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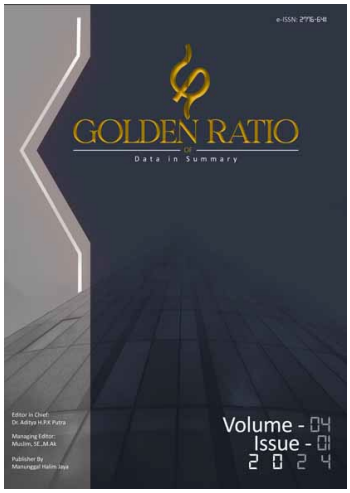

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DESCRIPTIVE OF QUANTITATIVE DATA | SUPPLEMENTARY

Effect of Oven Drying Temperature on The Tanin Content of Bungur (*Lagerstroemia speciosa* Auct. non (L.) Pers) *Leaves*

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Abstract: A study was conducted on the effect of oven drying temperature on tannin content of bungur leaves *(Lagerstroemia speciosa Auct. non (L.) Pers)*. Bungur leaves were picked, dry sorted, washed, wet sorted, and drained then oven dried at 30oC, 60(o)C, 90oC respectively. After drying, the samples were then measured for moisture content and then made into bungur leaf powder. Bungur leaf powder was then extracted by modified maceration using aquadem solvent. The extract obtained was tested qualitatively and quantitatively. Qualitative test results showed the presence of tannins. Quantitative tests using permanganometry, obtained tannin levels at drying temperatures of 30°C, 60°C, and 90°C were 7.27% ± 0.1429, 15.26% ± 1.0610, and 9.92% ± 0.7156, respectively. The highest tannin content was obtained at 60°C drying temperature.

Keywords: Drying Temperature, Tannins, Lagerstroemia speciosa Auct. non (L.) Pers, Bungur Leaf.

1. INTRODUCTION

Medicinal plants have long been used for treatment and management of various health problems and have been passed down from generation to generation until now. Along with the rapid development of science and technology, various studies have been conducted aimed at the use of quality, safe and effective herbal medicines. Basically, efforts to obtain quality vegetable natural medicinal raw materials can be taken several steps starting from ensuring the quality of raw materials, handling includes post-harvest, namely wet sorting, washing, changing the shape (if necessary), draining, drying, dry sorting, packing and storage (Katno, 2008).

The drying process is one of the most important stages because in addition to affecting the quality of simplisia physically, it will also affect the content of active compounds, especially for compounds that are thermolabile. Similarly, with medicinal plants, one of the active compounds of which is tannin, the drying process is expected to affect the difference in tannin levels contained in these plant parts (Asmara, 1980).

Tannins are much needed in both the industrial and health fields. In the health sector, tannins have uses as digestive tract astringents and skin abrasion, treating burns (weak antiseptics) (Tyler et al., 1976). Phenol groups contained in tannins cause astringent, antiseptic, and color effects with iron salts (Trease and Evan, 1996). Tannins are usually also called tannic acid or galloranian acid. Tannins are in the form of yellowish to light brown shiny flakes or amorphous powders, odorless, or slightly smelly (Ministry of Health RI, 1995). The solubility properties of tannins are very soluble in water, soluble in alcohol, and soluble in acetone, soluble 1: 1 in warm glycerol, practically soluble in preteleum, chloroform and ether (Reynold, 1996). Tannins are natural compounds with a molecular weight of 500-3000, with several free phenol hydroxy groups, forming stable bonds with protein biopolymers (Karamać, 2007).

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For the quantitative determination of tannins by the permanganometric method, the bungur leaves obtained a tannin content of 4.10%. The tannin content in bungur leaves is greater than the stem (Susanto, 2007). So that in this study using the leaves of the Bungur plant (*Lagerstroemia speciosa* Auct. non (L.) Pers) from the Lythraceae tribe which contains more tannins than the stem of the Bungur plant. The content of active compounds contained in Bungur plants is one of tannins (Lemmens, 1992). Parts of the Bungur plant that are usually used as medicine are seeds, leaves, bark (BPPT, 2011).

