



Physicochemical Properties of *Morinda Citrifolia* Fruits and Leaves Produced by Oven and Freeze Drying

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ABSTRACT

Morinda citrifolia, also known as Noni, is a nutritional plant that contains beneficial characteristics and has been explored in both the pharmaceutical and food industrial fields. This study was carried out to compare the physicochemical properties of Noni fruits and leaves powder produced by freeze-drying and oven drying methods. Oven drying was carried out at different temperatures (50 °C, 60 °C, and 70 °C) while the freeze-drying method was carried out at constant temperature (-50 °C) and time (40 hours). The physicochemical properties (antioxidant, moisture content, water activity, density, water solubility, and color) of the fruits and leaves powder were analyzed. Freeze-drying produced the lowest moisture content and water activity of Noni fruit and leaf powder. The study revealed that increased in oven drying temperature decreased the color properties (L^* , a^* , b^*), moisture content and water solubility of both Noni fruit and leaves powder. Bulk and tapped density showed slightly changes with an increase in drying temperature. Oven dried Noni fruit and leaf powder at 50 °C had the highest percentage of DPPH radical scavenging activity. Besides, freeze-drying proved to have better physicochemical properties in Noni fruits and leaves. The results obtained indicate a significant difference between the physicochemical properties of freeze dried Noni fruits and leaves powder in terms of water activity, color, and DPPH radical scavenging activity.

Keywords: Freeze drying, Oven drying, antioxidant, *Morinda citrifolia* L., powder

INTRODUCTION

Morinda citrifolia L. is an evergreen shrub or small tree that is cultivated in tropical and subtropical countries including Vietnam, Malaysia, India and China (Jahurul et al., 2021). It is known as Noni or Indian mulberry in India, mengkudu in Malaysia, and cheese fruits in Australia (Almeida et al., 2019). Noni fruit is soft and fleshy and immature when light green and ripe when yellowish-white in colour. Noni fruit contains up to 260 seeds which are discarded as waste during the production of Noni juice, powder, and puree. The fruits and leaves, in

particular, have been utilized for food and medicinal due to the presence of different bioactive components (Wang et al., 2023).

Noni fruit possesses about 200 bioactive phytochemicals, including polyphenols, flavonoids, iridoids, coumarins, anthraquinones, lignans, terpenoids, alkaloids, and glycosides (Fontes et al., 2023). Many studies have also reported that Noni fruits have many functions such as antibacterial, antifungal and antihelmintic (Abou Assi et al., 2017; Jongjai et al., 2021), antioxidant (Fontes et al., 2023) and anti-inflammatory (Prasad et al., 2019). The potential use of *M. citrifolia* fruit as a nutritional supplement attracted the interest of the food industry, and the pharmaceutical sector incorporated it in a variety of products. Various food products, including ice cream, sweets, cereal, nutritious drinks, food supplements, sauces, and condiments, have used Noni fruit juice and puree as unique food ingredients (Almeida et al., 2019).

However, Noni fruit and leaf are perishable plants, and the drying process encourages a reduction in moisture content, minimising water activity, and preventing deterioration caused by microorganisms (Mahayothee et al., 2020). Although drying could extend product shelf life, it also had negative effects on product quality, such as overall appearance and bioactive constituent degradation, resulting in low commercial acceptance (Nguyen, 2020). Many drying processes such as freeze drying, oven drying and spray drying are currently used in the food industry to produce powders (Kha et al., 2021; Krishnaiah et al., 2012). The variations in drying methods and temperatures can significantly impact the nutritional value and overall quality of the food powders. Additionally, understanding these differences can help in selecting the most suitable drying method to preserve desired characteristics and enhance consumer acceptance. In the food drying process, drying method and drying temperature are key factors for preservation of the product's quality.

In this study, we aimed to investigate the influence of drying temperature and drying methods i.e. oven drying and freeze drying on physicochemical properties of Noni fruit and leaf powder. In addition, the optimum drying conditions were also determined and nutritional content of Noni fruit and leaves were compared.

