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DETERMINATION OF EXTRACTION PROCESS CONDITIONS OF GAMBIER CATECHIN (UNCARIA GAMBIER ROXB) FROM SOLOK BIO BIO LIMA PULUH KOTA DISTRICT – WEST SUMATERA

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Abstract. Catechin content is the determinant key of quality in gambier trade. The required Catechin content of gambier extracts as a herbal medicinal ingredient is greater than 90%. Mostly, Local gambier that produced by community is not uniform and low quality, thus lowering the price in the export markets. The quality improvement of gambier can be done by extraction and purification processes. This study aims to determine the best extraction process of catechin from Gambier (*Uncaria Roxb*) which derived from Solok Bio Bio Lima Puluh Kota, West Sumatra. The research methodology includes pre purification: raw materials preparation, washing, filtration, extraction, drying and testing. Washing was done on 100 gr gambier with a variation of water at 500, 600, 700, and 800 ml, heating for an hour at a temperature of 70^oC, screened, filtered, and allow to stand until a precipitate is formed, wash repeatedly, filtered, and dried. Further, extract with a solvent variation of : water, etyl acetate, heated at 70^oC temperature for 1 hour, then filtered. Filtrate then thickened by using a Rotary evaporator, dried at 50^oC temperature for 48 hours and analyzed. The results showed that the best conditions of the extraction process is by using a solvent etyl acetate, at a temperature of 70^oC, grading 97.40% catechins.

Keywords: Catechin; Gambier; Extraction

1. Introduction

Gambier contains important chemical substances, including tannins catechu acid (20-50%), catechin (7-33%), and Pyrocatechol (20-30%), while other chemical substances in a limited percentage [1]. Bachtiar [2] states that the chemical content of gambier most widely used are catechins and tannins. Gambier is not only used to eat the betel, but also used as a raw material for various industries, such as pharmaceuticals, cosmetics, batik, paint, tanners, bio-pesticides, growth hormones, pigments, and as a mixture of complementary foods [3]. Gambier can also function as antioxidants to inhibit the occurrence of Atherosclerosis [4]. Besides, etyl acetate from gambier extract acts as types of inhibitors and serve as an effective mild steel corrosion inhibitor in aqueous solutions [5].

Catechin content is the determinant key of quality in the trade of gambier. Price of gambier with catechin content of more than 99% (for analysis) is Rp. 888,000/ 10 mg, while catechin content of more than 90% is Rp. 984 000 per gram [6]. The catechins content required in pharmaceuticals industries is greater than 90% [7]. High-quality products will also provide a high price, so as to compete well in the global market, and vice versa.

But the instability price of gambier at this time clearly influences the community income and entrepreneurs economic conditions, especially gambier farmers and distribution chains that play an important role in the local gambier trades. Some factors influencing the problems in the business commodity of gambier include 1) Less qualified of product, and many gambier



lost during processing, so requiring repair quality, 2) the bargaining position of farmers are low, and there is no guarantee that the stable prices can benefit farmers, 3) the farmer habit to mix gambier with other ingredients so lowering the selling price [8]. There is no knowledge and applied technology that can improve the productivity and quality of gambier in West Sumatra, so they can not access the market, both for domestic and export needs. However, the exporter usually does not directly export the local gambier to the destination country, but gambier extract should be processed first to be qualified gambier, then separated according to quality, after that the exporters export the qualified gambier extracts to the destination country [9]. Therefore, need to find the right extraction process in processing gambier to improve gambier quality, and high-quality of gambier can produce with a high catechin content. Thus, gambier prices could rise as expected, so as to improve the community economy

Gambier agribusiness has been going on for decades, but there has been no catechin production industry in West Sumatra until now. Actually gambier with catechin levels above 90%, with prepurification method using water and extraction with ethyl acetate has been obtained on a small scale (laboratory) [10, 11]. Research on gambier extraction with water, followed by semi-polar solvents (ethyl acetate) resulted the highest catechin levels ($97.82 \pm 2.01\%$) [12]. While the research results of Youfa and Rahman [13] also demonstrated catechin levels above 90%, namely 96.73% and 99.33% in the absorption of charcoal and coconut shell after dissolved using acetic acid solvents, ethers and Pb acetate. Next, Ferdinal [14] got a simple catechin purification method of gambier, with 9.594 g catechin weighing result from 21.997g dirty catechin. The equipment used in laboratories include goblets, erlemeyer, funnel and big oven at material processing capacity approximately at 0.5kg. Process conditions have been carried out at a temperature of 70°C [15], handling materials in large quantities (industrial scale) would pose complex problems than the small amount material. Catechin purification method of gambier, usually start by utilizing the solubility difference properties. This process known as extraction that is taking solids from materials using liquid.

