

Evaluation of factors affecting the freeze-dried process of durian (*Durio zibethinus* L.)

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Abstract

Durian (*Durio zibethinus* L.) is a famous fruit with high economic values in Vietnam and is gradually becoming popular worldwide. However, studies have shown that durian flesh can quickly deteriorate after 3-4 days of storage at room temperature. Besides developing suitable preservation methods, research on diversifying durian products is a sustainable solution that should be considered. The objective of this research is to develop a freeze-drying process for durian-based products with good organoleptic quality as well as high nutritional value. Several factors were selected for the present study, including durian flesh thickness (1, 2 and 3 cm), freezing time (180 mins, 360 mins, 540 mins and 660 mins), and drying time (360 mins, 600 mins and 840 mins). The experiments were carried out in a one-factor arrangement. Results have shown that all surveyed factors have a statistically significant influence on moisture, hardness, color difference, and total polyphenol content ($p < 0.05$). The best drying efficiency was obtained when durian flesh thickness was 1 cm, freezing time of 540 min and drying time of 600 min. Samples after drying have moisture 2.87%, hardness 20.76 N/cm², total polyphenol content 107.02 mg GAE/100 g dry material and no fat oxidation (as evidenced by peroxide index). This research result was considered basic data for the application of freeze-dried durian products on an industrial scale in the future.

1. Introduction

Drying is a method of reducing the moisture content of food materials to reduce the growth and spoilage of microorganisms, in order to prolong the shelf life of the product (Serna-Cock *et al.*, 2015). Dried products will reduce costs, packaging time, and transportation (Mulatu, 2010). This method is widely applied to fruit and vegetable products because it is very perishable due to high humidity and consumers' demand for year-round supply (Omolola *et al.*, 2017). Commonly used drying methods include hot air drying, heat pump drying, osmotic dehydration, and microwave drying (Serna-Cock *et al.*, 2015). These drying methods reduce water activity and thereby reduce the number of enzymatic, chemical, and microbial reactions (Qiu *et al.*, 2019; Chen *et al.*, 2020). Dehydration methods should minimize the loss of nutrients and antioxidant compounds (Santos *et al.*, 2008). However, there are some antioxidant compounds that are weakly bound to the water in the

fruit, so these nutrients are lost. In other words, some amount of soluble solids in the fruit is lost during dehydration (Ceballos *et al.*, 2012).

In addition, the freeze-drying method which is the most popular process today, is the process of separating water from the product from a solid (freezing) to a vapor under conditions of low temperature and pressure below the triple-O point (0.0098°C, 4.58 mmHg), the temperature below the crystallization point of moisture in the product ($< 0^\circ\text{C}$, pressure below 4.58 mmHg) (Różyło *et al.*, 2020; Bhatta *et al.*, 2020; Chumroenphat *et al.*, 2021). As a result, the product after drying almost retains the original natural quality of the raw materials: proteins are not denatured or hydrolysed, glucid is not gelatinized, lipids are not oxidized, vitamins and other activities biological matter is not destroyed, color, taste are not changed, and fiber and mineral substances are preserved.

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Products after freeze-drying will have a porous structure, especially when immersed in water, and will revert to its original state, which other methods cannot do (de Ancos *et al.*, 2018; Silva-Espinoza *et al.*, 2019; Nowak *et al.*, 2020). Thanks to the above advantages, it often applies freeze drying to dry high-class products such as royal jelly, cordyceps, bird's nests, reishi mushrooms, pharmaceuticals, vaccines and probiotics, fruit pulps of difficult-to-preserve fruit such as durian, and avocado, allows to retain vitamins, enzymes and rare biological active substances, preserving the quality of raw materials, which still bring high economic value to the manufacturer, although this technology consumes more energy than other conventional drying methods, the equipment cost is still quite high, the operator requires a high level of expertise, and difficult to make the chamber vacuum sealed.

