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Original Research Article

Effect of Different Drying Methods on Physical Properties of *Azolla pinnata* Powder

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Abstract: *Azolla pinnata* is a small floating plant that is rich in protein and other nutrients and is suitable for livestock feed. Drying is one of the traditional techniques to preserve food to stay longer in shelf life, besides it is the easiest way and can help in maintaining the properties of the plant or food. There is a lack of information on the physical properties of *Azolla pinnata* in powder condition. In this study, four different combinations of two drying methods using shade drying as a control variable and oven drying at different temperatures at 40°C, 50°C, and 60°C as the test variable on grinded and non-grinded Azolla were performed. The objective of the study was to determine the effects of different drying methods on the physical properties of Azolla powder containing either grinded or non-grinded Azolla. The results obtained of tapped density, bulk density, Carr's Index (CI), and Hausner ratio values were recorded in Table 1. Based on Table 1, the good value of powder flowability was determined by the values of CI and Hausner ratio. According to results obtained, oven drying at 50°C provided the most suitable drying method to be used because it provides good physical characteristics, especially for grinded Azolla powder.

Keywords: drying methods, *Azolla pinnata*, physical properties, powder

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1. Introduction

Azolla is a free-floating aquatic plant belonging to the *Azollaceae* family and it is also known as the Mosquito fern. *Azolla pinnata* is a widely distributed species in both tropical and temperate. It can be quickly propagated but requires abundant standing water, 85–90% relative humidity, 4.5–6.5 in pH, 90–150 mg/land salinity, and sufficient phosphorus for its nutritional needs (Mohamed *et al.*, 2018). Azolla is a promising biofertiliser for crops. The leaf cavities of *Azolla* contain symbiotic cyanobacterium also known as *Anabaena azollae* that provides about 30–60 kg N/ ha making *Azolla* an important biological source of N for agriculture and animal production (Maswada *et al.,* 2020). Currently, Azolla can be used for multiple purposes such as animal feed, biofertiliser, human food, a form of water purifier, green manure, and reduced ammonia volatilisation after chemical nitrogen application. It helps in improving the water quality by removing an excess quantity of nitrates and phosphorus (Costa *et al.*, 2010; Maswada *et al.*, 2020; Mohamed *et al.*, 2018; Roy *et al.*, 2016;). The weight of harvested fresh Azolla can reach 15–20 t/ha in about 20 days (Shaltout *et al.,* 2012).

Drying is a method that reduces the amount of free water in food, allowing for a higher concentration of dry matter while preserving the tissue, wholesomeness and physical properties of the food. Shade drying and conventional (oven) drying are the most common drying methods. Conventional (oven) drying is more user-friendly compared to shade drying which might be affected by the environmental condition. According to Alara *et al*. (2019), the drying methods used are shade drying as a control variable and the conventional (oven) drying. The different temperatures which are at 40^{o}C, 50^{o}C, and 60^{o}C are applied in this study.

Shade drying uses ambient air from the free UV drying process. Shade drying is generally recommended for cloudy, low-humidity, and heavily polluted environments. According to Chen and Mujumdar, (2006), a similar drying pattern is used for drying herbs. Besides, Eric (2013) also used this method for drying basil leaves.

Oven drying is the best way to dry food since virtually no special equipment is required. It is faster than the open sun and the shade drying. De Santana *et al.,* (2014) stated that the forced ventilation oven at 50^{o}C is best recommended for drying *Ocimum gratissium* leaves without significantly varying the content and composition of the essential oil. Another research indicated that drying in an oven at 60^{o}C causes reduced nutrient loss and is used efficiently to dry curry, mint, and coriander leaves (Pratap, 2015). Finding from Mohamed *et al.,* (2018) found that an oven at 50^{o}C preserved the maximum content of vitamins especially water-soluble vitamins in Moringa leaves.

