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Submission date: 21-Dec-2023 09:26PM (UTC+0700)

Submission ID: 2263663930

File name: EXTRACTORS_OF_CATECHIN_ISOLATION_FROM_COMMUNITY_GAMBIER_Unca.pdf (229.78K)

Word count: 2551

Character count: 13636

EVALUATION OF PRE-PURIFICATION EXTRACTORS OF CATECHIN ISOLATION FROM COMMUNITY GAMBIER (*Uncaria Gambiere Roxb*)

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Abstract. The pre-purification heat extraction process separates the catechins and tannins from the impurities present in the gambier. This process uses water as a solvent, at a ratio of 1: 7 to the sample, with a heating extractor, because at a temperature of 70°C, catechins and tannin dissolved in water. The solution is then filtered in a hot state. A cold process further extracted the catechins-tannin extract to separate the catechins and tannins, using a cooling extractor, to obtain sediment. A vacuum filter then filters the deposit to get pre-purification catechins with high levels of catechins. At the same time, the tannin passes from filtration and is accommodated in a tannin reservoir. Pre-purified catechins were dried in an oven at 50°C, then weighed and measured their catechins level using a spectrophotometer. This study aims to evaluate the prototype of catechins isolation extract from community gambier (*Uncaria Gambier*) since this extractor is newly designed and newly operated. The evaluation is done by varying the cooling temperature, the extractor cooling time by 3x washing onto the catechins yield. The highest sediment obtained from the 5°C temperature variation and 15 hours cooling time was 817.66 grams of 3 kg of dry gambier, at the highest catechins content of 96%. Thus it is concluded that the best performance of cooling extraction on gambier pre purification is still at 48.55%.

Key words: *Uncaria gambier*, Catechins, Tannin

INTRODUCTION

Gambier is one of the economic potentials plants for the West Sumatra community, especially for gambier farmers, because gambier is much needed for industry and pharmaceuticals, among others: as an antioxidant to inhibit the occurrence of atherosclerosis (Yunarto, N et al. 1, 2015) and as an anthelmintic, to inhibit the growth of worms. In addition, the ethyl acetate extract of gambier could act as a mixture-type inhibitor and serve as an effective mild steel corrosion inhibitor in aqueous solutions (Hussin, HM et al. 1, 2010). According to Bakhtiar (1991), the most widely used gambier chemical constituents are catechins and tannin. Catechin from gambier (*Uncaria gambier Roxb*) is a phenolic compound that has a potential as a natural antioxidant (Yeni, G et al, 2014). Gambier processing results in changes in the composition of the main content of gambier, namely catechins (Amos, 2010). A lot of research has been conducted to increase catechins content on the laboratory scale, among others:

From research Ferdinal, N (2014) and Nazir, N, (2000) that is. Purification of gambier is done by using water and activated charcoal of palm oil as absorbent. The levels of catechins obtained were only 72.4%, and a relatively high yield of 55, 25%. While Muchtar (2010) isolated catechins by using hot water at temperature 70°C, and several times of cold water wash, followed by drying at 50°C for 48 hours, to obtain 95% catechins, the yield is still low. However, can improve it the catechin level. Actually gambier with catechin levels above 90%, with prepurification method using water and extraction with etyl acetate has been obtained on a small scale (laboratory) (Rahmawati, N et al, 2012).

Furthermore, Rahman, E.D et al (2018) has conducted research about Determination of Catechins Extracting Process Condition from Gambier (*Uncaria Gambier Roxb*) Solok Bio Bio of Limapuluh Kota District, with prepurification method of various variations on the washing and solvent amount at catechins level. The results showed that the best solvent and material ratio was at 1: 7, with a water temperature of 70°C, 3 times washing, in cold temperature of 16°C.

During this time, the catechins pre-purification process is still on the laboratory scale. Therefore it is necessary to develop pre-purification technology on a pilot plant scale. Thus, it is required to make a prototype of a heating-cooling catechins extractor consisting of several processes, namely heating, extracting, cooling, filtration, and washing. Thus, it helps isolate the more considerable amount of catechins. The purpose of this research is to create a prototype of a pre-purification extractor and evaluate the tool's performance.

METHODOLOGY

Conducted this research through several stages: tool design, toolmaking, and tool performance testing.

A. Materials

1. The material used for the manufacture of a set of heating-cooling extractor prototypes is Stainless Steel 316 construction material (especially for food)
2. Added motor + pulley + belt for motors stirrer

While the materials used in the extraction process are gambier, water, pure catechins (99%), and ethyl acetate as a solvent on a UV-Vis spectrophotometer.

B. Equipment

The primary tool used is a prototype of a heating-cooling extractor, while the supporting equipment used are: oven and a filter container to separate the solution from the impurities. The extractor is designed in a cylindrical shape; the extractor cover has ellipsoidal-shaped, while the bottom surface has a cone-shaped, equipped with a stirrer for homogenizing material. The heater and cooling extension design circuit can be seen in Figure 1.