Durian (*Durio zibethinus* L.), family Bombacaceae, genus Durio, a typical fruit variety in the tropics, especially in Southeast Asia such as Malaysia, Thailand, Indonesia and the Philippines (Husin *et al.*, 2018; Mardudi *et al.*, 2021). In Vietnam, durian is grown mainly in the provinces of Tay Ninh, Tien Giang, and Dak Lak province with two main varieties of durian that are grown a lot, namely Ri 6 durian and Thai Monthong durian. Durian carries a variety of phenolic compounds used in various pharmaceutical formulations that have been implicated in antioxidant, antiallergic, antitumor, anti-inflammatory and antimicrobial activities (Brown *et al.*, 2012). Polyphenols have strong endothelial vasodilator activity, preventing nitrite acid degradation. Caffeic acid and quercetin are the predominant antioxidants found in the fruit. In a rat study that showed reduced effects on the aorta and liver, durian was suggested as an important nutritional supplement with cardioprotective and hepatoprotective effects (Leontowicz *et al.*, 2007). Durian contains Inositol which is basically an antioxidant that improves glucose metabolism and also reduces body mass index (BMI) (Ansari, 2016). In addition, durian also supports the treatment of polycystic ovary syndrome and supports hypoglycemia (Ansari, 2016). However, durian contains a lot of calories, when combined with milk, which is also rich in nutrients, it can cause indigestion, digestive disorders, interfere with the functioning of the liver and stomach, worse cases can lead to hypertension and heart attack. At present, freeze-dried products are not very popular in the Vietnamese market, so the process of studying the factors affecting the sublimation drying process of durian products.

The objective of this study is to develop a technological process for processing sublimation dried durian and to evaluate the physico-chemical and sensory

components of the dried product. In this study, three main contents were carried out such as investigating the effects of thickness, freezing time and drying time on product quality (moisture, hardness, color difference, and total polyphenol content).

2. Materials and methods

2.1 Raw materials

Durian (*Durio ziberhinus* L.) cultivar of type Ri 6, purchased in Ho Chi Minh City, Vietnam. Raw materials are collected from gardens in Tien Giang province. With a weight of 2-4 kg, the spines are evenly expanded, the fruit flesh is bright yellow, fragrant, and the seeds are flat, and the pulp obtained after peeling has an average weight of 0.6-1.8 kg as shown in Figure 1. After purchase, durian is brought to the laboratory and processed within the same day. The initial moisture content in durian is about 65%, color values include L* (77.66±1.89), a* (-1.18±0.54), and b* (32.43±0.99), the total dissolved solids content is about 55 - 57 (g/100 g dry matter), the total acid content is 0.4 - 0.42 (g/100 g dry material), and total polyphenol content is 129.29 - 132.00 (mg GAE/100 g). The chemicals performed during the survey were purchased in Merk, Germany and of high purity. The equipment used included a freeze-drying device (TKD-FD50, Takudo), colorimeter (Konica Minolta, CR-400, Japan), UV - VIS machine (Thermo Scientific, EVOLUTION 60S, USA), weighing scale moisture drying (MB90, Ohaus, USA), refractometer (YIERYI, 0 -33%, China), water activity measuring device (Novasina, LabTouch, Switzerland), hardness tester (Vietnam).



Figure 1. Input materials for research.

2.2 The technological process of freeze-drying durian products

Durian fruit after purchase is washed many times with water to remove all dirt on the surface of the fruit skin. After being drained, the durian fruit is separated by a sharp knife to remove the segments, and the hard durian segments or borers are removed from the processing process. Durian fruit is placed in a tray and frozen for the first time in the freezer at a temperature of

-20°C. Samples were frozen until the core temperature of durian rice reached -10°C. Usually, this process takes about 4 hrs. Durian fruit flesh after freezing for the first time is cut into shape and placed in a drying tray with a thickness of 1 cm. When the temperature of the drying chamber reaches -20°C, proceed to place the durian sample trays in the drying chamber. Sample drying was carried out through steps including -20°C for 600 mins, 40°C for 840 mins, 45°C for 360 mins and 50°C for 420 mins. After enough time to dry the sample, the durian sample was packed and preserved with aluminum packaging and assessed the criteria over time.

2.3 Methods of analysis and evaluation

2.3.1 Determine the total color change (ΔE)

The color change is used in the CIE Lab* color space, the reference color is based on the three values L^* , a^* and b^* . Color is determined through the CR-400 colorimeter. The colorimeter is pre-calibrated with a white standard cell. Durian samples before and after drying are placed on the measuring shelf. Hold the projector straight down to the sample and take the measurement. The sample is color measured 3 times, each time at two different locations of the sample, and then the average value is taken

The total color difference of durian samples before and after drying is calculated by the formula:

$$\Delta E = \sqrt{(L_2^* - L_1^*)^2 + (a_2^* - a_1^*)^2 + (b_2^* - b_1^*)^2}$$

Where ΔE is the total color difference value. Values a_1^* , b_1^* and L_1^* are measured values of fresh durian samples, a_2^* , b_2^* and L_2^* obtained from dried durian samples.