In this study, the physical properties that will be evaluated are tapped density, bulk density, Carr's Index (CI), and Hausner ratio. The tapped density is the improved bulk density achieved by mechanical tapping a container carrying the sample of the powder.

The bulk density of the powder was determined to be the ratio of the mass of the sample to its volume. Reporting by Petrucceli *et al.,* (2015) values of bulk density were 0.31

g/cm^3 (control substrate, GMC), 0.27 g/cm^3 (GM1), 0.21 g/cm^3 (GM2), and 1.5 g/cm^3 (GM3) which linearly decreased with Azolla content which is mixed with other substrates.

The CI is a calculation of the flowability of powder materials dependent on bulk and tapped density values. Pre-treatment or bleaching increased the flowability of oven-dried powder by decreasing the CI in comparison to the control powder (Table 1), which was consistent with the findings of (Singh & Prasad, 2013). Singh and Prasad (2013) suggested that a compact system with less frictional force would contribute to a better flowability of samples.

The Hausner ratio is a measure of the tapped density to the bulk density of the powders. Where if the Hausner ratio is greater than 1.5, the powder is viscous with poor flowability and filling capability. When the ratio is less than 1.2, this means that the powder has good flowability and filling capabilities (Ali *et al.*, 2017; Chenguang & Jianguo, 2013). The relationship between CI, Hausner ratio, and powder flowability is shown in the following Table 1.

Table 1. Relationship between CI, Hausner ratio, and Flowability

Flowability	CI	Hausner Ratio
Excellent	1~10%	1.00~ 1.11
Good	11%~ 15%	1.12~ 1.18
Average	16%~ 20%	1.19~ 1.25
Eligible	21%~25%	1.26~1.34
Poor	26%~ 31%	1.35~1.45
Terrible	32%~37%	1.46~1.59
Can hardly flow	>38%	>1.6

Source: Lebrun, (2012); Saifullah M, Yusof Y, Chin N *et al*., (2016).

Azolla has been reported to be a very good source of protein, essential mineral elements, and vitamins. According to previous findings, there are many studies on the determination of chemical properties of *Azolla pinnata* such as mineral elements (pH value, N, P, K, Mg, Mn, Zn, Ca), vitamins, proximate analysis (crude protein, crude fibre, total ash) were recorded (Bhatt *et al*., 2020; Cherryl *et al*., 2014; Najar & Khan, 2010). However, there is a lack of knowledge about the study of the physical properties of *Azolla pinnata* specifically in powder form. The physical properties of *Azolla pinnata* are important besides the chemical properties contained because they can help for further research or in producing new products by using Azolla as a main element.

Therefore, this research was carried out to study the effect of different drying methods which are shade drying and oven drying at different temperatures; 40°C, 50°C, and 60°C on physical properties such as tapped density, bulk density, CIvalue and Hausner ratio of *Azolla pinnata* powder.

2. Materials and Methods

2.1 Sample Collection

The fresh *Azolla* pinnata is cultivated by using Vertical Multilayer Shelves (VERSMAP) at Food Processing Machine Design Laboratory, Faculty of Engineering, Universiti Putra Malaysia. The matured fresh Azolla will be harvested on day 10 in the morning. The harvested fresh Azolla will be drained out of the water by using a basket within 30 minutes for further analysis and process.

2.2 Drying Azolla pinnata

Four different drying treatments were used to dry *Azolla* leaves including shade drying and oven drying at 40°C, 50°C, and 60°C. The different temperatures of oven drying are chosen because to prevent the huge changes in the physical properties of the sample, a temperature below 60°C is most suitable. Thus, oven drying is one of the economical and controlled methods of drying, however, the higher temperature can affect the heat-labile nutrients (Ali *et al.*, 2017).

2.2.1 Shade drying

Azolla leaves were spread evenly in a thin layer on the black netting provided under the roof of VERSMAP for 24 hours. The leaves were turned over after every 1 hour for uniform drying.

