverage, cap tikus. Saguer is stored and left for several hours to undergo a natural fermentation process [14].

The fermented palm sap is further processed traditionally to make cap tikus using simple equipment in the form of bamboo in production house cap tikus. The bamboo used is arranged upward and downward along ± 50 m. Each end of the bamboo is connected to a distillation container and the other to a drum, where the distillation takes place for approximately 1.5 to 2 h at a temperature above the boiling point of alcohol, which is 78 °C, which will then produce a cap tikus liquid. The cap tikus distillation process first involves the fermentation of the sap (saguer) into a drum on the stove and then heating it to a temperature above the boiling point of alcohol, 78°C. The rising steam will enter the bamboo, and the steam will be channelled and flowed through bamboo pipes to the distillation container. These droplets are then known as cap tikus drinks. The time required is approximately 1.5 to 2 h [15].

Next, cap tikus is redistilled in the laboratory at a temperature of 78 ^{o}C. The goal is to increase the ethanol content [16]. Then the ethanol obtained is used to isolate and identify metabolite compounds on leylem leaves using the maceration method. Maceration is usually carried out at 15 - 20 °C for 3 days until the soluble materials dissolve [17]. The filtrate evaporated using an evaporator produced 28.54 g of crude ethanol extract. The filtrate was collected from the first day to the last day, amounting to 8.8 L. Then, it was evaporated using an evaporator, producing a crude ethanol extract content of 3.81 %. Gas Chromatography (GC) testing and analysis of cap tikus was carried out before and after redistillation using a GC 8890 MPS instrument.

RESULTS AND DISCUSSION

Production of bioethanol from Cap Tikus

The sap obtained from the seho (palm) tree undergoes a fermentation process with the reaction mechanism in Eq. (1):

$$\underset{\text{sugar dioxide}}{C_6H_{12}O_6} \rightarrow \underset{\text{alcohol}}{2C_2H_5OH} + \underset{\text{carbon dioxide}}{2CO_2} \quad (1)$$

The process of fermenting sap into alcohol involves several stages of reaction. First, sucrose in the sap undergoes inversion into glucose and fructose through enzymes found in the sap. The next stage is fermentation, where the glucose and fructose formed will then be converted into alcohol by yeast or yeast such as saccharomyces microbes. Finally, the ethanol produced will undergo oxidation into acetic acid by bacteria such as acetobacter acetic [18].

Ethanol can be produced through a hydration reaction, which adds water molecules catalysed by acid (H^+) [19]. In this reaction, alkenes absorb protons from the acid. Through protons bound to alkenes, protons will be bound to less substituted carbon. In the second step, a bond is formed between oxygen from the H_2O molecule and the more substituted alkene carbon. Other H_2O molecules will take excess protons. Protons bound to oxygen are then released through hydroxyl anions that bind them to water molecules (H_2O).

$$CH_2 = CH_2 + H_2O \xrightarrow{H^+} CH_3CH_2OH \quad (2)$$

The reaction in Eq. (2) is exothermic and produces many unwanted by - products, such as diethyl ether, in ethanol production. Hydration reactions are classified as addition reactions. Addition reactions combine two or more molecules into a larger molecule accompanied by a reduction in the double bond of one of the reacting molecules due to the combination. The reaction of ethanol formation occurs through the addition of alkene hydrocarbons because alkenes are aliphatic hydrocarbons that have double bonds (C_nH_{2n}) so that the double bonds can experience saturation [20].

Several genera and species can convert sugar into ethanol, carbon dioxide, and other small but important metabolites, such as glycerol, acetate, succinate, pyruvate, higher alcohols, and esters. This biochemical process, alcoholic fermentation, is responsible for some of the most important foods and beverages ever known to humanity, such as wine, beer, and bread. Traditionally, these fermentation products are obtained when yeast is included in the natural microflora of carbon - rich substrates (cereals for bread/beer and grape must for wine) and spontaneously ferments sugar [21].

The yeast used in fermented drinks is S. cerevisiae, which is elliptical with a diameter of 5 - 10 µm [22]. The reaction of alcohol fermentation into acid is shown in Eq (3).

$$C_2H_5OH + O_2 \rightarrow CH_3COOH + H_2O \quad (3)$$

The distillation process of ethanol from cap tikus depends on the needs of the maceration process. The amount of ethanol obtained from 40 L of cap tikus is 22 L, with a distillation time of 3 days. The purpose of re-distillation is to obtain purer ethanol. Re-distillation is carried out to increase the purity of ethanol by removing components that have lower or higher boiling points than ethanol. In this study, distillation was used to separate ethanol from its azeotropic mixture with water, so that the ethanol content increased. Alcohol is a hydrocarbon compound in the form of a hydroxyl group (- OH) with two carbon atoms (C). The most widely used alcohol species are CH_3OH, which is called methyl alcohol (methanol), C_2H_5OH, which is called ethyl alcohol (ethanol), and C_3H_7OH, which is called isopropyl alcohol (IPA) or propan - 2 - ol.