One of the variables in the drying process is the drying temperature. Drying can be done between 30°C - 90°C and the best temperature can be used at 60°C (Agoes, 2009). In a previous study, the effect of physical parameters on the tannin content of *Caesalpinia coriaria* was tested. One of the physical parameters seen is the temperature and time of extraction, the results obtained show that the best extraction temperature for tannin content is 90 °C at 0.748 mg/ml, and the best extraction time is for 30 minutes at 0.786 mg/ml (Lokeswari, 2011)

Analysis of tannin levels in a simplisia can be done by several methods, namely gravimetry, volumetry / permanganometry with titration, and colorimetry with Prussian blue reagent spectrophotometrically (Rahayu, 2012). One of the commonly used methods is permanganometry, because this method is one of the specific volumetric analyzers that can calculate the amount of tannin compounds contained in the bungur leaves (Materia Medika Indonesia, 1995). By previous research on "Determination of Tannin Type and Determination of Tannin Content of Cooked Banana Fruit Peel (*Musa paradisiaca* L.) Spectrophotometrically and Permanganometrically", that in determining the tannin content of kepok banana fruit peel by spectrophotometry the results were 2.45%, while by permanganometry the results were 0.8%, this shows that the method of determining levels by means of permanganometry is better for determining tannin levels than spectrophotometry (Rynata, 2014).

This study is intended to determine how the effect of temperature in the drying process on tannin levels contained in bungur leaves. In this case, the researchers compared the tannin content in bungur leaf simplisia that had been dried by artificial drying using an oven at different temperatures, namely 30°C, 60°C, and 90°C in the same period of time.

The plant material used in this study was the leaves of bungur plants (*Lagerstroemia speciosa* Auct. Non (L.) Pers) which are old with the provisions after 4 rows from the end. This material was taken around May 2015 in the city of Surabaya Prapen area which was then determined bungur plant (*Lagerstroemia speciosa* Auct. Non (L.) Pers) at the Traditional Medicine Development Center at the Faculty of Pharmacy, University of Surabaya (Appendix 1).

Figure 3.1 (a) Bungur leaf and (b) Bungur tree

The chemicals used for this research are as follows: Aqua demineralized, Oxalic Acid ($H_2C_2O_4$) p.a, $KmnO_4$ p.a, H_2SO_4 4 N, Indigo Sulfonic Acid, Potassium ferricyanide reagent, Ammonia, $FeCl_3$ p, and Gellatin 1% which has NaCl in it.

https://doi.org/10.52970/grdis.v4i1.1001

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The tools used in this research are: Laboratory glassware, oven, blender, mesh 20 sifter, *moisture content balance* (Mettler Toledo), analytical *balance* (OHAUS), drip board, burette (Pyrex), volume pipette (Pyrex), *magnetic stirrer* (Cimerec ® 2), *magnetic bar*, lighter, filter paper, drip pipette, wooden clamp, stative & holder.

2. RESEARCH DESIGN AND METHOD

The part taken is the old leaves on one tree by picking manually, then the bungur leaves are collected, washed, wet sorted, drained, then the group of bungur leaves is then dried in the oven at 3 different temperatures with a drying time of 7 hours. The bungalow leaves were divided into 3 groups (groups A, B, and C) where each group consisted of 3 samples, each group was given a different treatment. Group A was oven-dried at 30°C, while group B was oven-dried at 60°C, and group C was oven-dried at 90(°)C as well.

