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Determination Of Optimum Drying Condition On Flavonoid Content Of *Ficus Carica L.* Leaves Using Tray Dryer

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A B S T R A C T

This research aims to find out the characteristics of the Ficus carica L. drying process using a tray dryer and determine the effect of temperature and drying time on Ficus Carica L. flavonoid content using a tray dryer to obtain Ficus carica L. herbal leaf tea which is high in antioxidants. At the experimental stage, the water content of fresh leaves of Ficus carica L. was determined, so the water content is 74 ± 1.11% (wet base), then dried using a tray dryer at 35; 40; 45; 50; and 55°C, with a variation of the drying air flow rate of 1.3; 1.6; 1.9; 2.2; and 2.4 m/s. The optimum temperature and drying air flow rate are 50°C and 1.9 m/s since the highest flavonoid content was obtained at those conditions as much 0.78 ± 0.001 gram QE/100gram extract. Dried products packaged in tea bags have a content of 23.60% carbohydrate, total fat of 3.50%, protein 5.35%, antioxidant of 154.25 ppm and a calorific value of 1430 Kcal/100 grams. It can be concluded that the Ficus carica L. drying process using a tray dryer can be used to produce herbal leaf tea which is high in antioxidants and flavonoid content.

INTRODUCTION

Ficus carica L., or ara in Indonesian, Kerma in Arabic and figuier in French. Kind of plant that belongs to the genus Ficus and the family Moraceae [1]. This plant is known as a native of Southwest Asia and has been grown in the Mediterranean since the ancient era [2].

Since many bioactive compounds, such as phenolic compounds, flavonoids, coumarins, sterols, and volatiles [3] contained in this plant, this particular plant can also be use as a medicine because it has biological activities such as antioxidant, anticancer, antibacterial, and others effects [1], [4].

There are some parts of this plant that can be use to extracted its bioactive compound such as its fruit, root, and leaves. In this study we will use Ficus carica commonly called as Tin leaves, which will be the primary focus of this study.

It is worth noting that Tin leaves exhibit remarkably high antioxidant activity [5]. Antioxidant and other chelating properties are part of the flavonoid class [6]. Flavonoids are important since their capability to act both as reducers and scavengers on free radical formation [7].

One popular method to extract flavonoids is by maceration technique, this method gives an advantage due to the low-cost method [8] despite its disadvantages that require a lot of solvents and takes a long time [9], [10].

A sample needs to dry a sample to vaporize the water content before extraction. The drying process is a crucial step in manufacturing many chemical products the objective of the drying process is to remove moisture from a solid material to slow down the bacteria growth. Therefore it needs to optimize the dryer operating condition to achieve optimum flavonoid. Several studies have approached the task of finding the optimum variable during the drying process such as: a study of four different sample drying pretreatments on flavonoid and antioxidant activity of Dryopteris erythrose leaves as follows: Direct liquid nitrogen freezing-grinding, direct shade-drying, direct sun-drying, and direct heating at 75 °C [11]; Another study investigated the effect of drying technique by a method of freeze and hot-air oven form citrus waste [12]; Additionally, a research project investigating effects of two temperature were sets in this research as follows 24 °C and 40 °C on the total phenolics, antioxidant activity, and flavonoid contents of four common Mediterranean herbs as follows: Mint, Sage, Lemon balm, and Thyme [13]. Furthermore, a study focus on the effects of drying temperature variation on the phenolic content, the antioxidant activity of *Myrtus communis L.* leaves extraction using ethanol, the study was found at higher temperatures around 100 °C and 120 °C could impact the losses of amount extract [14].

This study aims to identify the optimal drying air temperature, drying time, and airflow rate to dry the *Ficus carica L.* leaves using a tray dryer, and find out the amount of Flavonoid extracted after the pretreatment process at those optimum conditions.