2.2.2 Oven drying

Azolla leaves were spread uniformly on the tray in the oven (UNB500, Memmert, Germany). The leaves were dried at 40°C, 50°C, and 60°C temperature (Ali *et al*., 2017) in an oven. The drying process was continued for 24 hours. After drying, samples were cooled under ambient laboratory conditions and stored in desiccators for further analysis.

2.3 Grinding and Sieving Processes

After the drying process, the leaves will be ground with a domestic blender (EBM-B1235 (IV), ELBA, Italy) and sieved through a 500µm by using a sieve shaker (MINOR, Endercotts Limited, England).

2.4 Determination of Tapped Density

Tapped density is the maximum packing density of powder that is achieved under the influence of an externally applied force. It is the ratio of the mass of the powder to the volume of the powder bed after tapping. A tapped density analyser (GeoPac 1360 T.A.P Density analyser, Micrometrics Instrument Corporation, Norcross, GA) was used to determine the tapped density of powders. A pre-weighed sample was poured into the graduated glass chamber then the plunger was inserted and mounted on the analyser. According to Ali *et al.*, (2017), to start the analysis, data was entered such as sample ID, no. of cycles (5), and applied force using the analyser keyboard. During analysis, the analyser agitates the sample chamber. While the chamber is being agitated, the analyser drives the plunger into the chamber until the desired consolidation (compression) force (50 N) is achieved. At the end of the test weight of the sample was entered and the analyser's LCD screen displayed the results of density in g/cm^3 and standard deviation.

2.5 Determination of Bulk Density

Bulk density is an important parameter when the powder is freely poured into a hopper. It is a ratio between the mass and volume of a freely poured powder in a container. 100 mL measuring cylinder filled up with water and sample to determine the volume. The mass and volume of the sample were used to calculate the bulk density using Equation 1.

$$\text{Bulk density} = \frac{\boldsymbol{mass}}{\boldsymbol{volume}} \tag{1}$$

2.6 Determination of CI

The CIis extrapolated from the bulk and tapped densities using Equation 2.

$$\text{CI (\%)} = \frac{(\boldsymbol{Tapped\ density - Bulk\ density})}{\boldsymbol{Tapped\ density}} \text{ X } 100\% \tag{2}$$

2.7 Determination of Hausner Ratio

The Hausner ratio is also extrapolated from the bulk and tapped densities using Equation 3.

$$\text{Hausner Ratio} = \frac{(\boldsymbol{Tapped\ density})}{(\boldsymbol{Bulk\ \ density})} \tag{3}$$

2.7 Statistical Analysis

Data were analysed statistically using the software Minitab 16. Each analysis was done by having triplicate data, and the differences between means were assessed by Tukey's

Test, $p < 0.01$ was considered statistically significant. They were analysed by one-way ANOVA.

3. Results and Discussions

The physical properties which were analysed in this study were tapped density, bulk density, CI, and Hausner ratio *Azolla pinnata* powder due to different drying methods presented in Table 2. The results for all properties were divided into two types of categories for grind *Azolla* and not grind *Azolla*. Size limitation for grind *Azolla* was carried out by passing through 500 µm of sieving. The sample for *Azolla* passing through > 500 µm was considered as not grind. The values showed for both densities, CI, and Hausner ratio indicated the characteristic of the flowability powder.

3.1 Tapped Density

Based on Table 2, shade drying was the control variable and the value for tapped density obtained was 0.26±0.01 g/cm^3. The different drying methods used with different temperatures for oven drying on grind Azolla showed that the highest value of tapped density was obtained at 40^{o}C of oven drying which were 0.45±0.01 g/cm^3. However, the lowest values of tapped density were 0.25±0.01 g/cm^3 for both 50^{o}C and 60^{o}C of oven drying. According to Fazaeli *et al.* (2012), the higher inlet air temperatures, in general, result in lower density powders, which are due to the higher drying temperature, which in turn causes faster particle drying with less droplet shrinkage, resulting in a decreased tapped density.