MATERIALS AND METHODS

Materials and chemicals

The fresh *M. citrifolia* fruits and leaves were collected from Taman Herba, Faculty of Bioresources and Food Industry, Besut Campus, Terengganu. The chemicals used were 0.2% sodium metabisulphite, 2,2-diphenyl-1-picrylhydrazyl (DPPH), ethanol, acetone and methanol solution.

Preparation of *M. citrifolia* powder

Noni fruits and leaves were washed using tap water to remove any dirt, dust, or any other foreign matter. The leaves were chopped into smaller pieces. The skin fruits were peeled off from the fruits. Then, the flesh was cut into uniform size (5 mm) with the seeds. To prevent the browning reaction during drying, the flesh was soaked in 0.2% sodium metabisulphite for 15 minutes to inhibit enzyme activity. The small cut of fruits and leaves were dried using two methods which were freeze-drying and oven-drying.

Freeze drying of Noni fruits and leaves were carried out according to Jiang et al. (2019) with some modifications. Both fruits and leaves were frozen by convectional freezing (Haier Deep Freezer, DW-40L508, China) at the temperature of -18 ± 1 °C and proceed to dehydrate in a freezer dryer (Freeze Dryer Christ, Alpha 1-4 LDplus, Germany) at -50 °C for 40 hours. For oven drying method, the small cut of Noni fruits and leaves were spread on aluminium petri dishes and dried in the Memmert Universal Oven (Mettler, Germany) at different drying temperatures; 50 °C, 60 °C and 70 °C, for 8 hours.

After both drying processes, dried fruits and leaves were ground using a stainless steel grinder (Electromagnetic Sieve Shaker) and sieved through a 250 µm to 500 µm mesh screen to obtain a uniform particle size range. Then, the powder was kept at room temperature in an airtight plastic for further analysis.

Determination of drying curve

Drying curve was determined from the mass loss in the sample using method according to Rosidi et al. (2021). During the drying process, the weight of sample was recorded for every two hours until constant weight. Moisture content in the sample at each time intervals was calculated using Eqn. 1:

$$MC_{db}(\%db) = \frac{W_o - W_f}{W_f} \times 100 \quad \text{Eqn. 1}$$

Where;

W_o = initial weight of sample at each interval time (g)

W_f = final weight of sample (g)

Moisture content of dried powder was determined by oven drying at 105°C (AOAC 1995).

Determination of water activity (a_w)

Water activity of the powder was measured using water activity meter (Aqua Lab, Malaysia). The powder was spread evenly in the Retronic cup before placed in the water activity meter. Triplicate samples were analyzed and the means were reported.

Colour analysis

The colour changes of Noni fruit and leaves powder were analyzed using chroma meter (Minolta Chroma Meter CR-400, Osaka, Japan). The colour was expressed in term of L^* , a^* and b^* . The a^* axis displays the variation from red to green, the b^* axis displays the variation from yellow to blue, and the L^* parameter indicates the colour variation from black to white. Before the measurement, the chroma metre was calibrated with a white reference tile. Hunter values for each powder was measured in triplicate.

Determination of water solubility

Water solubility and water absorption were determined according to method Kha et al. (2021), with slight modifications. Firstly, 0.4 gram of the sample dispersed in 40 ml of distilled water and poured into the centrifuge tubes. Afterwards, the centrifuges tubes were placed in a water bath at 80 °C for 30 minutes, followed by chilled (4 °C) centrifugation for 30 mins at 8000 rpm. The supernatant was collected in pre-weighed aluminium petri dishes. Then, the supernatant was weighed after drying in the oven at 105 °C overnight. The percentage of water solubility of fruits and leaves powder were calculated based on the formula in Eqn. 2.

$$\text{Water solubility} = \frac{\text{Weight of supernatant}}{\text{Weight of sample powder}} \times 100 \quad \text{Eqn. 2}$$

Measurement was measured triplicates and the average of the data was calculated.