METHOD

Materials

In this study, we employed tray dryers as the primary equipment for the research process. We utilized fresh *Ficus carica L.* leaves sourced from Sariwangi, 95% ethanol, 10% $AlCl_3$, 1% quercetin, 5% acetic acid, 1 mg of magnesium powder, and HCl.

Experimental Apparatus

The Chemical Engineering Department of Politeknik Negeri Bandung supplied a tray drier with three trays for this research. This tray drier control the temperature and airflow during the drying process. We set up a tray dryer in a closed area to create a controlled environment with multiple trays. A blower was employed to circulate hot air within the dryer. The experimental approach involved three main steps: first, we measured the initial water content of the samples; then, we performed the drying process; and finally, we assessed the yield and final flavonoid content.

Procedure for Measuring the Initial Water Content

Accurate measurements of the initial water content in *Ficus Carica L.* leaves by weighing the leaves before and after drying. Conducting a water content analysis through this method is essential as it provides us with information about the initial concentration of water content.

Procedure for the Drying Process

The investigation was conducted at the Food Engineering Laboratory in the Chemical Engineering Department of Politeknik Negeri Bandung during sunny and cloudy weather conditions. The drying process involved achieving an equilibrium moisture content. The drying process consisted of two steps. Firstly, we adjusted the temperature while maintaining a medium airflow rate of 1.9 m/s. In this step, we measured the rate of water evaporation (R) and water content (Xt) at various time points and temperatures to identify the optimal drying temperature.

Next, we continue the drying process with various airflow while maintaining the optimal temperature from the first step. In this second step, we measure R and Xt to identify the optimal airflow. In both steps, we determine the optimum temperature and airflow while observing the pattern of R to identify the different stages of the drying process. **Table 1** refers to temperature and airflow variations as factor-1 and factor-2, respectively.

Table 1. Drying Variation

Factor-1: Drying Temperature (°C)	Factor-2: Air Flow Rate (m/s)
35	1.3
40	1.6
45	1.9
50	2.2
55	2.4

Procedure for Evaluating Yield and the Final Flavonoid Content

The yield is determined by comparing the final weight of the extracted material (extract) obtained through maceration with the initial weight of the dried fig leaves. Each extract was collected using 96% ethanol after maceration. Meanwhile, the final Flavonoid content was observed by measuring each extract by UV-Vis spectrophotometry, then the flavonoid content can be calculated using standard curve of certain flavonoid compound.

Data Analysis

After drying, we analyzed the data to determine the optimum temperature and airflow rate. We will consider the gradient of the drying characteristic, which will be explained in the subsequent chapter, to determine the optimal condition. Additionally, we measured the flavonoid content within this range to determine if the highest content was achieved at this condition.

RESULT AND DISCUSSION

In the previous chapter, we discussed the three main steps involved in drying Ficus Carical Linn. These steps are as follows:

Measuring the Initial Water Content

The average water content of Tin leaves (*Ficus carica L.*) was 74 ± 1.11% (wet basis) or approximately 2.85 grams of H2O per gram of dry sample, based on the initial water content measurement.

Drying Process

The drying process is generally divided into two zones: the constant-rate period (Rc) and the falling-rate period (Rf) [15]. The two zones are separated by a breakpoint called the critical moisture content (Xc) [15]. The critical moisture content is the moisture content at which the drying rate changes from constant to falling. Once the drying rate changes to falling, the equilibrium moisture content (X*) is reached when a material is in equilibrium with its environment.

Acknowledging that the drying characteristics may vary depending on the specific substance being dried is essential. Therefore, it is necessary to estimate the effect of drying temperature and airflow rate on drying characteristics before determining the optimum temperature and airflow rate.

Evaluation of Drying Characteristics

The curve displayed in **Figures 1** and **2** demonstrates a decrease in water content on the surface of the leaves during the Rc period, along with an Rf period indicating a reduction in water content within the leaf pores. Xc represents the transition point of water content between the Rc and Rf periods, while X* represents the equilibrium moisture content.