There are two types of ethanol: synthetic ethanol, often called methanol methyl alcohol or wood alcohol, and made from ethylene, one of the derivatives of petroleum or bura stone. This material is obtained from chemical synthesis called hydration, while bioethanol is engineered from biomass (plants) through biological processes (enzymatic and fermentation). Given the diverse use of bioethanol/ethanol, the grade of ethanol used must be different according to its use. Ethanol with a grade of 90 - 96.5 % can be used in industry, while ethanol with 96 - 99.5 % can be used as a mixture of liquor and essential materials for the pharmaceutical industry. The ethanol grade used as a vehicle fuel mixture is 99.5 - 100 %. The difference in grade will affect converting carbohydrates into water-soluble sugar (glucose) [23].

Because it is an alcohol compound, ethanol has several properties: a colourless solution (transparent), the liquid phase at room temperature, volatile, and flammable. Cap tikus is one example of a traditional drink containing ethanol [24]. Ethanol has the general properties of eels, does not evaporate, does not dissolve easily in water, molecular weight 46.07, boiling point 78.52^{o}C, freezing point - 114.1^{o}C, density 0.79360 (15^{o}C) - 0.78937 (20^{o}C) - 0.78504 (25^{o}C), and refractive index 1.36143 (20^{o}C) - 1.35941 (25^{o}C) [25, 26].

Maceration results using bioethanol filtrate

Maceration is one of the conventional extraction methods that are very useful and simple and does not require significant costs in its implementation because the facilities and infrastructure needed to carry out extraction using the maceration method are straightforward. In general, the maceration procedure consists of several stages in extraction. The plant material to be macerated is ground to increase the surface area of the plant material so that the plant material can be mixed well with the solvent. This process is carried out in a closed container, and the appropriate solvent is added [27].

In this maceration method extraction technique, coarse plant material ground into powder form is soaked in a solvent (water, oil, or alcohol) for an extended period. Long soaking times damage cell walls and push bioactive compounds into the solvent. Then, the solvent is filtered through a filter media that suppresses plant residues and recovers bioactive compounds from plants. The efficiency of extraction of bioactive compounds from plants depends on the type of solvent, solvent polarity, plant material, agitation interval, and extraction time [28, 29].

The main advantages of the maceration process include its simplicity, no need for heating, suitability for heat - sensitive materials, and low installation and maintenance costs. However, the disadvantages of maceration are long extraction times, poor yields, potential growth of spoilage organisms, and the possibility of fermentation. The solvent used for plant extraction is also called menstruation. The choice of solvent depends on the type of plant, the part of the plant to be used, the nature of the bioactive compound, and the availability of the solvent. Polar solvents such as water, methanol, and ethanol extract polar compounds. In contrast, for nonpolar compounds, nonpolar solvents such as hexane and dichloromethane are used [30].

Factors that need to be considered in selecting a solvent for the extraction process are that it must be able to extract the active substance, be non-toxic and non-flammable, be inexpensive, must not react with the extract, have a reasonable recovery rate and can be separated from the extract, have low viscosity, and the boiling point must be low [31]. Considering all the factors that influence the selection of solvents for maceration, ethanol is one of the solvents that is often used. Because of its ability to extract polar secondary metabolite compounds and its low cost and easy availability of ethanol, ethanol is the optimal solvent for extracting natural materials using the maceration method.

Ethanol and air form an azeotrope $EtOH.H_2O$ with an ethanol content of about 95.6 % by weight. Ethanol and air are difficult to separate by simple distillation. Therefore, more advanced techniques such as extractive distillation, pressure swing distillation, or the addition of special entrainers are required [27]. The application and effectiveness of heterogeneous - azeotropic extractive distillation were investigated on non-ideal mixtures by simulation and experiments verifying the accuracy of the modelling [32]. This method shows that EHAD is a powerful tool for separating highly non - ideal mixtures containing azeotropes and water with maximum boiling points [29]. The combination of various types of entrainer chemical structures has a significant effect on improving the purity of ethanol. The combination of an n - heptane stretching chain and cyclic cyclohexane structure has better performance in improving ethanol's purity than the cyclic - cyclic trainer structure. This is due to the different characteristics of entrainers, which result in higher boiling points, which are different from azeotropic mixtures [30].

Gas chromatography (GC) analysis

Gas chromatography (GC) analysis of cap tikus before and after purification (redistillation) produced chromatograms, as shown in Fig. 1 and Fig. 2. In contrast, tabulation of retention time data and % area is shown in Tables 1 and 2. The results show that cap tikus has the same number of chemical components before and after redistillation. Five chromatogram peaks indicate at least five compound components found in both types of cap tikus with almost the same retention time and % peak area. The most dominant peak in the cap tikus chromatogram before redistillation is the compound at peak 2 with a retention time of 7.197 min and % an area of 94.03 % and peak 1 with a retention time of 6.716 m and an area of 5.77 %.