2. Methodology/Experimental

The samples used in this study are the local Gambier (*Uncaria Gambier* (Hunter) Roxb) from Nagari Solok Bio-Bio District Lima Puluh Kota, solvent of water and ethyl acetate. Catechin was analyzed using a UV-Visible spectrophotometer to determine the catechin level.

2.1. Tools and materials used

2.1.1. Tool

Erlenmeyer, beaker, measuring cup, rod stirrer, vacuum pump, thermometer, hot plate, glass funnel and plastic funnel, filter cloth, Oven, mortar, analytical scale, centrifuges, Blender, Grail vaporizer, Pipette drops, Pipette peck of 10 ml, Rotary Vacuum Evaporator, distillation apparatus, cooling Leibig. UV-Visible spectrophotometer, porcelain cup, furnace, Aluminum foil.

2.1.2. Material

Material used are Gambier, Aquadest, water (H_2O), ethyl acetate, pure catechin, Ice cubes.

2.2. Parameters

2.2.1. Variables Parameter

1. Amount of water solvent prepurification :500, 600, 700, and 800 ml
2. Types of purification solvents (water, Etyl acetate)

2.2.2. Fixed Parameter

Total Gambier, prepurification temperature, Amount of leaching: 3 times and purification of 70 ° C.

2.2.3. Output Parameter

1. Acquisition Percentage, 2. Catechins content, 3. Catechin Weight

2.3. Working procedures.

Gambier powder weighed as much as 100 grams, then put into erlemeyer 1 L. Add water with a volume variation of 500, 600, 700, and 800 ml. Heat for 1 hour at temperature of 70⁰C, then strain. Let stand filtrate until a precipitate is formed. Next wash sediment according predetermined 3times, using the washing solvent volume as the the initial amount. The precipitate was dried in the oven, then smoothed. Added a solvent (water/etyl acetate) with a solvent in accordance at the best ratio in the prepurification extraction. Then heated using reflux for 1 hour, at purification temperature of 70⁰C, filtered in hot conditions. The filtrate was thickened using a rotary evaporator, then dried and analyzed. Repeat 3 times.

3. Results and Discussion

Catechin making process begins with a test sample of solok bio bio.Gambier measurable catechin level of 47.72%. A sample of 100 grams is done prepurification using water and heated at 70oC while stirring for 1 hour, then filtered and washed three times with water reatment at 16° C operating conditions. Once it is done filtration followed by purification using water or ethyl acetate solvent.

3.1. Relations with the amount of solvent prepurification weight gain after washing

Total acquisition catechin on prepurification process can be seen in Figure 1.

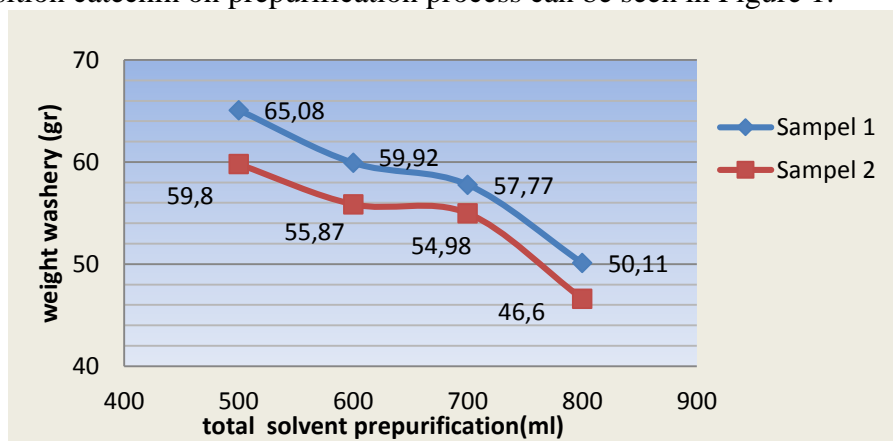


Figure 1. Relationship catechin number solvent for the acquisition of laundering

Figure 1 shows that the larger the volume of the solvent, the smaller the amount of acquisition catechin, can be seen in samples 1 and 2, from 65.08 into 50.11 and from 59.8 to 46.6, is due to the greater number of tannin who participated dissolved together with the

solvent, but the solvent amount of 700-800 ml, a decrease in the acquisition of catechin very much when compared with a decrease of 500-600 ml and 600-700 ml, is caused by the amount of solvent is too large, estimated more catechin participating qualify along with tannin and solvent on filtration. From this research in getting the condition an effective amount of solvent volume is the volume of 700 ml with a tally of the sample 1 was 57.7 grams and for sample 2 of 54.98 grams.