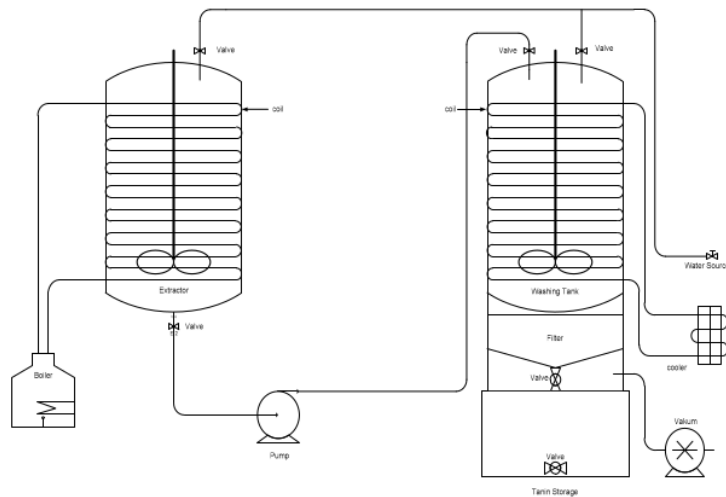


FIGURE 1. Extractor design circuit

The heating extractor designed has a diameter of 26 cm and a height of 60 cm. The liquid level and extractor diameter ratio is 2.3. The theory states, if the ratio of liquid level to extractor diameter > 1.4 then more than one impeller must be installed. Therefore, a 3-level stirrer is installed in this extractor. The extractor is also equipped with a coil that serves as a heater at a temperature of 70°C . The result of heating extractor design can be seen in Figure 2.

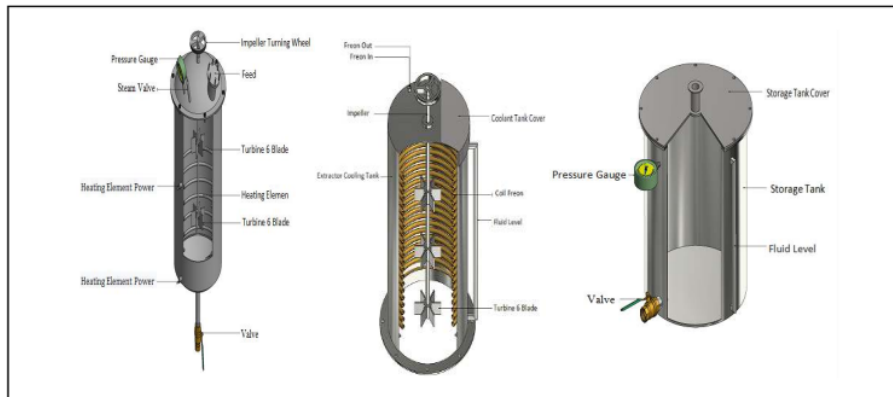


FIGURE 2. Design. Heater Extractor Coolant and tannin container

The cooling extractor is also designed with a diameter of 26 cm and a height of 60 cm, with a ratio of liquid level and diameter of extractor 2.3. In accordance with the theory that, if the liquid level to extractor diameter ratio > 1.4 then it must be installed more than one impeller, therefore this cooling extractor is also installed with 3 level of stirrer.

The cooling extractor is also equipped with a cooling coil that serves as a cooling at temperature of $5-16^{\circ}\text{C}$, and a filter at the bottom to separate tannins with catechins. The results of cooling, filter extractor and the tannin container design can be seen in Figures 2. This study will evaluate the performance of a heating and a cooling extractor on the catechins levels and other parameters following the results data (Rahman ED, et al 2018) on a laboratory scale with an initial sample catechins level of 57.7%

The catechins extraction process from gambier is done through two treatments, namely prepurification and purification process (Youfa, R et al, 2006). From the prepurification process the initial catechin will be obtained, thereby facilitate the further process, ie the purification process to obtain higher quality catechins, from gambier as raw material and water as solvent. Analysis of catechins content using UV-Vis spectrophotometer, conducted at Laboratory of University of Andalas Padang and Laboratory of Kopertis Region X Padang

RESULT AND DISCUSSION

This research is reviewed from 2 points of view, first is the tool design in the form of a heating-cooling extractor prototype, and second is the performance evaluation of heating-cooling extractor prototype. The prototype form of heating-cooling extractor can be seen in Figure 4 and Figure 5.