Determination of bulk and tapped densities

Bulk density (g/mL) was measured in a graduated cylinder by gently adding twenty grams of Noni fruits and leaves powder into an empty 250 mL graduated cylinder and the volume was recorded. The ratio of the mass

of the powder to the volume occupied in the cylinder determined the bulk density value in g/ml using the Eqn. 3.

$$\text{Bulk density, } \rho_{\beta} \text{ (g/cm}^3\text{)} = \frac{\text{mass of Noni fruits /leaves powder}}{\text{volume occupied by the powder}} \quad \text{Eqn. 3}$$

The tapped density was measured by tapping the powder in the graduated cylinder for 100 times. The tapped density was calculated by the Eqn. 4.

$$\text{Tapped density, } \rho_t \text{ (g/cm}^3\text{)} = \frac{\text{mass of Noni fruits /leaves powder}}{\text{volume occupied by the powder}} \quad \text{Eqn. 4}$$

Determination of antioxidant activity

The antioxidant activity was determined according to Nascimento et al. (2018) using 2,2 diphenyl-picrilhidrazyl (DPPH) assay with some modifications. For extraction procedures, 1 g of Noni fruit and leaves powder were macerated and mix with 25 mL of solvent (2 acetone:2 ethanol:1 water, v/v/v) and centrifuged at 8000 rpm for 20 minutes at 5 °C. After that, it was filtered using filter paper. The filtration residue then was re-extracted in 15 ml of the same solvent. Then, the extraction was kept for further analysis to determine their antioxidant capacity.

For the DPPH assay, 200 μ L (using micropipette) of the stock extraction was diluted and mixed with 2.85 ml of 0.06 mM DPPH. Then, the mixture was agitated in a vortex for 30 seconds and left in dark for 1 hour, and analyzed at 517 nm using a spectrophotometer (Shimadzu, Europa). Methanol solution was used as a blank and the results were determined and expressed in free radical scavenging (%) according to Eqn. 5.

$$\% \text{ Free Radical Scavenging} = \frac{Abs_B - Abs_A}{Abs_B} \times 100 \quad \text{Eqn. 5}$$

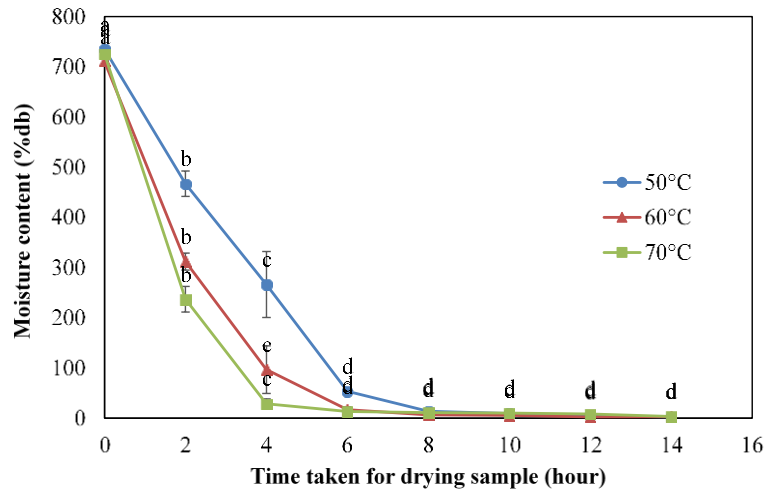
Statistical analysis

The results were presented as mean values with standard deviations. Different mean values were analyzed by analysis of variance (ANOVA), least significant different (LSD) and Tukey's test at significant level of 95% ($\alpha=0.05$) using statistical software SPSS 20 (SPSS Inc., USA).

