A Simple Purification Method of Catechin from Gambier

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Abstract— Isolation of catechin gambier has been done by maceration using methanol, then treated with water. For purification using ethyl acetate-Hexana with only one repetition time, this is a much simpler way than previous studies. The result catechins weighing 9.594 g from 21,997 g of dirty catechins, with TL = 175.5 to 177.2 °C, the maximum wavelength of 280.10 nm by UV spectroscopy and the presence of OH groups, stretching C-O, C = C aromatic and alkane CH stretching, followed by CH2 bend by IR spectroscopy.

Keywords— gambier, isolation, catechin

1. INTRODUCTION

Gambier (Uncaria gambier Roxb) is one among the Rubicaceae family which has a high economic value. Extract (sap) of leaves and twigs contains Tannat katechu acid (tannin), katechin, pyrocatecol, florisin, wax, fixed oil. The main components of gambier are Tannat katechu acid (20-50%), katechin (7-33%), and pyrocathechol (20-30%).

Catechin included in the flavonoid, not colored, and in pristine condition slightly soluble in cold water but is readily soluble in hot water, soluble in alcohol and ethyl acetate. When catechin heated at a temperature of 110 °C or heated in an alkaline solution of carbonate, it will lose a molecule of water and turn into tannat catechu acid or tannin [1].

Gambier use as a complement to traditional chewing betel nut and medicine, such as in Malaysia gambier used for burn treatment, diarrhea, dysentery and sore throat medicine. In modern way gambier widely used as raw materials of pharmaceutical and food industries. [2], [3].

Indonesia as the world's gambier main supplier, reaching 80%, mostly from the province of West Sumatra with the destination countries of Bangladesh, India, Pakistan, Taiwan, Japan, South Korea, France and Switzerland. [4].

Purification of catechin gambier with good quality will certainly increase the added value of the product itself. It is therefore necessary to find the right method for the purification of these catechins. Based on the literature search, previous researchers have done the isolation of catechin gambier using the solvent ethyl acetate, hexane and water, but the process is done up to 3 times repetition, besides the addition of ethyl acetate as partially dissolved when the tannins tannins come it should not be soluble in ethyl acetate. This is due to the presence of water in which we will gambier purified [5], [6]. Catechins Isolation by prepurification Method using water and the addition of ethyl acetate were repeated 3 times [7]. For that we need to find better methods of drying so that catechins are not damaged and simpler method to get the catechins.

![Fig.1 Purification catechin Scheme](image)
II. METHODOLOGY

A. Chemicals, equipment and instrumentation

The samples used were obtained from the Gambier Halaban, Payakumbuh. Gambier is used is processed from the leaves of gambier, as much as 0.50 kg are used for the isolation of catechins as shown in fig. 2 [2]. 0.50 kg of samples that have been refined gambier, macerated with methanol for 3 days as shown in fig. 2. After it was filtered and the solvent was evaporated using a rotary evaporator to obtain crude methanol extract as shown in fig. 3.

![Fig 2. Gambier of Payakumbuh](image)

![Fig 3. Methanol extract](image)

The crude methanol extract of the wind dried it to dry for a few days. After 1L of distilled water is added to dry heated at 70°C with stirring for about 20 minutes and filtered with cotton to obtain water fraction. Fraction of water is cooled in the freezer, then filtered with a cloth as shown in fig. 4.

![Fig 4. Fraction of water](image)

Fraction of water is dried and found the dirty catechins = 240 g as shown in fig. 5.

![Fig 5. Water fraction after drying.](image)

Chemical used as a solvent in the extraction and purification process is distilled water, hexane, ethyl acetate, methanol.

The tools used in this study is erlenmeyer, glass cup, mumps pipette, funnel, thermometer, oven, analytical balance, rotary evaporator (Heidolph Laborota 4000), UV spectrophotometer (Shimadzu UV-1700 Pharma Spec), IR FTIR spectrophotometer (Thermo Scientific Nicolet is10).

B. Research procedures

Dirty catechins 75 grams wind dried for 2 months, then 21,997 g dirty catechin dissolved with ethyl acetate with heated and stirring on a water bath at a temperature of 70°C, after which it was filtered to obtain the filtrate. The filtrate was added hexane little by little until the brown precipitate and then separated it. The filtrate was added hexane again so catechin precipitate, then separated from the solution as shown in fig.1. Catechin dried and obtained a white catechin = 9.594 g as shown in fig. 6.

![Fig 6. Catechin](image)
Characterisation of catechins done by melting point determination, UV and IR spectrophotometer measurements. Determination of catechins with a UV spectrophotometer using Ciba-Geigy method (SP-SMP-377-1985): Samples of catechins with a watch glass heated for 3 hours at a temperature of 105 °C, then 50 mg diluted with ethyl acetate in 50 mL volumetric flask. This solution was placed into the ultrasonic bath for 5 minutes and then filtered. 15 mL of the filtrate was discarded the first screening results and continue filtering. 2 mL of the filtrate was pipetted into a 100 mL erlenmeyer and add 50 ml of ethylacetate. This solution was placed into the ultrasonic bath for 5 minutes and then measured with a spectrophotometer UV absorbance at a wavelength of 270-300 nm [2].

III. RESULT AND DISCUSSION

A. Purification of Compounds

From the results of the purification 21,997 g of dirty catechin with the addition 600 ml of ethyl acetate, dyestuffs a little tannins still carried away. This shows that the little water is still contained in a dirty catechins, which with the addition of hexane a little tannin will separate. The filtrate was added hexane again so catechins precipitated and after being separated and dried white colored catechins obtained as 9.594 g.

B. Characterization

From the test results obtained melting point of the crystalline is 175.5 °C -177.2 °C. Based on the value of the melting point range <2, it can be indicated isolated compounds have been pure. UV spectrum produced by the compound using ethyl acetate solvent gave maximum absorption at a wavelength of 280.10 nm which can be seen in figure 7.

Maximum absorption peak at 280.1 nm in the UV region that is a characteristic of compounds of catechins, with absorbance = 0.683 nm.

Figure 8 shows the IR spectrum of OH absorption bands in the vibrational strain 3395.35 cm⁻¹, is supported by the presence of CO stretching vibration in the region 1000-1300 cm⁻¹. At 1621.93 cm⁻¹ indicating the presence of C = C aromatic. In the region 2850-3000 cm⁻¹ is the alkane CH stretching, followed by CH₂ bond bending at 1468.10 cm⁻¹.

IV. CONCLUSIONS

Method for purification of catechins is much simpler than previous studies, with only one repetition. Catechins are found as many as 9.594 g from 21,997 g dirty catechins.

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