Extraction of bungur leaf powder was carried out, the extract was obtained by modified maceration (Rynata, 2014).

a. Bungur leaf powder was carefully weighed ± 2 grams and put into a beaker glass.
b. After that, it was put into a glass beaker containing bungur leaf powder with 50 ml of boiling water, while stirring on a water bath for 30 minutes.
c. Allow to stand for a few minutes, then pour through a cotton swab/filter paper into a 250.0 ml volumetric flask.
d. The remainder of the beaker glass was re-distilled with boiling water and filtered the solution into the same volumetric flask.
e. The distillation is done several times until the solution does not show a color change to blue black when reacted with $FeCl_3$.
f. The solution was cooled and added aquadem to 250.0 ml quantitatively into a volumetric flask. (Materia Medika Indonesia, 1995)

Qualitative tannin tests are used to determine the presence of tannins in several ways, including:

a. The extract plus $FeCl_3$, gallotanin and ellagotanin will give a blue-black precipitate and condensed tannin will give a greenish-black precipitate (Rynata, 2014).
b. Gelatin test

The extract is added with 1% gelatin solution containing NaCl, if a precipitate appears, it means that it contains tannins (Trease and Evan, 1996).

The determination of tannin content in this study was determined by permanganometry (Materia Medika Indonesia, 1995).

Determination of tannin content of bungur leaves with $KMnO_4$

a. 25.0 ml of bungur simplisia extract was pipetted, transferred into a 1,000 ml erlenmeyer, 750.0 ml of water and 25.0 ml of indigo sulfonic acid L.P. were added.
b. Then titrated with 0.1 N $KmnO_4$ until the solution is golden yellow. And recorded the volume of $KMnO_4$ used.
c. Three replications were performed. (Materia Medika Indonesia, 1995)

The formula for calculating tannin content by permanganometry:

$$\overline{[(a-c) \times 0{,}004157) \times {}^{10}/_{b}] \times 100\%}$$

Description:

a = $KMnO_4$ volume (ml)

b = Sample weight (gram)

c = Blank Volume (ml)

Equivalence of 1 ml of $KMnO_4$ 0.1N is equivalent to 0.004157 grams of tannin. (Rynata, 2014)

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a. Pipette 25.0 ml of water into an erlenmeyer, add 750 ml of aquadem, then add 25.0 ml of indigo sulfonic acid.
b. Then titrated with KmnO $_4$ until the solution is golden yellow. The volume of $KmnO_4$ used was recorded.

Three replications were performed. (Materia Medika Indonesia, 1995)

3. RESULT AND DISCUSSION

Data on tannin content in bungur plant leaves (*Lagerstroemia speciosa* Auct. Non (L.) Pers.) based on the drying temperature was determined permanganometrically, then calculated **(Katno *et al.*, 2008).** The results of the calculation of the levels were averaged and then presented in the form of graphs and compared the tannin levels of each drying temperature. The results of qualitative determination of the presence of tannins were carried out on bungur leaf extract. The observation data can be seen in table 1.

Table 1. Qualitative Determination of The Presence of Tannins

No.	Reagents	Results	Tannins
1	FeCl3	Blue-green	+
2	Saline solution + 1% gelatin	Presence of Deposits	+
3	Potassium ferrycyanide + ammonia	Red	+

The results of the determination of tannin content of bungur leaves by permanganometry after drying based on temperature can be seen in table 2, table 3, and table 4.

Table 2. Results of Determination of Tannin Content of Bungur Leaves at 90°C Drying Temperature

No.	Sample Weight (g)	Normality KMnO4 (N)	Vol. Titrant (ml)	Vol. Blank (ml)	Tannin Content	
1	2,0022	0,0964	0,00 - 7,00	0,00 - 1,70	10,61%	X = 10,57% SD = 0.0577 KV = 0.54%
2			0,00 - 6,95	0,00 - 1,70	10,51%	
3			0,00 - 7,00	0,00 - 1,70	10,61%	
1	2,0022	0,0972	0,00 - 6,10	0,00 - 1,60	9,08%	X = 9,15% SD = 01101 KV = 1.20%
2			0,00 - 6,11	0,00 - 1,60	9,10%	
3			0,00 - 6,20	0,00 - 1,60	9,28%	
1	2,0023	0,0984	0,00 - 6,30	0,00 - 1,50	9,81%	X = 10,04% SD = 0.3272 KV = 3.25%
2			0,00 - 6,35	0,00 - 1,50	9,91%	
3			0,00 - 6,60	0,00 - 1,50	10,42%	
X+ SD						9,92%±0,7156
KV						7,23%