For the not grind Azolla, shade drying as the control variable obtained a value of 0.21±0.01 g/cm^3 for tapped density. With the aforementioned methods carried out for not grind Azolla, the trend of the tapped density value was found to be similar to grind Azolla. According to the results recorded in Table 2, the value of tapped density at 40^{o}C oven drying gained the highest value of 0.36 ± 0.01 g/cm^3 followed by 50^{o}C, 0.21 ± 0.01 g/cm^3. Meanwhile, the lowest value of tapped density was 0.05±0.01 g/cm^3 at 60^{o}C of oven drying.

Therefore, the tapped density of Azolla powder indicated a significant reduction ($p < 0.01$) which was affected by drying methods. The tapped density values for the shade drying were found to be 0.26±0.01 g/cm^3 and 0.21±0.01 g/cm^3 for the grind and not grind Azolla, respectively. For the oven drying at 40^{o}C, the value of tapped density for not grind Azolla (0.36±0.01 g/cm^3) was lower than the grind Azolla (0.45±0.01 g/cm^3). At 50^{o}C of oven drying, not grind Azolla had the lowest tapped density with the value of 0.21±0.01 g/cm^3 when compared to grind Azolla (0.25±0.01 g/cm^3). When comparing between not grind Azolla (0.05±0.01 g/cm^3) and grind Azolla (0.25±0.01 g/cm^3), not grind Azolla had the lowest value of tapped density at 60^{o}C of oven drying.

3.2 Bulk Density

According to Table 2, shade drying (control variable) which obtained value for bulk density was 0.21±0.01 g/cm^3. At different drying methods used with different temperatures for oven drying on grind Azolla, the highest value of bulk density was obtained at 40°C of oven drying which were 0.41±0.00 g/cm^3. However, the lowest values of bulk density were 0.22±0.00 g/cm^3 for both 50°C and 60°C of oven drying. In a previous study reported by Raja *et al.* (2019), for blanched samples, there is a reduction of bulk density due to an increase in the temperature of oven-dried powder of papaya leaves.

Based on the results obtained for not grind Azolla, shade drying (control variable) produced a value for bulk density of 0.21±0.01 g/cm$^{3.}$ Using the same methods as applied to grind Azolla for not grind Azolla, the trend of the bulk density value between not grind and grind Azolla was relatable. Based on Table 2, the value of bulk density at 40°C oven drying gained the highest value of 0.36±0.01 g/cm^3 followed by 50°C with a value of 0.21±0.01 g/cm^3. However, the lowest value of tapped density was 0.05±0.01 g/cm^3 at 60°C of oven drying.

Therefore, the bulk density of Azolla powder showed a significant reduction (p < 0.01) which was affected by drying methods. The bulk density values for the grind (0.21±0.01 g/cm^3) and not grind Azolla (0.21±0.01 g/cm^3) on shade drying were the same. For the oven drying at 40°C, the value of bulk density for not grind Azolla (0.36±0.01 g/cm^3) was lower than grind Azolla (0.41±0.00 g/cm^3). At 50°C of oven drying, not grind Azolla had the lowest bulk density with a value of 0.21±0.01 g/cm^3 when compared to grind Azolla (0.22±0.00 g/cm^3). When comparing not grind Azolla (0.05±0.01 g/cm^3) to grind Azolla (0.22±0.01 g/cm^3), not grind Azolla had the lowest value of bulk density at 60°C of oven drying.

Likewise, the findings of Raja *et al.* (2019) showed a reduction trend for bulk density value that was similar to grind and not grind Azolla performed in this study which was due to an increment of oven drying temperature.

Table 2. Physical properties of *Azolla pinnata* powder.