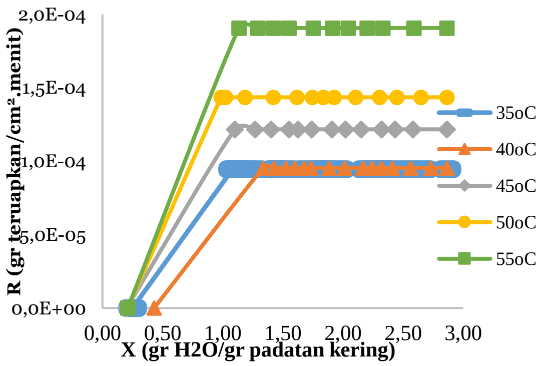

Figure 1. Drying curve with temperature variations and an airflow rate of 1.9 m/s

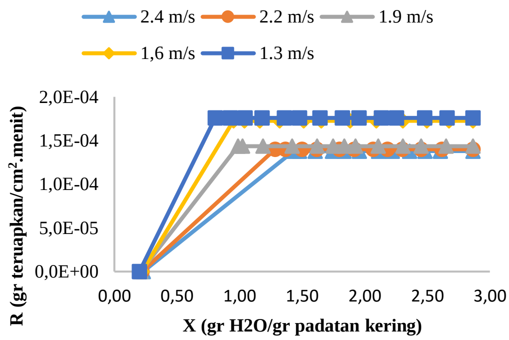

Figure 2. Variations in airflow rate and optimal temperature on the drying curve

The analysis presented in **Figure 1** and **2** demonstrates that the observed pattern represents the completion of surface water evaporation, followed by the evaporation of moisture content from the internal pores, indicates the presence of two distinct stages in the drying process: the period of constant drying rate and the period of decreasing drying rate. The constant drying rate (Rc) corresponds to the rate at which water content decreases during a stable phase. During this stage, water evaporation primarily occurs from the surface, exhibiting characteristics similar to ordinary evaporation.

A separate study observed that water transfer from the material occurs during the falling rate period, influenced by diffusion [16]. Hani et al. [17]conducted a study on the drying characteristics of potatoes, while Hestiningrum [18] examined the drying characteristics of leeks, revealing periods of Rc and Rf.

To provide a comprehensive understanding, **Table 2** and **Figure 3** and **4** illustrate the variations in the constant drying rate (Rc) at different temperatures while keeping the drying airflow constant. It is worth noting that higher drying air temperatures accelerate the drying rate. However, rapid drying can cause material degradation. In particular, when the surface dries too rapidly, it does not correspond proportionally with the water passage from the inside of the material to the surface. This process causes surface hardening, often called case hardening [19].

Table 2. Critical Moisture Content to Dryer Air Temperature

Factor-1: Constant Air Flow		Factor-2: Constant Temp.	
T(°C)	Xc	V (m/s)	Xc
35	1.08	2.4	1.35
40	1.34	2.2	1.20
45	1.10	1.9	0.99
50	0.99	1.6	0.86
55	1.13	0.2	0.75
Average	1.13	Average	1.03

Table 3. Constant Drying Rate at The Critical Moisture Content

Factor-1: Constant Air Flow		Factor-2: Constant Temp.	
T(°C)	R @Xc	V (m/s)	R @Xc
35	9.45E-05	2.4	1.76E-04
40	9.53E-05	2.2	1.73E-04
45	1.22E-04	1.9	1.44E-04
50	1.44E-04	1.6	1.40E-04
55	1.91E-04	0.20	1.38E-04

The critical moisture content (Xc) was determined for five different drying temperatures, and the average values are presented in Table 3. The Xc values for columns factor-1 and 2 were found to be 1.13 ± 0.11 and 1.03 ± 0.22 gram H2O/gram dry solid, respectively.