Table 3. Results of Determination of Tannin Content of Bungur Leaves at 60°C Drying Temperature

No.	Sample Weight (g)	Normality KMnO4 (N)	Vol. Titrant (ml)	Vol. Blank (ml)	Tannin Content	
1	2,0042	0,0990	0,00 - 8,65	0,00 - 1,70	14,27%	X = 15,13% SD = 1.0713 KV = 7.08%
2			0,00 - 9,65	0,00 - 1,70	16,33%	
3			0,00 - 8,90	0,00 - 1,70	14,79%	
1	2,0043	0,0961	0,00 - 8,70	0,00 - 1,60	14,15%	X = 14,27% SD = 0.8174 KV = 5.73%
2			0,00 - 9,20	0,00 - 1,60	15,15%	
3			0,00 - 8,39	0,00 - 1,60	13,53%	

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No.	Sample Weight (g)	Normality KMnO4 (N)	Vol. Titrant (ml)	Vol. Blank (ml)	Tannin Content	
1	2,0065	0,0956	0,00 - 10,10	0,00 - 1,70	16,64%	X = 16,38% SD = 0.2254 KV = 1.37%
2			0,00 - 9,90	0,00 - 1,70	16,24%	
3			0,00 - 9,91	0,00 - 1,70	16,26%	
X+ SD						15,26%±1,0610
KV						6,95%

Table 4. Results of Determination of Tannin Content of Bungur Leaf at Drying Temperature 30°C

No.	Sample Weight (g)	Normality KMnO4 (N)	Vol. Titrant (ml)	Vol. Blank (ml)	Tannin Content	
1	2,0020	0,09570	0,00 - 4,65	0,00 - 1,69	5,88%	X = 6,08% SD = 0.2 KV = 3.29%
2			0,00 - 4,85	0,00 - 1,69	6,28%	
3			0,00 - 4,75	0,00 - 1,69	6,08%	
1	2,0856	0,09552	0,00 - 5,60	0,00 - 1,85	7,15%	X = 7,27% SD = 0.1430 KV = 1.97%
2			0,00 - 5,75	0,00 - 1,85	7,43%	
3			0,00 - 5,65	0,00 - 1,85	7,24%	
1	2,0013	0,09309	0,00 - 4,89	0,00 - 1,80	6,19%	X = 6,42% SD = 0.2212 KV = 3.44%
2			0,00 - 5,11	0,00 - 1,80	6,63%	
3			0,00 - 5,02	0,00 - 1,80	6,45%	
X+ SD						6,59%±0,6130
KV						9,30%

Table 5. Comparison of Tannin Content at Each Drying Temperature

Group	Sample	Tannin Content (% w/b)	Average tannin content (%b/b ± SD)
A	1	6,08	6,59%±0,6130 KV = 9.30%
	2	7,26	
	3	6,42	
B	1	15,13	15,26%±1,0610 KV = 6.95%
	2	14,27	
	3	16,38	
C	1	10,57	9,92%±0,7156 KV = 7.23%
	2	9,14	
	3	10,04	

Table caption:
Group A = Drying temperature 30°C
Group B = Drying temperature 60°C
Group C = Drying temperature 90°C

In this study, a comparison of tannin content in bungur leaves based on oven drying temperature (30°C, 60°C, and 90(o)C) was conducted. Based on previous research on Dutch teak leaves, the best drying time is 8 hours with the highest tannin content, which is about 0.6440 ± 0.0183% b/w, then 7 hours with tannin content of 0.5684 ± 0.0096% b/b and 6 hours of drying with tannin content of 0.4833 ± 0.0254% b/b **(Katno *et al.*, 2008).** However, due to the insignificant difference between tannin levels at 7 hours and 8 hours, supported by several internal factors, the researchers used a time of 7 hours for drying.