Likewise, the compound in cap tikus after redistillation has the most dominant peak at peak 2 with a retention time of 7.170 min and an area of 94.75 % and peak 1 with a retention time of 6.719 and 5.05 %. Meanwhile, peaks 3, 4, and 5 have an excellent retention time. However, the smaller area indicating the compounds in the three peaks is much less than in peaks 1 and 2. The comparison was made by taking the characteristics of the chromatogram data of pure methanol and pure ethanol (Fig. 1 and Fig. 2). The aim was to determine the type of compound in each dominant chromatogram peak (peaks 1 and 2) of cap tikus before and after redistillation.

The results of GC analysis showed that the prominent peaks in the ethanol chromatogram before and after redistillation indicated the dominance of ethanol compounds. It also appeared that ethanol after redistillation showed an increasing dominant ethanol peak, which means that the purity of ethanol increased. Then, based on the chromatograms of pure methanol and ethanol, the retention time at the dominant peak of cap tikus before and after redistillation (peaks 1 and 2) is similar or the same. It is suspected that the compounds at the first and second peaks are methanol and ethanol, although other compounds are still impurities at peaks 3, 4, and 5. An example of a compound obtained from natural ethanol is a secondary metabolite compound derived from naphthalene 1,2' - oxirane [33].

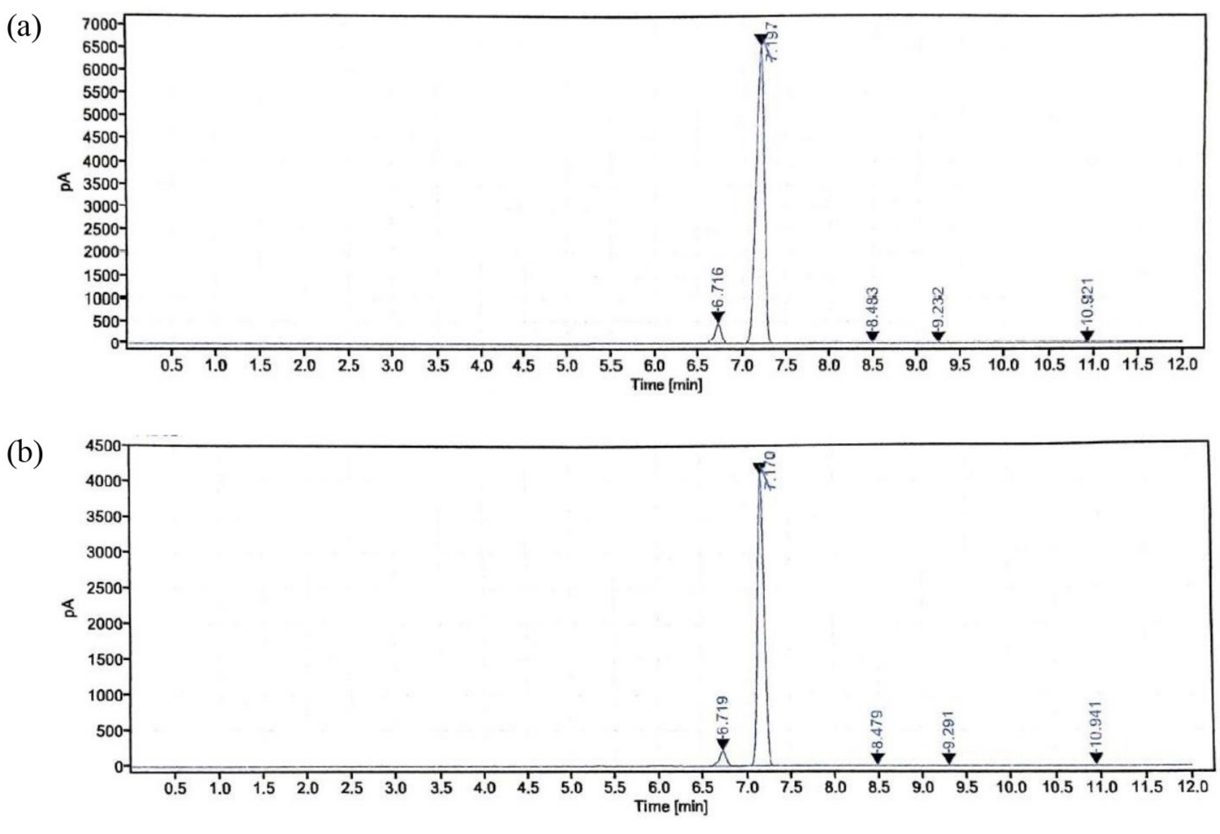

Fig. 1. (a) GC chromatogram of Cap Tikus before redistillation and (b) GC chromatogram of Cap Tikus after redistillation.