The impact of drying air temperature on the critical moisture content of dried fig leaves did not demonstrate a significant effect. Each type of material has a critical water content in a specific range because the critical water content is a material property. Nevertheless, it is noteworthy that the critical moisture content value obtained in this study aligns with the findings of Mujumdar and Devahastin [14], which suggest that the critical moisture content for vegetables and fruits is typically>0.8 grams H2O/gram dry solids. However, at a temperature of 40°C, the critical moisture content value deviates considerably. Hence, when averaging the data from the four available measurements, the value becomes 1.09 ± 0.06-gram H2O/gram dry solid.

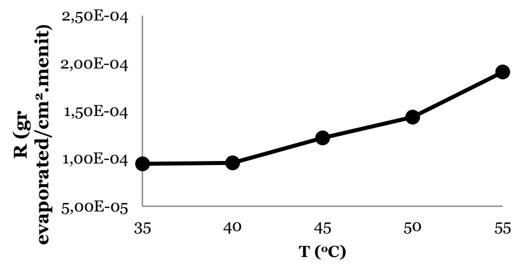

Figure 3. Relationship between constant drying rate and dryer air temperature at a fixed airflow rate of 1.9 m/s

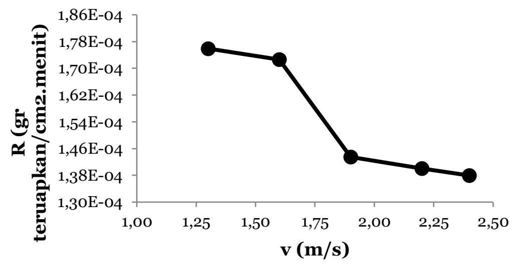

Figure 4. Relationship between constant drying rate and dryer air temperature at optimum temperature.

Table 4. Equilibrium Moisture Content to Dryer Air Temperature

Factor-1: Drying Temp. (°C)		Factor-2: Air Flow Rate (m/s)	
T(°C)	X*	V (m/s)	X*
35	0.25	2.4	0.23
40	0.43	2.2	0.22
45	0.20	1.9	0.21
50	0.21	1.6	0.22
55	0.21	0.20	0.20
Average	0.26	Average	0.22

The equilibrium moisture content (X*) is a solid material's minimum moisture level during drying under specific environmental conditions, including material properties, relative humidity, and surrounding air temperature.

The values of X* at different drying temperatures are presented in **Table 4**. This measurement indicates how sensitive the material is to microbial growth, which, if not controlled, can lead to damage or spoilage during storage [19].

The observed equilibrium moisture content at a drying air temperature of 40°C stands out from the rest of the data. This disparity can be attributed to variations in drying conditions, specifically the humidity of the air surrounding the dryer. During this particular instance, the humidity was measured at 81.57%. In contrast, the drying process for other temperature variations took place under humidity conditions ranging from 68% to 69%

The equilibrium moisture content of a material depends on ambient air humidity. In environments with high humidity, the air becomes saturated with water vapour, causing the material to retain more water due to low driving force. Conversely, when the air humidity is low, water can diffuse from the material's interior to its surface due to differential concentration. The higher water concentration in the air propels this diffusion process. However, as the air humidity increases, particularly near the surface, the concentration difference diminishes. Consequently, water is retained within the material, resulting in a higher equilibrium moisture content [20].

Since the operating temperature of 40°C showed different results compared to other data, it was considered an outlier and excluded from calculating the average moisture content of the equilibrium. The equilibrium moisture content obtained from the remaining four data points was 0.22 ± 0.02 grams $H2O$/gram dry solid.

The critical moisture content (X*) was determined for five different drying temperatures, and the average values are presented in **Table 3**. The X* values for columns factor-1 and 2 were 0.26 ± 0.09 and 0.22 ± 0.01 gram $H2O$/gram dry solid, respectively.