The first step taken is the drying process of the simplisia which was previously picked from the tree, dry sorted, washed one by one, then carried out wet sorting and finally penetiran. Furthermore, the drained simplisia was dried in the oven at a predetermined temperature and time (30°C, 60°C, and 90(o)C, each for 7 hours). After drying and measuring the moisture content of bungalow leaf simplisia, then pollination is carried out by blending and sifting using a mesh 20 sieve so that bungalow leaf powder with the same particle size is produced. Furthermore, the extraction of bungur leaf powder is

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carried out by means of modified maceration where bungur leaf powder is weighed ± 2 grams and then dissolved in 50 ml of boiling aquademineralisata, then heated and stirred on a water bath for 30 minutes, then filtered. This is done so that tannins can be extracted in water, because basically tannins are soluble in water **(Reynold, 1996)**. From this, the pulp and filtrate will be obtained, the filtrate is collected, the pulp is re-dried in the same way until the pulp when reacted with $FeCl_3$ is not greenish blue. The collected filtrate was put in a 250.0 ml volumetric flask, then added aquademineralized to the mark

In this study, a qualitative test was also carried out to determine the presence of tannins contained in bungur leaves. The reagents used in this qualitative test are $FeCl_3$, Lar. gelatin salt, and $K_3Fe(CN)_6$ plus ammonia. Qualitative tests carried out with $FeCl_3$ form a blue-green complex due to the presence of phenol groups in tannins that bind to $FeCl_3$ to form a blue-green complex **(Depkes RI, 1979)**. Using salt solution plus gelatin produces a precipitate that indicates the presence of tannins **(Trease and Evan, 1996)**. This is because, the nature of tannins can be strongly bound to proteins, all tannins cause a little or a lot of sediment when added with gelatin, because gelatin includes natural proteins **(Harborne, 1995)**. With $K_3Fe(CN)_6$ plus ammonia formed a dark red color. The results of the tannin qualitative test can be seen in table 4.4.

The extract that has been obtained is used to determine the tannin content by the permanganometric titration method. The principle of this method is based on oxidation-reduction or redox processes. In this study, $KMnO_4$ was used as a standard oxidizing agent or secondary standard, because this compound is a strong oxidizer, often used, easily obtained, and affordable, and oxalic acid was used as the primary standard solution **(Rynata, 2014)**.

Determination of tannin content was carried out by the permanganometric titration method. The extract obtained in a 250.0 ml volumetric flask was pipetted as much as 25.0 ml, added 25.0 ml of indigo sulfonic acid, and titrated with $KMnO_4$ solution which had previously been standardized with oxalic acid. The end point of the titration is marked by a color change from blue to golden yellow **(Underwood and Day, 1998)**

From the results of the study, the average tannin content at 90°C oven drying was 9.91%, while at 60°C oven drying the tannin content was 15.25%, and at 30°C oven drying it decreased to 6.50% tannin.

The results showed that different drying temperatures using an oven affected the stability of tannin content. Tannin levels at 60°C drying were higher than those at 30°C, and 90°C drying. The tannin content of the results of drying at 30°C is lower than at 60°C, this can be caused because at 30°C the water content is still high, so it affects the weight of the sample when weighed, causing the tannin content in bungur simplisia at the drying temperature to be low. Meanwhile, the same thing happened at 90°C drying where the tannin content was lower than drying at 60°C.