3.2. Relationship between the amount of solvent pre-purification and the catechin levels

Catechin levels on the prepurification process can be seen in Figure 2.

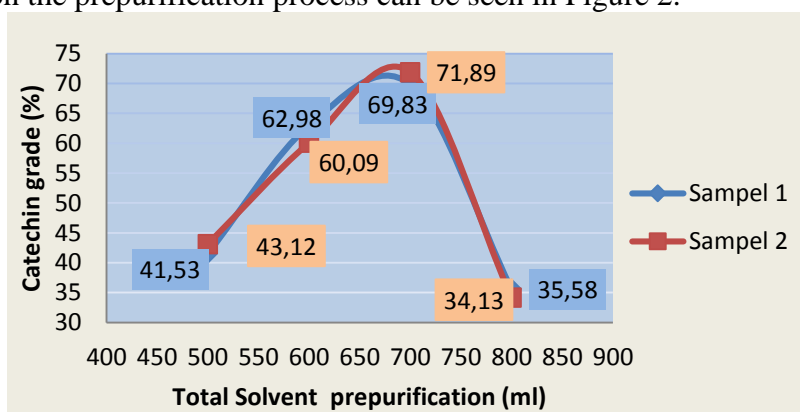


Figure 2. Relationship between the amount of solvent prepurification and the catechin levels

From Figure 2, it can be seen that catechin levels increased with the large amount of solvent in the process of the amount of solvent prepurification i.e. 500 ml to 700 ml for both samples, namely from 41.53% - 69.83% for sample 1, and 43.12% - 71.89% for sample 2, while the amount of solvent 800 ml decreased levels of catechin to 35, 58% for sample 1, and 34.13% for sample 2. Thus the study shows the number of 700 ml of solvent is the most good for prepurification process on levels of catechins, because the solvent volume of 800 ml, the amount of catechins that come together to qualify solvent, thus decreasing the percentage of catechin to material.

3.3. Relationship between the amount of solvent purification and the weight gain of

The amount of solvent is obtained from the amount of the acquisition of the volume of solvent prepurification to his best with a ratio 1; 7. Since the wash product from 65.08gr to 57.77gr for ethyl acetate solvent and from 59.8 gr to 54.98 gr for water solvent. So the amount of catechin recovery in the purification process can be seen in Figure 3.

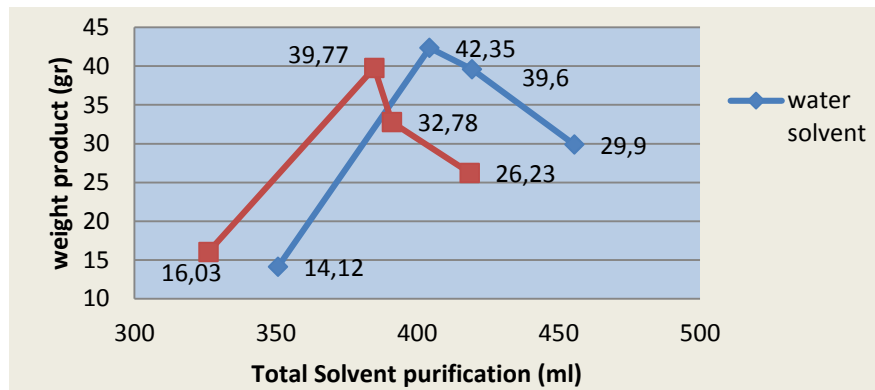


Figure 3. Relationship Number of solvents to the acquisition catechin

Figure 3 shows that the amount of catechins with solvent water acquisition larger than the solvent ethyl acetate is highest achievement in 42.35 grams of water, and the ethyl acetate tertinni acquisition is 39, 77 grams. When viewed from the polar solvent, water is a polar solvent while ethyl acetate is semi-polar solvent, then allowing the solvent water produces more catechin acquisition compared with ethyl acetate, because polar solvents can dissolve polar compounds, semi-polar and even non-polar. Besides, the compound catechin its polar compounds vary greatly, depending on origin gambier. While the decline is due to the acquisition of catechinsolvent more will lead to the possibility of many catechins are shipped to a solvent.

