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THE EFFECT OF TIME EXTRACTION DURING LLE (LIQUID LIQUID EXTRACTION) ON THE CONTENT OF COUMARIN COMPOUNDS IN CINNAMON WATER EXTRACT POWDER PREPARATIONS

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ABSTRACT

Cinnamon is a plant that is widely processed and utilized. However, the presence of coumarin in cinnamon causes negative trade issues. This study aimed to determine the adequate solvent volume to remove the coumarin compound content in cinnamon water extract powder. The extraction method used was digestion and Liquid-Liquid Extraction (LLE) with variations in extraction time of 5, 10, 15, 20, and 25 minutes, while the method for identifying coumarin compounds was Thin Layer Chromatography (TLC). The results showed that the yield of extract powder for each variation carried out was ± 3.0%, the drying shrinkage test obtained a result of ± 0.9%, and the TLC test obtained a negative band result at the best time of 20 minutes. The resulting extract powder was slightly reddish brown, had a distinctive cinnamon odor, and a slightly astringent taste. The greater the solvent used, the smaller the amount of coumarin compounds in the extract.

Keywords: LLE, Cinnamon, TLC, Coumarin, Extract Powder

1. Introduction

Indonesia is the largest cinnamon-producing country. So far, Indonesian cinnamon bark has a big influence on the world market. According to Sundari (2002), in 1991, 94.1% of cassava in world trade came from Cinnamomum burmannii from Indonesia, while 4.2% came from Sri Lanka. As much as 80% of cinnamon in Indonesia is produced in West Sumatra, which is known as the center of cinnamon bark (cassia vera).

Cinnamon bark, leaves, and oil are processed, used as a flavoring for food and drinks, and widely used in cosmetics. In addition, cinnamon is also beneficial for health, including protecting the stomach. (Bambang,2012)

The presence of coumarin in Cinnamomum burmannii creates negative issues in trade because of biological activities such as anticoagulants, which are not needed for food and beverage products. This can impact the decline in the competitiveness of cinnamon worldwide.

Based on the description, this study aims to determine the adequate solvent volume to remove the coumarin compound content in cinnamon water extract powder.

2. Material and Methods

Materials

This research process uses the following materials: Cinnamon, PW Water, Dichloromethane, n-hexane, Ethyl Acetate, NaOH, and Silica Gel Plate 60 F254 with aluminum support.

Tools

This research process uses the following tools: 1000mL Beaker glass, 500mL Separating funnel, 1000mL Measuring cup, 100mL

Measuring cup, 2000mL Duran bottle, 100 and 25mL Measuring flask, 5mL volume pipette, 2000mL Erlenmeyer vacuum, Filter paper, TLC Sprayer, Heat gun, Magnetic stirrer bar, Oven, Hot plate with stirrer, Bulp, Automatic TLC sampler, Imaging TLC, Vacuum pump, Orbital shaker, 10x10cm TLC Chamber, Rotary evaporator.

Research Methodology

Cinnamon Extraction Preparation

The dried cinnamon bark weighed as much as 100 g. 500 mL of pw water was added, and then it was digested for 6 hours at 60^{o}C. Furthermore, filtration was carried out and continued with Liquid-liquid extraction (LLE) using 200 ml of dichloromethane with time variations of 5, 10, 15, 20, and 25 minutes. The LLE results were then concentrated using a rotary evaporator and dried using an oven at 70^{o}C.

Analysis Method

Yield Analysis

To determine the yield of the extract powder produced, the extract powder is weighed and then calculated using the formula:

Organoleptic Analysis

Organoleptic testing is a test based on the sensing process. The parts of the body that play a role in sensing are the eyes, ears, sense of taste, smell, and touch. The ability of the senses to provide an impression or response can be analyzed or distinguished based on the type of impression. These abilities include the ability to detect (detect), recognize (recognize), distinguish (discriminate), compare (scaling), and the ability to express likes or dislikes (hedonic) (Saleh, 2004).

Drying Loss Analysis

The drying loss parameter measures the remaining extract after drying at 105^{o}C for 30 minutes or until constant weight is expressed as a percentage value. In special cases (if the material does not contain volatile oils/essentials and residual organic solvents evaporate), it is identical to the water content. The value or range of permissible water content is related to purity and contamination (Emilan et al., 2011).

Thin Layer Chromatography Analysis

TLC tests were conducted to identify coumarin compounds in the extract powder after separation.