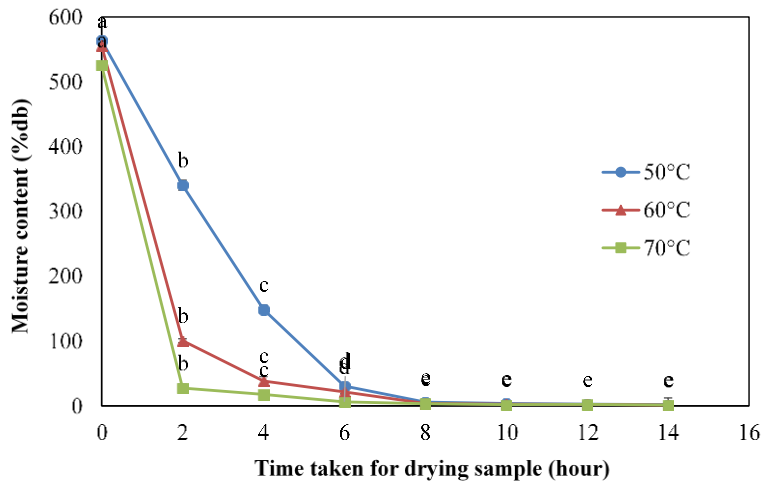
RESULTS AND DISCUSSION

Drying curve of Noni fruits and leaves

Drying curves of Noni fruits and leaves that undergoes oven drying at 50 °C, 60 °C and 70 °C are shown in Fig. 1. Both Noni fruits and leaves show the same drying trend where the moisture contents decrease with the time. The decreasing trend was continuous until the moisture content of both samples reached the equilibrium values. In this drying process, two phases of drying curve were shown; falling rate and diffusion phase. Falling rate of dried Noni fruits and leaves occur from initial time of drying to 8 hours while diffusion phase occurred from 8 hours to 14 hours. This finding was consistent with previous findings by (Mireles-Arriaga et al., 2016) for Noni slices and (Rosidi et al., 2021) for dragon fruit peels.



(a)



(b)

Fig. 1. Moisture profile of (a) Noni fruits and (b) Noni leaves during drying at 50 °C, 60 °C and 70 °C.

The falling rate of drying curve occurs when the migration moisture from inner of each particle in the sample to the outer surface. In this case, the moisture content eventually began to decrease and start to constant after 8 hours of drying time. The wet regions on the surface continue to plummet until the surface is completely dry during the first falling drying rate. After that, the second falling rate will conquer where the evaporation plane fades away from the surface. In comparison to the constant rate and initial falling rate periods, the volume of water lost during this phase may be quite little. Thus, the moisture content loss was slower. The constant moisture content began after 8 hours of drying for both Noni fruits and leaves.

Fig. 1 indicated that the drying curve for leaves was faster than the drying curve for fruits. On the other hand, Noni leaves that dried at 70 °C was the quickest to dry (Table 1). It can be seen that the drying curve for leaves at 70 °C drop rapidly after 2 hours of drying. Meanwhile, the earliest drying curve for fruits to dry was after 4 hours of drying at 70 °C, followed by 6 hours drying time at 60 °C and lastly 8 hours drying time at 50 °C. This consistent with previous study by (Norhadi et al., 2020) that stated the substance's moisture content was extremely high during the early drying phase, resulting in higher energy requirement.

Table 1. Drying time for both Noni fruits and leaves at different temperatures

	Treatment	Drying time (hour)
Fruits	50 °C	14
	60 °C	12
	70 °C	10
Leaves	50 °C	12
	60 °C	10
	70 °C	8