Estimating of Optimum Temperature

In order to determine the optimum temperature, we need to assess the gradient value at a specific temperature, as depicted in Figure 5. The gradient is calculated using the following formula:

$$Gradient(T\ °C) = \frac{R|_{X_C} - R|_{X^*}}{X_C - X^*}$$

Here, R|Xc and XC are obtained from Table 2 and 3, respectively, while R|X* is 0 since there is no mass transfer at the equilibrium moisture content.

As a result of drying at various temperatures in **Figure 5**, there is no significant change in gradient temperature at 35 °C and 45 °C. Meanwhile, when the drying temperature is set to 45°C, there is only a minimal change in the gradient. This observation suggests that the decline in the drying rate initiates at a temperature of 45°C. Then, the slope of the falling-rate period becomes gentler as the drying temperature increases from 45°C to 50°C. The gradient value increases again as the drying temperature goes from 50°C to 55°C. A more significant gradient value indicates a faster falling-rate period. The drying temperature affects the continuous diffusion activity. At lower drying air temperatures, achieving the same X* requires a slower drying rate than at higher drying air temperatures.

Based on the data obtained from drying fig leaves using various drying air temperatures, it can be determined that the optimal drying temperature is 50°C.

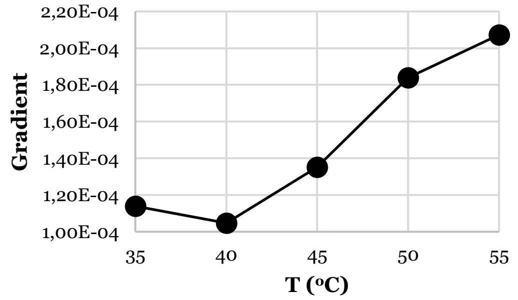

Figure 5. Relationship between Gradient R and Temperature at Fixed Airflow Rate of 1.9 ms/s.

Estimating of Optimum Airflow

To determine the optimum airflow rate, we need to assess the gradient value at a specific airflow rate, as depicted in Figure 6. The gradient was calculated the same way as in the previous chapter.

When considering different airflow rates, certain observations can be made. At an airflow rate of 1.6 m/s, a substantial change in the gradient is observed. However, at 1.9 m/s, the gradient shows a slight decrease. The highest gradient occurs at an airflow rate of 2.2 m/s, although it slightly decreases again at 2.4 m/s. Analyzing these findings, unlike the way we set the optimum temperature we set the optimum airflow at 1.9 m/s since it gave a gradient value near the mid value of within the gradient range.

Effect of Drying on Flavonoid Content

Even though we have already determined the optimum condition, we still need to find what are the yield and composition of flavonoids at that condition. After maceration using ethanol 96%, of each extract was obtained, and the yield was calculated and measured by UV-Vis as described earlier in the previous subchapter.

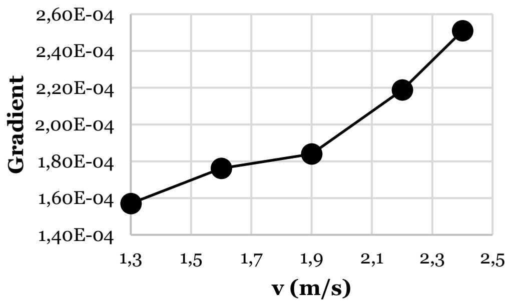

Figure 6: Relationship between Gradient of R and Airflow Rate at Optimum Temperature of 50°C.

Table 5. Effect of Drying Process on Yield Extract

No	Sample	T (°C)	F (m/s)	Yield (%)
1	Fresh leaves	-	-	20.32
2	Dried leaves	45	1.3	8.54
3	Dried leaves	45	2.4	4.67
4	Dried leaves	45	1.9	6.18
5	Dried leaves	50	1.9	7.83
6	Dried leaves	55	1.9	3.99

Table 5 shows the yield value for fresh leaves was 20.32%. While the highest extract yield obtained at the temperature and drying air flow rate of 45°C and 1.3 m/s was 8.54% and the lowest extract yield obtained at the temperature and drying air flow rate of 55°C and 1.9 m/s was equal to 3.99%. The increase of extract yield value occurred in samples with drying temperatures of 45°C to 50°C. In accordance the study by Taufiq [21] reported that the yield increase in Cornflowers by heat treatment is due to the cell walls of the sample being exposed and important compounds from the sample easily exiting during extraction.