Research Results and Discussion

Yield Analysis Results

Based on the results obtained in Table 1, the most significant yield is at a time variation of 10 minutes, with a result of 3.0055%.

Table 1. Yields Calculation Results

LLE Time Variation (minutes)	Sample of Weight (g)	Yield of Weight (g)	% Yields (w/w)
0	50.0024	1.5009	3.0017
5	50.0024	1.5021	3.0041
10	50.0024	1.5028	3.0055
15	50.0024	1.5022	3.0043
20	50.0024	1.5017	3.0033
25	50.0024	1.5014	3.0027

Nurhayati et al. (2009) stated that a high yield value indicates the amount of bioactive components contained. According to Dewastisari (2018), the yield value is related to plants' bioactive content. Budiyanto (2015) stated that the higher the extract yield, the higher the content of substances attracted to a raw material.

The yield calculation results in the cinnamon water extract preparation show that the highest yield, 3.0055%, was obtained in the LLE process using 200 mL of dichloromethane.

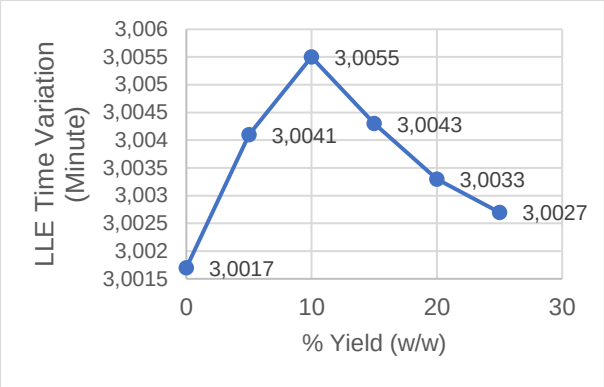


Figure 1. LLE Yields of Result

From figure 1. the variation in the volume of dichloromethane addition does not significantly affect the yield obtained. It is because the coumarin compound dissolved in dichloromethane does not cause a difference in the weight or volume of the cinnamon water extract, and the remaining dichloromethane that may still be contained in the water extract is also lost in the concentration process using a rotary evaporator and oven drying.

Organoleptic Analysis Results

The organoleptic analysis showed that the cinnamon water extract powder with various variables had a slightly reddish brown colour, a distinctive cinnamon odour, and a slightly astringent taste.

Organoleptic observations were conducted to determine whether each variable performed differently in terms of physical differences, colour, shape, and aroma. The results showed that the variables performed did not affect the description, taste, and aroma of cinnamon water extract powder.

Drying Loss Analysis Results

Based on the results of the drying loss test in Table 2, the extract powder with a time variation of 10 minutes produced the largest LOD, namely 0.97%.

The drying loss parameter measures the remaining extract after drying at 105oC for 30 minutes or until constant weight is expressed as a percentage value. In special cases (if the material does not contain volatile oils/essentials and residual organic solvents evaporate), it is identical to the water content. The value or range of permissible water content is related to purity and contamination (Emilan et al., 2011).

Table 2. Drying Shrinkage Test Results

Extraction Time Variation (minute)	Yields of Weight (g)	%LOD (w/w)
0	0.545	0.92
5	0.539	0.93
10	0.515	0.97
15	0.534	0.94
20	0.535	0.93
25	0.545	0.92

The water content in traditional medicine preparations, including extracts, should be at most 10% (Ministry of Health of the Republic of Indonesia, 1994). Water content exceeding 10% can cause the extract to be quickly overgrown with mould (Isnawati & Arifin, 2006). Therefore, hydrotropic extracts must be dried again before being used for pharmacological activity tests or made into dosage forms. The low water content will prevent the growth of microorganisms and mould (fungi).

Figure 2 shows that variations in the volume of dichloromethane addition do not significantly affect the %LOD results. Variations in samples' %LOD measurement results are estimated to be due to differences in contact time between the extract powder and air during the refining and packaging processes. A high %LOD can occur if the cinnamon water extract powder storage process needs to be carried out in the right place because the extract can absorb water in the air.

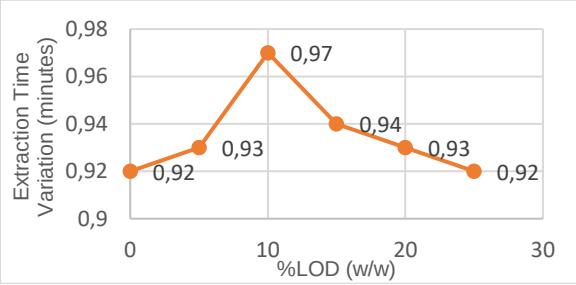


Figure 2. Drying Shrinkage Results

Thin Layer Chromatography Analysis Results

The results of the TLC test showed that the adequate volume for removing coumarin in cinnamon extract was 20 minutes in 200 ml of dichloromethane compared to 200 mL of cinnamon extract.