Physicochemical Properties

Table 2 shows moisture content and water activity of Noni fruits and leaves powder dried using oven and freeze drying methods. Table 2 indicated a decreasing trend in moisture content (MC) and water activity (a_w) of Noni fruit powder with an increase in oven drying temperatures. For MC of Noni leaves powder, the mean values were significantly different with an increase in drying temperature compared to Noni fruit powder. A similar trend was observed for a_w where the mean values are decreased with an increase in oven drying temperature. For Noni leaves, drying at 50 °C and 60 °C show no significant difference with mean value of water activity was 0.61. However, drying at 70 °C had shown significant difference for Noni leaves powder. This outcome was in line with the earlier research on dried pumpkin powder, which found that drying at 70 °C resulted in the lowest moisture content and water activity compared to 50 °C and 60 °C drying (Roongruangsri & Bronlund, 2016). When compared to freeze drying, water activity of both Noni fruits and leaves powder were significantly reduced compared to oven drying. This result proved that freeze drying was better drying methods due to the lyophilization which involve the process of sublimation to achieved moisture removal (Fan et al., 2019). Hence, better quality of fruits and vegetables produced as higher amounts of free water will lead to shorter shelf life and spoilage of microbe. Thus, the removing water to a certain level to prevent deterioration from chemical reaction and microbial spoilage by drying was crucial (Oprica et al., 2019).

Table 3 summarized the color values for both powdered and fresh fruits and leaves. For Noni fruits, L^* value shows no significant difference between different drying temperature. The fresh fruits indicate higher value of L^* than oven dried but lower than freeze drying. This related to the process of freezing that can reduce the oxidation. The fresh leaves had highest value of L^* in leaves. Drying leaves at 70 °C exhibited a significant difference in L^* value compared to drying at 50 °C and 60 °C. Despite the fact that there was no significant difference in L^* value for fruits in the oven drying method, it had been shown that the lightness decreased as temperature increased. This behaviour occurs due to the Mailard reaction and non-enzymatic processes that generate the brown pigment cause the lower lightness value (Mireles-Arriaga et al., 2016). The result was consistent with Isik et al. (2019) for dried bee pollen samples and Arslan & Özcan (2011) for red pepper oven dried at 50 °C and 70 °C, which reported the L^* values decreasing in the range of 24 to 31% from fresh product. There is also a significant difference in L^* value between fruits and leaves.

In contrast to fresh fruits, Noni fruits that had been oven-dried had greater values for both a^* and b^* . However, compared to the leaves, the fresh leaves having a greater value of b^* than the fresh fruits. A greater a^* value meant that the leaves were more green than yellow, whereas a lower b^* value meant that the leaves were less yellow. B^* values decreased when the drying temperature increased, as reported in a previous study (Isik et al., 2019). Comparing freeze drying and oven drying, there was significant variation in the L^* (lightness) and a^* values (red/green) of both fruits and leaves

Table 4 present water solubility, bulk density and tapped density for oven drying and freeze drying of Noni fruits and leaves.

Table 2. Moisture content and water activity of fresh, oven drying and freeze drying for Noni fruits and leaves

	Treatment	Moisture content (%)	Water activity, a_w
Fruits	Fresh	82.80±2.07 ^a	24.84±0.030 ^a
	Oven drying		
	50 °C	9.79±0.04 ^b	0.59±0.003 ^b
	60 °C	9.78±0.04 ^b	0.54±0.005 ^c
	70 °C	8.51±0.17 ^b	0.51±0.006 ^c
	Freeze drying		
	-50 °C	8.35±0.26 ^b	0.43±0.003 ^d
Leaves	Fresh	79.83±0.56 ^a	24.86±0.015 ^a
	Oven drying		
	50 °C	9.80±0.06 ^b	0.61±0.007 ^b
	60 °C	9.06±0.04 ^c	0.61±0.002 ^b
	70 °C	7.92±0.16 ^d	0.59±0.006 ^c
	Freeze drying		
	-50 °C	7.57±0.17 ^d	0.49±0.002 ^d

Mean within the same column with different letters are significantly different ($p < 0.05$), $n=3$

Table 3. Color of fresh, oven dried and freeze dried Noni fruits and leaves powder