2.3.2 Determination of moisture content

The moisture content of the raw materials was measured using an infrared hygrometer (MB90, Ohaus, USA). The weight of the durian sample added was 0.5 ± 0.02 g.

2.3.3 Determination of hardness

The hardness of durian products was determined using a hand-held thermometer (GY-3). The measurement method is performed according to the instructions of the machine supplier. Calibrate the instrument's clock hands to zero, the measuring head used has a diameter of 7.9 mm. The test sample is placed on the table, using a moderate force penetrates the sample with the measuring tip of the device. At this time, the meter hand of the device will show the value of force used to penetrate the sample with the unit of kg/cm^2 .

2.3.4 Determination of total polyphenol content

Total polyphenol content was done according to the study of Dao *et al.* (2021) and with improvements (Dao *et al.*, 2021; Dao *et al.*, 2022). First, weigh 1 g of the sample and grind with 50 mL of alcohol in a blender, filter the sample through a filter cloth into the volumetric flask, continue to grind the residue with the remaining 50 mL, pour into the volumetric flask and makeup to 100 mL, filter the sample through The filter paper and the filter cloth are on top of the filter paper. Collect the extract and weigh 1 g sample or 10 mL sample solution. Then, use a micropipette to suck up 0.1 mL of sample, 0.5 mL of 10% folin, 0.4 mL of 7.5% Na_2CO_3 , shake vigorously by machine and continue incubating in the dark for 1 hr. Photometric measurement at 765 nm based on the gallic acid calibration curve. The total phenolic content was expressed in mg of gallic acid equivalent per gram of dry matter (mg GAE/g dry matter).

2.4 Statistical processing method

All treatments performed in the study were repeated 3 times. The results are presented as mean \pm standard deviation (SD). The data recorded in each experiment were calculated and graphed using Excel 2016. The analysis of variance one-factor (ANOVA) and the least significant difference (LSD) were performed using JMP software 13.0 at $p < 0.05$.

3. Results and discussion

3.1 Effect of durian flesh thickness on product quality after drying

The influence of material thickness on the quality of dried durian products is presented as shown in Figures 2 and 3. From Figure 2A, it shows that the moisture content of durian products increases gradually with the investigated thickness. The sample with a thickness of 1 cm gave the lowest moisture content with 2.24%, followed by the 2 cm sample with 4.2%, and the 3 cm sample with 6.55%. The difference in moisture content between the samples is because the thicker the sample, the longer the distance for moisture to travel from the inside of the material to the surface (Kek *et al.*, 2014). A similar experiment on the effect of material size on product quality after drying was also performed by Md Saleh *et al.* (2019). The analysis results show that the moisture content of the product after drying at a thickness of 6 mm is higher than that of the sample with a thickness of 3 mm. The vacuum-dried mango product made by Jaya and Das (2003) also showed the same trend as the moisture content of the dried samples increased with the investigated thickness.