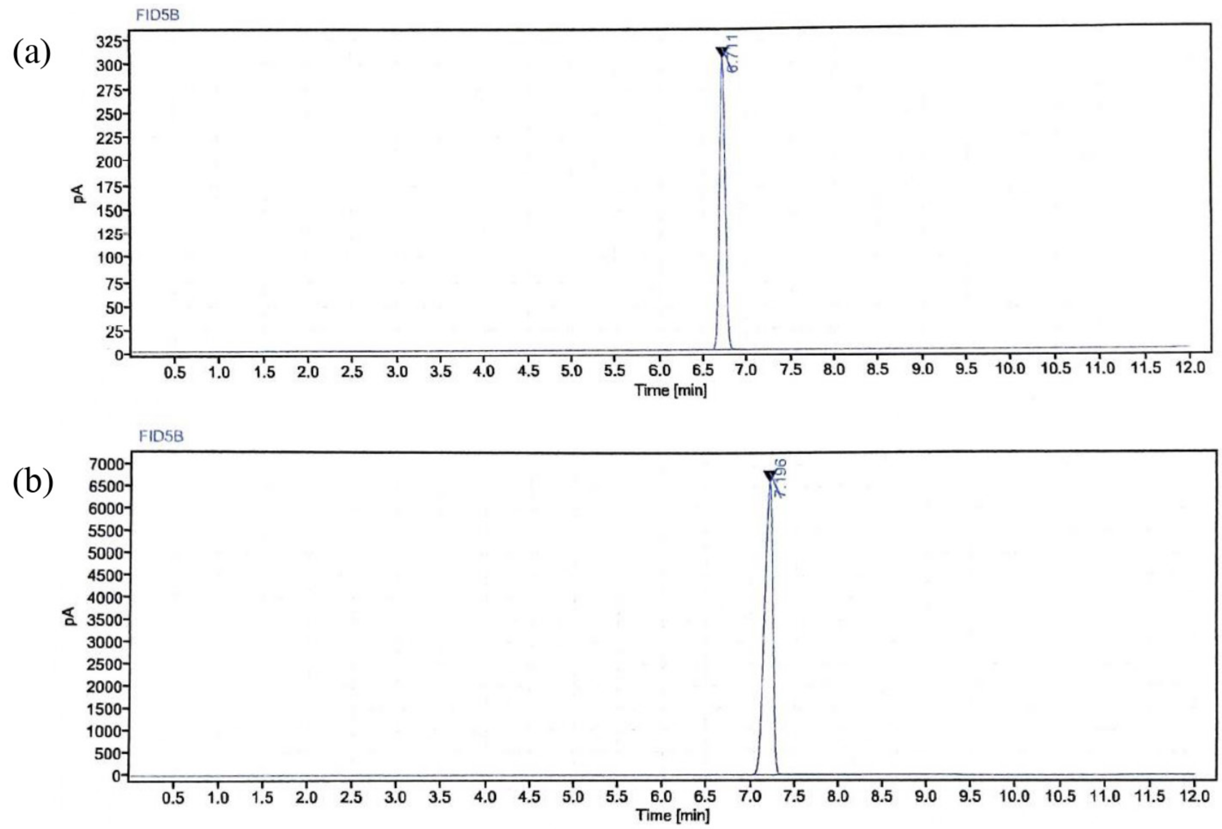

Fig. 2. (a) GC chromatogram of pure methanol and (b) GC chromatogram of pure ethanol.

Table 1. Tabulation of GC chromatogram data of Cap Tikus before redistillation.

No. peak	Retention time, min	Area, %
1	6.716	5.77
2	7.197	94.03
3	8.483	0.08
4	9.232	0.03
5	10.921	0.09

Table 2. Tabulation of GC chromatogram data of Cap Tikus after redistillation.

No. peak	Retention time, min	Area, %
1	6.719	5.05
2	7.170	94.75
3	8.479	0.08
4	9.291	0.03
5	10.941	0.09

CONCLUSIONS

The manufacture of cap tikus through the distillation process of a chemical separation method based on differences in evaporation rates (volatility) has been successful. In the manufacture of cap tikus, an additional reaction of alcohol production occurs through hydration recreation. The maceration process using natural ethanol solvents produces levels that are by the content of secondary metabolites. In the next stage, namely isolation and identification of compound groups, relatively pure compounds are obtained, which can be used to identify suspected compounds. The compounds obtained are secondary metabolite compounds derived from naphthalene 1,2' - oxirane, which uses ethanol solvents from cap tikus in the maceration process to obtain thick extracts.

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