3.4. Relationships amount of solvent purification on levels catechin

Catechin levels in the purification process can be seen in Figure 4.

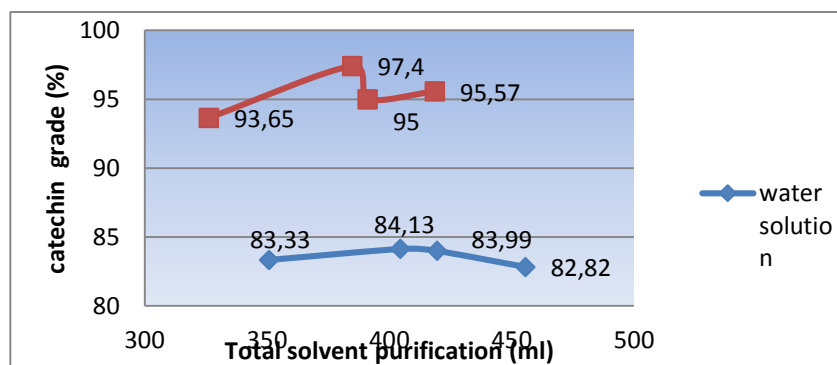


Figure 4. Relationship Number of Solvents on levels of solvent catechin with water and ethyl acetate

Figure 4 shows that the greater the amount of solvent volume, the higher the catechin content obtained in the 385 ml ethyl acetate solvent is 97.4% and the 405 ml of water solvent is 84.13%, but the higher solvent appears to decrease the catechin level. This is due to the higher volumes of solvents being inefficient, as many catechins escape with the solvent. When compared between water solvent and ethyl acetate, it appears that catechin levels are higher in ethyl acetate, in accordance with Sousa et al. [16], because the ethyl acetate solvent is capable

of dissolving the compound to be extracted. Easily separated and purified again, Ethyl acetate is volatile compared to water solvents, resulting in a purer catechin.

3.5. Efficiency of pre-purification and purification process

Prepurification process is the initial process is done in Gambier to obtain initial catechin, performed by dissolution of the Gambier using solvent water and stirred at 70° C conditions of temperature conditions. Then filtered and washed 3 times in cold conditions 16°C. From this process efficiency prepurification process work can be seen in Figure 5.

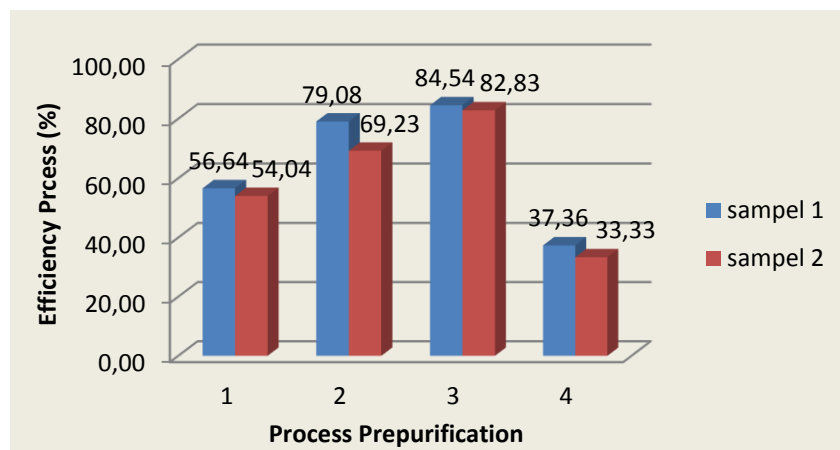


Figure 5. Efficiency process of prepurification

From Figure 5 it can be seen that the treatment to 3 provides the highest efficiency is 84.52% and 82.83%. This means that the extraction process beginning with material Gambier 100 g and 700 ml of solvent with 70°C and washing and filtering of 3 times can be used as an initial determination prepurification process operating conditions. Purification process is a process of continuation of materials or products from prepurification diluted with water and ethyl acetate solvent. The purpose of the purification process is to obtain process efficiency as shown in Figure 6.