The hardness value of durian after drying is shown in

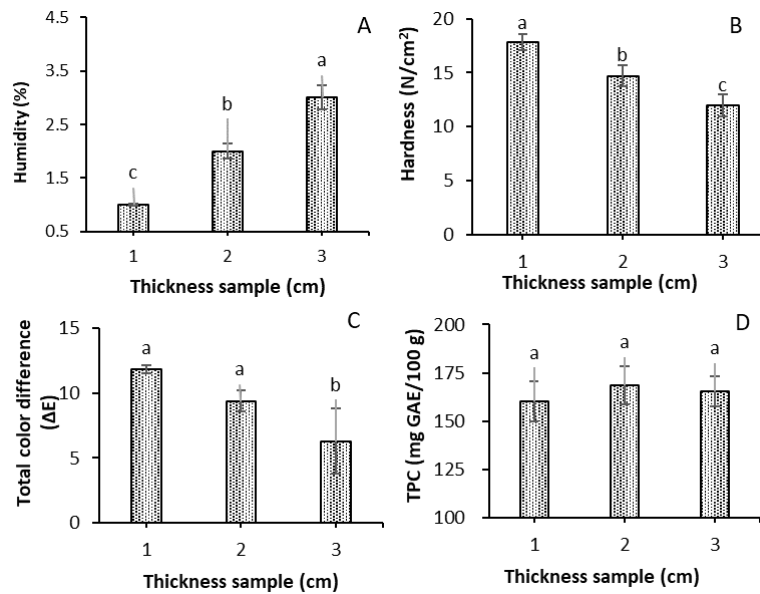


Figure 2. Effect of thickness on (A) moisture content, (B) hardness, (C) total color difference, (D) total polyphenol content in freeze-dried durian. Bars with different notations are statistically significantly different ($p < 0.05$).



Figure 3. Appearance of freeze-dried durian in 3 different thicknesses (A) 1 cm, (B) 2 cm, (C) 3 cm.

Figure 2B showing that the hardness decreases with increasing material thickness. The 1 cm sample with the highest hardness result of 17.82 N/cm^2 and the lowest value of 11.94 N/cm^2 was determined on the 3 cm sample. Carrying out a survey of the hardness value of some sublimated dried fruit products on the market such as purple dragon fruit, mango, pineapple, banana, the average hardness result was 15 N/cm^2 , 14 N/cm^2 , 13 N/cm^2 , 14 N/cm^2 , respectively.

The difference in color of post-dried durian compared with that of pre-dried samples at the three survey thicknesses (Figure 2C) can be seen with the naked eye because the ΔE values are all larger than 2 (Mokrzycki *et al.*, 2011). Durian after drying in three thicknesses is brighter and lighter in yellow color than that of pre-dried durian. The minimum ΔE value is 7.66 and was determined on a sample of 3 cm width. Durian after drying at a slice width of 1 cm had the most color difference compared to the pre-dried sample with ΔE value of 14.84. The characteristic yellow color deterioration of durian during drying is most likely due to auto-oxidation of total carotenoids during a rather long drying time (Zielinska *et al.*, 2007). In addition, the decreasing ΔE value with the slice thickness is thought to be related to the moisture value, the lower the water content of the sample, the carotenoid degradation will be faster (Pénicaud *et al.*, 2011). The total polyphenol content of the samples after drying at all 3 thicknesses

did not differ much with the value from $160.155\text{--}168.547 \text{ mg GAE/100 g}$ dry matter. When ANOVA analysis showed the effect of thickness on moisture, total color difference, hardness, and total polyphenol content of freeze-dried durian products with statistical significance (p value < 0.0001). The LSD classification test shows that there is a difference in 3 survey thicknesses of 1 cm, 2 cm and 3 cm at the 95% confidence level.

All 3 samples had bright yellow color (Figure 3), the characteristic aroma of durian, and crispy and spongy structure. However, according to the evaluation of the laboratory members, the sublimation-dried durian sample at the 1 cm sample had better brittleness than the 2 cm and 3 cm samples. In addition, this thickness gives the product the right size for a single use for the consumer.

3.2 Effect of freezing time on quality of freeze-dried durian

The effect of freezing time on the quality of freeze-dried durian is shown in Figures 4 and Figure 5. From Figure 4A, it shows that the moisture content of durian products after drying decreases gradually with freezing time. Samples frozen for 660 mins, 540 mins, 360 mins and 180 mins for the moisture values of durian after drying were determined to be 1.36%, 2.18%, 2.48%, 2.99%, respectively. The ANOVA analysis and the LSD showed that the four freezing periods examined had significant differences in humidity at the 95% confidence level.

The hardness value of the samples tended to increase gradually with increasing freezing time (Figure 4B). When frozen for 660 mins, the post-dried durian product showed the highest hardness of 26.16 N/cm^2 and the