Table 6. Effect of Drying Process on Flavonoid Content

No	Sample	T (°C)	F (m/s)	Flavonoid Content (g QE/100 g extract)
1	Fresh leaves	-	-	0.16 ± 0.00003
2	Dried leaves	45	1.3	0.19 ± 0.00010
3	Dried leaves	45	2.4	0.26 ± 0.00030
4	Dried leaves	45	1.9	0.52 ± 0.00100
5	Dried leaves	50	1.9	0.78 ± 0.00100
6	Dried leaves	55	1.9	0.39 ± 0.00010

From **Table 6**, it can be seen that the flavonoid content on fresh leaves of Fiscus carica is 0.16±0.00003 grQE/100 g extract. The theory states that the drying process affects the secondary metabolic content found in plants. At the drying air flow rate of 1.9 m/s, the highest value is 0.78±0.001 grQE/100gr extract. Similar to the value of yield extract, flavonoid content in the extract increases with increasing drying temperatures due to the opening of the cell wall and leaf pores. From **Table 6**, flavonoid content increase until the air temperature of 50°C, but decreases at a temperature of 55°C. This, according to [22], reports that at 50°C it is a relatively safe boundary that will not damage/degrade flavonoids. Research [23] reported that at certain heating temperatures flavonoids will undergo an oxidation reaction that can break the conjugated carbon double bonds that cause unreadable flavonoid compounds absorbance at predetermined wavelengths, resulting in a decrease in total flavonoid content of ethanol extract of cat whiskers leaves.

Characteristic of Ficus carica leaves herbal tea

In this study, dried leaves of *Fiscus carica* produced from the drying process at a temperature of 50 °C with a drying air rate of 1.9 m/s were packaged in tea bags (10 cm x 12.5 cm). Each pack of tea bags contains 2 grams of dried Fiscus carica leaves. **Table 7** shows the results of the analysis of protein, fat, carbohydrates, calories, and the number of antioxidants in dried *Fiscus carica* leaves.

It can be seen that dried tin leaves have a high antioxidant value (IC50), which shows a weak antioxidant power. Specifically, a compound called very strong antioxidant if the IC50 value is less than 50 ppm, strong for IC50 is worth 50-100 ppm, moderate if IC50 is worth 100-150 ppm, and weak if IC50 is worth 151-200 ppm [19]. This is because the IC50 value is a number that shows the concentration of the test sample (μg/ml) which gives a DPPH reduction of 50% (capable of reducing the DPPH oxidation process by 50%).

Table 7. Characteristic of Fiscus carica leaves herbal tea

No	Characteristic	Unit	Result
1	Total fat	% b/b	3.50
2	Protein	% b/b	5.35
3	Carbohydrates	% b/b	23.60
4	Calories	Kkal/100g	147.30
5	Antioxidants (IC50)	ppm	154.25

CONCLUSION

The optimum drying condition of tin leaves (*Ficus carica L.)* using a tray dryer to produce *Fiscus carica* leaves herbal tea products was at 50 °C and an airflow rate of 1.9 m/s. The obtained flavonoid content at that condition was 0.78 ± 0.001 gramQE/100-gram extract. Dried products packaged in tea bags contain 3.50% total fat, 5.35% protein, 23.60% carbohydrate, 1430 Kcal/100gram calories, and 154.25 ppm antioxidant (IC50).

For future research, we need to assess drying conditions by a fair temperature and airflow rate combination. Therefore we can get precisely the optimum conditions.

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