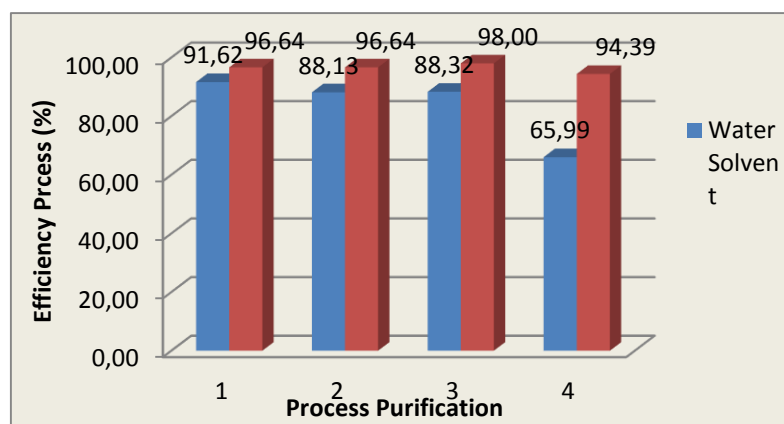


Figure 6. Efficiency process of purification

From Figure 6 it can be seen that the efficiency of work on the mean rat purification process are all high. But the highest point on the use of solvent acetic Ethy almost gives efficiency above 90% and the highest in treatment to 3 which provides process efficiency by 98%. And whatever the ultimate catechinnya quality is at a level of 97.4% catechin yangterlihat in Figure 4. This purification process is carried out at a temperature of 70 ° C and the ratio of raw material comparison of results of leaching and solvent is 1:7.

Overall, the extraction process catechin from Gambier starting from prepurification covering the initial extraction and leaching. Then followed by purification using ethyl acetate solvent or of the water at a temperature of 70° C. The efficiency of the extraction process catechin overall Gambier can be seen in Figure 7.

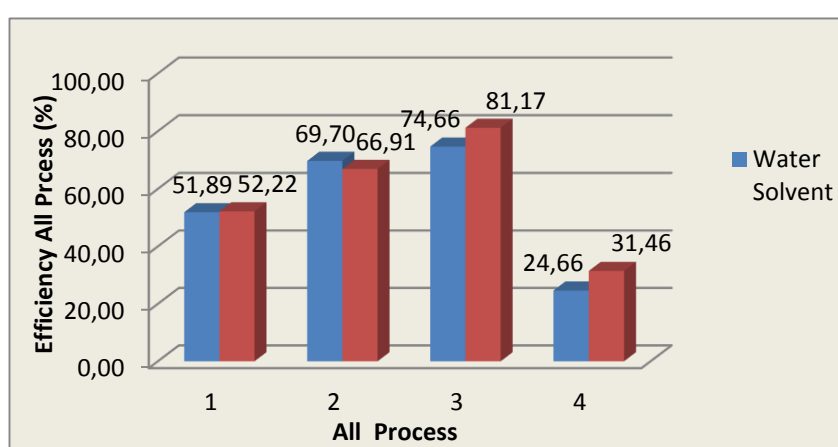


Figure 7. Efficiency of all process

From Figure 7 it can be seen that the working efficiency in the overall process highest in Ethyl Acetate solvent by 81.17%. It is seen from the many catechin acquisition of the initial content of the sample Gambier catechin 47.72% of 100 g which is 47.72 grams catechin obtained product with 97.4% of the catechin content of 39.77 g of product that is as much as 38.74 g catechinnya.

4. Conclusion

This study has been conducted to determine the best extraction process conditions and can be concluded that: For prepurification process, it was derived the best-laundering on the 700 ml volume, 70°C temperature, with the highest acquisition for sample 1 and 2 was 65.08 grams and 59,80 grams, with catechin levels were 71%, 89% and 69.83%. While the purification process it was obtained the best solvent is ethyl acetate, with the acquisition of catechin from water and ethyl acetate were 42.35 grams and 39.77 grams, respectively, with the acquisition of catechin levels were 84.13% and 97.4%. Thus, in the highest process work efficiency in the ethyl acetat solvent was 81,17%. This is from the number of acquisition of catechins from the initial content of catechin in gambier samples.

5. Acknowledgement

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