Previous research conducted by Lokeswari (2011) on the isolation of tannins from *Caesalpinia coriaria* and the effect of physical parameters showed that, the optimum temperature for tannin extraction was at 90°C with the highest tannin content of about 0,748 mg/ml, while at 95°C and 100°C the tannin content decreased to 0.506 mg/ml, and 0.326 mg/ml, respectively, and previously at 75(o)C, 80(o)C, 85°C with tannin levels of 0.417 mg/ml, 0.476 mg/ml, and 0.496 mg/ml, respectively. This happens because high temperatures will cause tannins to hydrolyze, so tannin levels are getting lower **(Lokeswari *et al.*, 2011).**

Previous research conducted by Siringoringo (2012) on the study of making coffee leaf tea showed that the tannin content at a drying temperature of 80°C was higher at 4.94%, while the tannin content obtained at a drying temperature of 85(o)C, 90(o)C, 95(o)C was 4.68%, 3.90%, and 3.64% respectively. The difference in drying temperature causes differences in tannin content where the higher the drying temperature, the lower the tannin content. This is because the heat given causes tannins to break down into simpler compounds **(Siringoringo, 2012).**

The results showed that the decrease in tannin levels at a drying temperature of 90°C occurred because at that temperature the tannins were hydrolyzed so that the tannin levels read were lower than

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drying at 60°C, in addition to the nature of tannins that cannot withstand too high heat **(Oematan, 2015)**. Based on the results of the research that has been carried out, it can be concluded:

a. The tannin content of bungur leaves at a drying temperature of 30°C was 6.50%, the tannin content of bungur leaves at a drying temperature of 60°C was 15.25%, and the tannin content of bungur leaves at a drying temperature of 90°C was 9.91%.
b. The results showed that the best oven drying was at 60°C, because at that temperature the tannin content was greater than at 30°C and 90°C drying temperatures

4. CONCLUSION

This study aims to evaluate the effect of drying temperature on tannin levels in bungur leaves (Lagerstroemia speciosa Auct. Non (L.) Pers) which has been done by permanganometry method. The results showed that different drying temperatures produced varying tannin levels. At a drying temperature of 30°C, the tannin content was recorded at 6.50%; at 60°C, the tannin content increased significantly to 15.25%; while at 90°C, the tannin content decreased again to 9.91%. This data indicates that a drying temperature of 60°C is the optimal temperature to maintain the highest tannin content in bungur leaf simplisia. The decrease in tannin content at 90°C is thought to be caused by tannin hydrolysis that occurs due to excessive heat exposure, while at 30°C, the high water content is likely to affect the efficiency of tannin extraction.

This research makes an important contribution to the science and practice of medicinal plant processing, especially in optimizing the drying process to maintain the levels of active compounds. Tannins, as compounds that have various benefits in health and industry, are very important to be optimally utilized. This study shows that a careful drying process can affect the quality of medicinal plant raw materials, so this research is relevant to be applied in the pharmaceutical industry and herbal medicine development. The uniqueness of this study lies in exploring the effect of drying temperature on tannin content using a specific analytical method, namely permanganometry, which has proven to be more reliable than spectrophotometric methods in measuring tannin content. Therefore, the results of this study not only enrich the scientific literature, but also provide practical guidelines for the medicinal plant processing industry to ensure production efficiency and effectiveness.

However, this study has several limitations that need to be considered. First, the research only focused on one type of plant, namely bungur leaves, so the generalization of the results to other plants containing tannins still requires further study. Secondly, the test was conducted on a laboratory scale under controlled conditions, so the influence of external factors such as environmental humidity and oven type variations has not been explored. Thirdly, the drying duration was fixed at seven hours for all temperatures, although shorter or longer drying times at certain temperatures might result in different tannin levels. For future research, it is recommended that comparative studies be conducted on various types of medicinal plants with different tannin contents, as well as testing on an industrial scale to evaluate the effectiveness of this method under real production conditions. In addition, additional analysis using other chemical methods, such as spectrophotometry or chromatography, may provide a more in-depth picture of tannin stability under various processing conditions. Thus, this research opens up great opportunities for further development in the optimization of holistic processing of medicinal plant raw materials.

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