Drying Method Types	**Tapped density (g/cm^3)**		**Bulk Density (g/cm^3)**		**CI**		**Hausner Ratio**	
	Grind Azolla	**Not Grind Azolla**	**Grind Azolla**	**Not Grind Azolla**	**Grind Azolla**	**Not Grind Azolla**	**Grind Azolla**	**Not Grind Azolla**
Shade Drying (Control)	0.26 ± 0.01[b]	0.21 ±0.01[b]	0.21 ± 0.00[d]	0.21 ± 0.01[b]	0.20 ± 0.00[a]	0.21 ± 0.01[b]	1.24 ±0.00[a]	1.36 ± 0.00[b]
Oven Drying at 40°C	0.45 ± 0.01[a]	0.36 ± 0.01[a]	0.41 ± 0.00[a]	0.36 ± 0.01[a]	0.07 ± 0.00[d]	0.36 ± 0.01[a]	1.08 ± 0.00[c]	1.08 ± 0.00[d]
Oven Drying at 50°C	0.25 ±0.01[c]	0.21 ± 0.01[c]	0.22 ± 0.00[c]	0.21 ± 0.01[c]	0.12 ± 0.00[b]	0.21 ± 0.01[c]	1.30 ± 0.01[b]	1.17 ± 0.00[c]

Drying Method Types	Tapped density (g/cm^3)		Bulk Density (g/cm^3)		CI		Hausner Ratio	
	Grind Azolla	Not Grind Azolla	Grind Azolla	Not Grind Azolla	Grind Azolla	Not Grind Azolla	Grind Azolla	Not Grind Azolla
Oven Drying at 60°C	0.25 ± 0.01^{d}	0.05 ± 0.01^{d}	0.22 ± 0.00^{b}	0.05 ± 0.01^{d}	0.09 ± 0.00^{c}	0.05 ± 0.01^{d}	0.06 ± 0.01^{c}	1.65 ± 0.01^{a}

Data are expressed as mean ± SD. Different letters indicate statically significant differences exist $p < 0.01$ for each column. This means that do not share a letter represents significantly different. Tukey's test was applied with 95% simultaneous confidence intervals**.**

3.3 Carr's Index

Based on Table 2, shade drying was the control variable that was carried out to obtain a value of 0.20±0.00 for CI. A different drying method used with different temperatures for oven drying on grind Azolla showed that the lowest value of CI was obtained at 40°C of oven drying which was 0.07 ±0.00. On the other hand, the highest value of CI was 0.12±0.00 at 50°C. At 60°C of oven drying, the value of Carr's Index was 0.09±0.00.

For not grind Azolla, shade drying was the control variable which obtained a value of 0.21±0.01for CI. Even though the same methods were used for grind Azolla was applied to not grind Azolla, the trend of CI value for not grind Azolla was not similar to grind Azolla. According to the results recorded in Table 2, the value of CI at 40°C oven drying gained the highest value of 0.36±0.01 followed by 50°C with a value of 0.21±0.01. Meanwhile, the lowest value of CI was 0.05±0.01 at 60°C of oven drying.

Therefore, the CI of Azolla powder indicated a significant reduction ($p < 0.01$) which was affected by drying methods. The CI values for the shade drying were found to be 0.20±0.00 and 0.2±0.01for the grind and not grind Azolla, respectively. For the oven drying at 40°C, the value of CI for the grind Azolla (0.07±0.00) was lower than the not grind Azolla (0.3 ±0.01). Meanwhile, at 50°C of oven drying, the grind Azolla had the lowest CI with a value of 0.12±0.00 when compared to not grind Azolla (0.21 ±0.01). Comparison of not grind Azolla (0.05±0.01) and grind Azolla (0.09±0.00) demonstrated that not grind Azolla had the lowest value of CI at 60°C of oven drying.

3.4 Hausner Ratio

According to Table 2, shade drying (control variable) on grind Azolla produced a value of 1.24 ± 0.00 for the Hausner ratio. By applying oven drying with different temperatures on grind Azolla, the value of the Hausner ratio obtained at 40°C of oven drying was 1.08±0.00. The highest value of CI was 1.30±0.01 at 50°C of oven drying while the lowest value of CI was 0.06±0.01 at 60°C of oven drying.