	Treatment	L*	a*	b*
Fruits	Fresh	58.72±1.19 ^b	-0.39±0.04 ^c	8.46±1.58 ^b
	Oven drying			
	50 °C	43.16±0.36 ^c	6.60±0.27 ^a	18.65±0.31 ^a
	60 °C	42.97±1.04 ^c	6.38±0.09 ^a	18.62±0.20 ^a
	70 °C	42.50±0.47 ^c	5.85±0.20 ^b	18.60±0.41 ^a
	Freeze drying			
	-50 °C	69.41±0.51 ^a	6.36±0.06 ^a	18.62±0.12 ^a
Leaves	Fresh	42.09±0.10 ^a	-17.37±0.60 ^c	31.02±0.61 ^a
	Oven drying			
	50 °C	33.19±0.47 ^c	-0.46±0.08 ^a	17.60±0.35 ^c
	60 °C	32.37±0.50 ^c	-0.57±0.03 ^a	15.67±0.21 ^d
	70 °C	29.81±0.09 ^d	-0.28±0.06 ^a	14.51±0.29 ^c
	Freeze drying			
	-50 °C	40.66±0.48 ^b	-5.35±0.11 ^b	27.87±0.28 ^b

Mean within a same column with different letters are significantly different ($p < 0.05$), $n=3$

Water solubility measures the amounts of chemical substances that can dissolve in water. The water solubility showed a decreasing trend with temperature during oven drying for both fruits and leaves. Drying at 70 °C and 60 °C had significant differences from 50 °C for both samples. This result was corresponding to Roongruangsri & Bronlund (2016). It implied that an increase in drying temperature was accompanied by a decrease in water solubility. Sengkhamparn et al. (2013) also had similar outcome where it reported that higher protein denatured due to higher drying temperature result in lower water solubility.

Table 4. Water solubility, bulk density and tapped density for oven drying and freeze drying of Noni fruits and leaves

	Treatment	Water solubility (%)	Bulk density (g/cm ³)	Tapped density (g/cm ³)
Fruits	Oven drying			
	50 °C	22.02±0.54 ^b	0.42±0.01 ^a	0.47±0.01 ^b
	60 °C	21.84±0.47 ^a	0.42±0.02 ^a	0.51±0.01 ^{ab}
	70 °C	19.10±1.00 ^a	0.39±0.02 ^a	0.51±0.03 ^a
	Freeze drying			
	-50 °C	23.30±0.12 ^a	0.29±0.01 ^b	0.48±0.01 ^{ab}
Leaves	Oven drying			
	50 °C	24.13±0.68 ^b	0.43±0.01 ^a	0.49±0.01 ^b
	60 °C	23.27±0.58 ^a	0.46±0.02 ^a	0.52±0.02 ^{ab}
	70 °C	21.12±0.10 ^a	0.42±0.03 ^a	0.54±0.02 ^a
	Freeze drying			
	-50 °C	25.08±1.23 ^a	0.37±0.01 ^b	0.39±0.01 ^c

Mean within a same column with different letters are significantly different ($p < 0.05$), $n=3$