It can be seen in Figure 3. that in the cinnamon extract without LLE treatment, there is a coumarin compound with a reasonably strong luminescence compared to the 10ppm coumarin standard. While in the extract with variations in extraction time of 5, 10, and 15 minutes, the luminescence is very weak, even at variations in extraction time of 20 and 25 minutes, the luminescence is no longer visible; this indicates that the coumarin compound content has disappeared or is still present in tiny amounts.

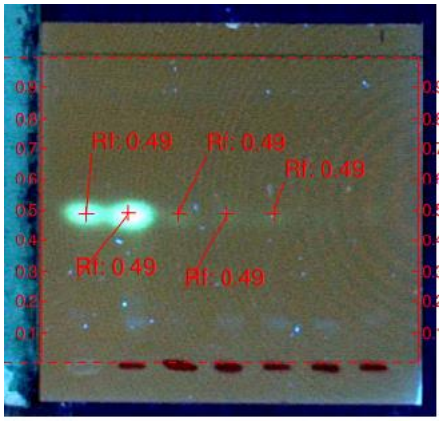
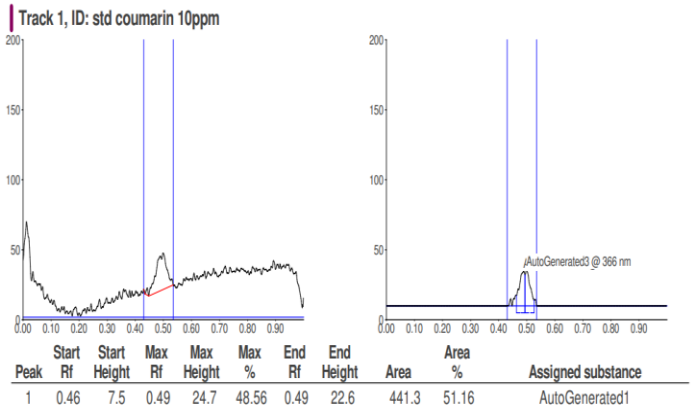


Figure 3. TLC Illumination Results at 366 nm

In Figures 4 and 5, it can be seen that the densitometry results of the extract without LLE treatment provide a much larger area compared to the 10ppm coumarin standard; this indicates that the coumarin compound content in the cinnamon water extract without LLE treatment is more than 10 ppm, which is 48.41 ppm. Variations in extraction time of 5, 10, 15, 20, and 25 minutes, the area is not readable; this indicates that the coumarin compound content has disappeared or is still present in tiny amounts.



Peak	Start Rf	Start Height	Max Rf	Max Height	Max %	End Rf	End Height	Area	Area %	Assigned substance
1	0.46	7.5	0.49	24.7	48.56	0.49	22.6	441.3	51.16	AutoGenerated1

Figure 4. Densitometry Results of Coumarin Standard 10 ppm

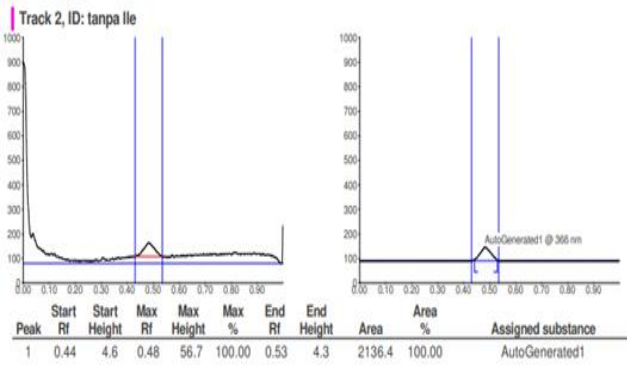


Figure 5. Densitometry Results of Cinnamon Extract Without LLE

Conclusion

This study concluded that the best LLE time required to remove coumarin compounds was 20 minutes in a volume of 200 ml of dichloromethane against 200 ml of cinnamon water extract. Variation of LLE time did not affect the amount of final yield, obtained a yield of ± 3% (w/w). Variation of LLE time did not affect the %LOD of cinnamon water extract powder, obtaining a %LOD of ± 0.9%.

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