For not grind Azolla, shade drying (control variable) on not grind Azolla obtained a value of 1.36±0.00 for Hausner ratio. By applying the same method of oven drying used on grind Azolla, the trend of the Hausner ratio values for not grind Azolla was relatable to grind Azolla. Based on Table 2, the value of the Hausner ratio at 40°C oven drying had the lowest value of 1.08±0.00. At 50°C oven drying, the value of the Hausner ratio was 1.17±0.00 while the highest value of the Hausner ratio was 1.65±0.01 at 60°C of oven drying.

Therefore, the Hausner ratio of Azolla powder showed a significant (p<0.01) fluctuation for the grind Azolla and increment for not grind Azolla which is affected by drying methods. The Hausner ratio values were 1.24±0.00 for grind and 1.36±0.00 for not grind Azolla on shade drying. For the oven drying at 40°C, the value of the Hausner ratio for the grind and not grind Azolla was the same with a value of 1.08±0.00. At 50°C of oven drying, not grind Azolla had the lowest Hausner ratio with a value of 1.17±0.00 when compared to the grind Azolla (1.30±0.01). Comparison between grind Azolla (0.06± 0.01) and not grind Azolla (1.65±0.01) demonstrated that grind Azolla had the lowest value of Hausner ratio at 60°C of oven drying

4. Conclusions

Based on the findings of this study, the effects of different drying methods on the physical properties of *Azolla pinnata* powder were evaluated. This study shows that the physical properties of Azolla powder such as tapped density, bulk density, CI, and Hausner ratio were affected by different drying methods. For grind Azolla, the shade drying for tapped density (0.26±0.01 g/cm^3), bulk density (0.21±0.01 g/cm^3), CI (0.20±0.00), and Hausner ratio (1.24±0.00) were determined. At 40°C of oven drying, grind Azolla had the values of 0.45±0.01 g/cm^3, 0.41±0.00 g/cm^3, 0.07±0.00, and 1.08±0.00 for tapped density, bulk density, CI and Hausner ratio, respectively. While, the values of tapped density, bulk density, CI and Hausner ratio for grind Azolla were 0.25±0.01 g/cm^3, 0.22±0.00 g/cm^3, 0.12±0.00 and 1.30±0.01, respectively at 50°C of oven drying. At 60°C of oven drying, grind Azolla had the values of 0.25±0.01 g/cm^3, 0.22±0.00 g/cm^3, 0.09±0.00, and 0.06±0.01 for tapped density, bulk density, CI and Hausner ratio, respectively. For not grind Azolla, the shade drying values were the same for both tapped density and bulk density of 0.21±0.01 g/cm^{3}while the CI value was 0.21±0.01 and the Hausner ratio was 1.36±0.00. At 40°C of oven drying, not grind Azolla had the same values of 0.36±0.01 g/cm^3 for tapped density and bulk density. The not grind values were 0.36±0.01 and 1.08±0.01 of CI and Hausner ratio, respectively. Meanwhile, the values of tapped density, bulk density, CI, and Hausner ratio for the not grind Azolla were 0.25±0.01 g/cm^3, 0.22±0.00 g/cm^3, 0.21±0.00, and 1.17±0.01, respectively at 50°C of oven drying. At 60°C of oven drying the not grind Azolla had the values of 0.05±0.01 g/cm^3 for tapped density and bulk density, while the value of CIwas 0.5±0.01 and the Hausner ratio was 1.65±0.01 were determined.

Author Contributions: N.A.A.A. conducted the experiments, collected and analysed the data of results, and wrote the manuscript. R.S. supervised the research and revised the manuscript. S.K. and M.S.A. supervised the experiments. All authors have read and agreed to the published version of the manuscript.

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