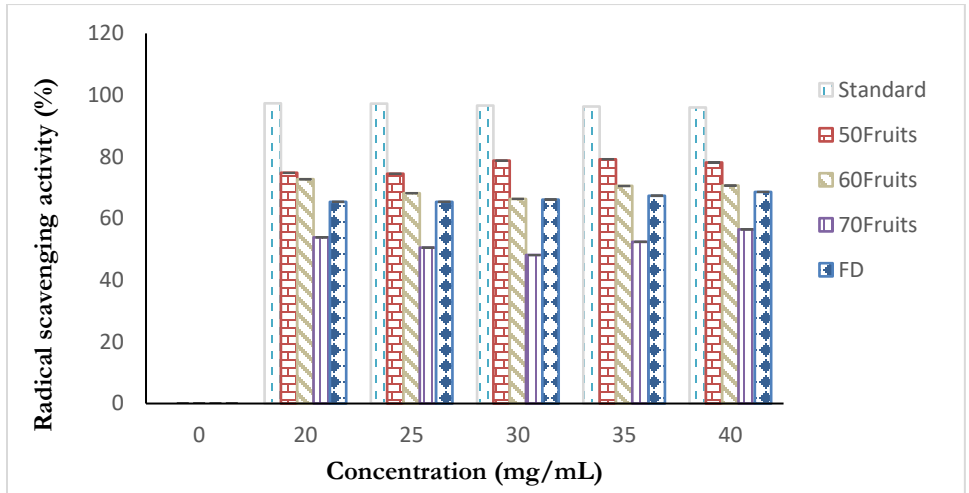
No significant variation was found in the water solubility of oven-dried fruits and leaves. However, freeze drying having a higher water solubility than oven drying in both samples. Lee et al. (2012) reported a finding of citrus "hallabong" powder that was freeze-dried had better water solubility than powder that was hot air-dried. This is because freeze drying can preserve and hold the powder's hard shape. As a result, the drying techniques affect a powder's ability to dissolve in water. Caparino et al. (2012) discovered that freeze drying methods had lower water solubility because cells were not disrupted. Consequently, a small quantity of material was dissolved.

Based on Table 4, there are no significant variations in bulk density between fruits and leaves that have been dried in an oven. It might be due to the fact that the powdered Noni fruits and leaves were sieved using a 250- to 500-mesh screen, which produces a narrow particle size distribution. According to previous studies, bulk density increased as particle size decreased (Lee et al., 2012). This showed that bulk density is influenced by particle size. Ferrari et al. (2012) reported that increased moisture content (lower temperature) results in higher bulking weight since water is present. Table 4 also shows that, with the exception of Noni fruits, the bulk and tapped densities for freeze-drying techniques were lower than those for oven drying. The lower value of bulk and tapped densities in freeze drying method is due to the particle size which not easily collapsed during tapping.

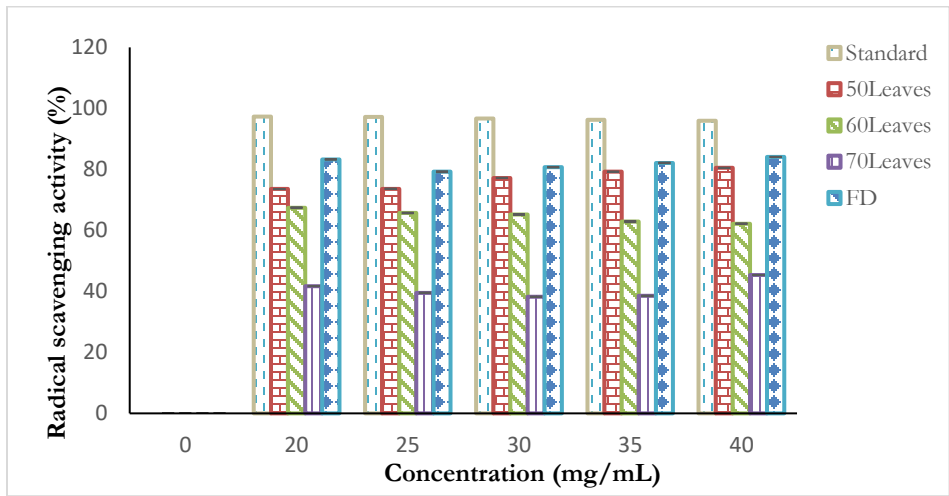
The scavenging of DPPH radical was measured in order to analyze or to evaluate the antioxidant activity of plant extract (Chen et al., 2020). Fruits, vegetables, and plants are high in phenolic compounds, thus, their extracts are frequently investigated and used as natural antioxidants in foods for preventing rancidity, delaying the generation of hazardous oxidation products, preserving nutritional content, and prolonging product shelf life (Shahidi & Ambigaipalan, 2015). Both figures indicated that the radical scavenging activity (%) for standard was the highest. A similar trend is shown for dried Noni leaves and fruits where drying at 50 °C was the highest (79% for fruits and 80% for leaves) percentage of radical scavenging activity which close to standard, 96%. Then, it was followed by drying at 60 °C and 70 °C. This result is agreed with Mireles-Arriaga et al. (2016)