Performance Evaluation of the Heating and Cooling Extractor

Performance evaluation of heating and cooling extractors are reviewed from several points:

1. Amount of heating extraction content
2. Amount of cooling extraction content, using 3 times washing treatment in various cooling temperatures
3. Catechins levels

The performance evaluation of a heating extractor is a series of the prepurification process; gambier weighed as much as 3 kg, dissolved in 21 liters of water, at a ratio of 1: 7. at 70°C for 1 hour. The solution is then filtered to separate with the impurities. The heating extractor tool used can be seen in Figure 4. Heating extraction aims to dissolve all the catechins in the gambier, filtered, so as to obtain filtrate and impurities.

The filtrate is then fed into the cooling extractor. The gambier extract is cooled from a temperature of 30°C to 5°C , with a variations of cooling time at 5, 10 and 15 hours. The gambier extract was washed 3 times in this process, with a sample and water ratio of 1: 7, thus produced sediment and liquid. Sediment and liquid obtain

then separated by vacuum filtration at a pressure of 0.8 to 0.2 atm. The purpose of this cooling extraction is to purify the earliest acquisition of catechins.

The results and data performance of the heating extractor on the catechins obtained can be seen in Table 1.

TABLE 1. The Heating Extractor output, the impurities and catechins content

Sample Number	Filtrate		Impurities		% Impurities Content
	Weight (gr)	% catechins	Weight (gr)	% catechins	
Sample 1	23802	83	50	6	0,21
Sample 2	23820	85	45	7	0,19
Sample 3	23790	72	48	5	0,20

Table 1 shows the heating extraction process, with variations of samples 1, 2, and 3 at processing temperature of 70°C. This process produces filtrate containing tannins and catechins, with catechins content about 72-85%, and filtration impurities of 0.19 to 0.21%. The catechins percentage in the impurities is relatively small, ie between 5 - 7%. This shows that all the catechins are already dissolved in the heating extract. The form of liquids, sediment, and impurities from heating extraction can be seen in Table 2.

TABLE 2. The picture of filtrate, catechin sediment, and impurities on heating extraction




Process	Filtrate	Katechin sediment	Impurities
Heating Extraction			

Table 2 shows that heating extraction process after cooling to a temperature of 5°C resulting a sediment and filtrate. The light brown sediments is catechins, while the solid red filtrate is tannin, as shown in Table 1. The impurities are blackish red, with a rather coarse particle size. The amount of impurities can affect many levels of catechins, if not separated (Portier, G, 2010)







The catechins quality from heating extraction is still low, so it needs to be washed. The washing process at the laboratory scale has been done by Rahman, E.D, et al (2003). This method is further applied to the cooling extraction prototype. The results of cooling extractor performance with variation of cooling time to the output and catechins content at temperature 30-5°C can be seen in Table 3.

TABLE 3 Variations of cooling Temperature on the output and catechins levels

Washing Number	Sample 1 (5 hours)		Sample 2 (7 hours)		Sample 3 (15 hours)	
	gr	% catechin	gr	% catechin	gr	% catechin
Washing 1	798	72	867	85	798	83
Washing 2	702	68	566	88	702	88
Washing 3	477	61	406	81	477	96
Extractor efficiency	18,1 %		28,70 %		48,55 %	

From Table 3 it can be seen that cooling extraction performance has an effect on cooling duration. The longer the cooling time, the more efficient the appliance works. This can be seen from the weight of the sediment in each wash. The highest efficiency was seen in 15 hours cooling time, which was 48.55%. This result is still not optimal yet, because the filter process has not functioned optimally. In the cooling extraction process there is a separation between the solvent-shaped tannins and the sediment-shaped catechins at low temperature (30 to 5°C). The solution is then filtered and the catechins levels in the sediment are measured. Thus it can be concluded that the cooling extraction filtering process is still not optimal to obtain maximum sediment. The form of liquid and sediment on washing in the cooling extraction process can be seen in Table 4

TABLE 4. The Picture of filtrate, catechinsediments, and impurities on heatingextraction process

Washing number	Filtrate	Sediments
Washing 1		
Washing 2		
Washing 3		

From Table 4 can be seen the form of filtrate and sediment catechins obtained in each washing. The observation on the filtrate liquid color show, the more frequently washed, the higher the purity level of catechins received; nevertheless, more catechins are carried out in the washing liquid. Sousa, A., et al. (2008), in comparison, the sediment catechins color getting brighter than light brown in the heating extract.

CONCLUSION

1. The research of pre purification process using a heating-cooling extractor prototype obtained the highest catechins sediments as 817,66 gram from 3 kg of dried gambier, with the highest rate of catechins of 96%, at 15 hours cooling process.
2. Cooling extractor efficiency performance on gambier pre-purification is still 48.55%.

ACKNOWLEDGEMENTS

Acknowledgments are given to all parties related to this research, namely: to the Department of Research, Technology and Higher Education, who has funded this research through PTUPT grant, and all the guidance students who helped this research.

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