findings where the percentage of DPPH inhibition for dried Noni slices at 50°C was the highest, 93.56%, among dried at 60°C (91.97%), 70°C (82.52) and was closely to the fresh (93.25). Previous study on dried fig (*Ficus carica* L.) leaves had reported that drying temperature up to 100°C caused thermal degradation which was the major cause of a notable drops in antioxidant activity (Elshaafi et al., 2020). This finding was supported by Kasunmala et al. (2021), which stated that temperature is the main factor in maintaining the antioxidant capacity of fruit pulp because high temperatures can contribute to irreversible oxidative processes for natural antioxidants

As shown in Table 5, the percentage of DPPH radical scavenging activity by freeze drying for both fruits and leaves had a significant difference with oven drying, where it was lower than drying at 50 °C but higher than 70 °C. Noni fruit and leaf powder had shown no significant difference in the percentage of DPPH radical scavenging activity by freeze-drying. In oven drying method, the percentage of DPPH radical scavenging activity for Noni fruits were lower than Noni leaves. This may be attributed to enzymatic processes by polyphenol oxidases (Kumar et al., 2022). Antioxidant content was different due to the variations in fruit pretreatment, maturity stage, and drying conditions. It was reported that in the transition from the green to the hard white stage, the overall concentration of phenolic compounds, antioxidant potential, and ascorbic acid content in the Noni fruit increases, then falls as the fruit ripens (Almeida et al., 2019). With the losses in total and individual polyphenolic compounds in freeze drying and oven drying techniques, it is possible to assume that alterations in phenolic compounds also contributed to the reduction in antioxidant activity of dried fruit (Coklar et al., 2018).



(a)



(b)

Fig. 2. Radical scavenging activity as a function of concentration for Noni leaves powder dried using oven drying and freeze drying methods

Table 5. DPPH radical scavenging activity of oven drying and freeze drying for Noni fruits and leaves

	Treatment	DPPH radical scavenging activity (%)
Fruits	Oven drying	
	50 °C	78.14 ± 4.19 ^a
	60 °C	70.66 ± 4.18 ^{ab}
	70 °C	56.47 ± 3.85 ^c
	Freeze drying	
	-50 °C	68.61 ± 0.29 ^b
Leaves	Oven drying	
	50 °C	80.50 ± 1.34 ^a
	60 °C	53.57 ± 6.23 ^c
	70 °C	45.36 ± 0.34 ^c
	Freeze drying	
	-50 °C	67.57 ± 6.87 ^b

Mean within a same column with different letters are significantly different ($p < 0.05$), $n = 3$

CONCLUSION

As conclusion, drying of Noni fruits and leaves by freeze-drying and oven drying was successfully conducted in this study. From the findings, the application of both drying processes was caused changes on the antioxidant, moisture content, water activity, water solubility, density and color of both Noni fruits and leaves powder. The results show that the decrease in water activity and moisture content in both oven drying and freeze-drying for Noni fruits and leaves contributes to shelf stability. Higher temperatures in oven drying can induce lower water solubility of Noni fruit and leaf powder. It can be concluded that drying at 50 °C is most preferable in oven drying as it has the highest percentage of DPPH radical scavenging activity and has the standard range of water activity (< 0.6). Meanwhile, bulk and tapped density in this study demonstrated that freeze-drying is slightly lower than oven-drying. The study suggest that freeze-drying could be used in future as it can retain the physicochemical properties of both Noni fruits and leaves powder. Drying on Noni fruits and leaves also could be carried out to produce powder product with longer shelf life and high nutritional value.

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