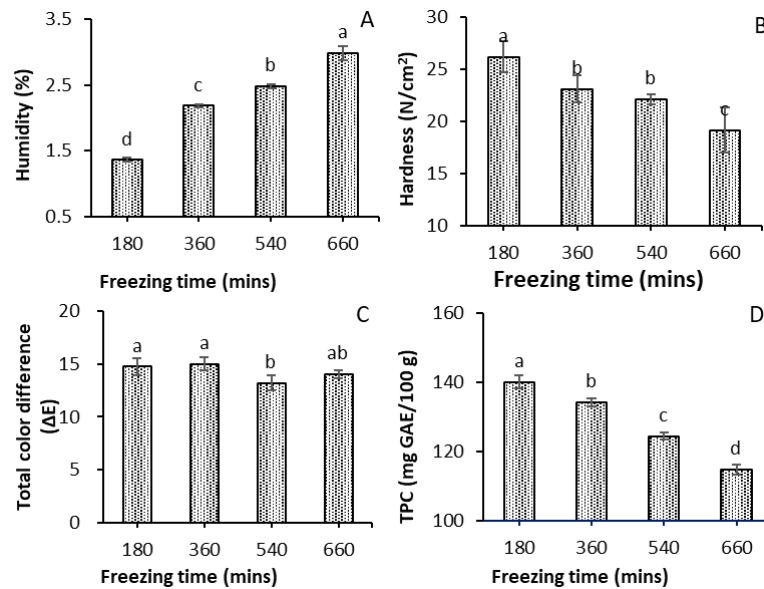


Figure 4. Effect of freezing time on (A) moisture content, (B) hardness, (C) color deviation, (D) total polyphenol content in freeze-dried durian. Bars with different notations are statistically significantly different ($p < 0.05$).

lowest value of 19.13 N/cm² at 180 mins. ANOVA analysis showed that the effect of freezing time on durian product hardness when freeze-drying was statistically significant with $p = 0.0028$ at 95% confidence level. The LSD classification test showed that the hardness value of durian after drying did not differ when frozen in the period from 360 mins to 540 mins. Meanwhile, at the remaining freezing time, the experiment recorded a statistical difference.

The total color difference value of the freeze-dried durian samples is shown in Figure 4C and the ANOVA analysis shows that the effect of freezing time on product color is not statistically significant with $p = 0.0362$ at the 95% confidence level. The ΔE value of durian after drying ranges from 14.01 to 15.01. From Figure 4D, it shows that the total polyphenol content in the dried durian samples gradually decreased with the prolongation of freezing time. The total polyphenol content of the samples after drying when frozen for 180 mins was the highest with 140 mg GAE/100 g dry matter and the lowest value was 144.722 mg GAE/100 g dry matter when frozen for 660 mins. The ANOVA analysis and LSD classification test showed that the total polyphenol content of durian after drying was different when frozen for 180 min, 360 mins, 540 mins and 660 mins at 95% confidence level. A study on the effect of freezing on frozen strawberry products explained that the freezing process will cause the cell wall of the fruit sample to break, leading to the loss of polyphenol content in the sample. During the freezing process, the water contained in the fruit pulp gradually turns to crystalline form, and sublimates during the freeze-drying process. The more water content of crystallization, the stronger the sublimation process of water. Sublimation may cause some water-soluble polyphenol compounds to escape from the pulp of durian fruit, leading to a

decrease in the content of this compound present in the dried sample (Ceballos *et al.*, 2012).

All 4 samples have bright yellow color, characteristic aroma of durian, and crispy and spongy structure. However, according to the sensory evaluation of the laboratory members, the freeze-dried sublimation durian sample frozen at 540 mins has better brittleness than the 660-minute sample and is not too dry compared to the sample 180 mins and 360 mins. Thus, based on the analyzed results, the freezing time of 540 mins was selected as the result of this experiment.

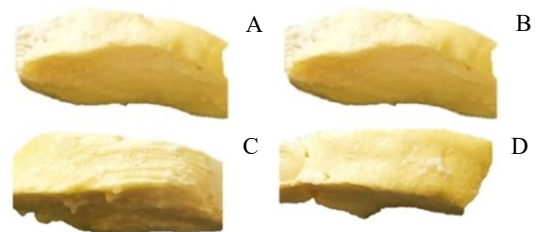


Figure 5. Appearance of durian at 4 freezing times (A) 180 mins, (B) 360 mins, (C) 540 mins, (D) 660 mins.

3.3 Effect of drying time on the quality of freeze-dried durian products

The effect of drying time on the quality of freeze-dried durian products is presented in Figure 6 and Figure 7. Figure 6A shows that the moisture content of durian samples after drying was 4.51%, 3.47%, and 2.89%, for the time levels of 360, 600, and 840 mins, respectively. In addition, the drying process took place in 360 mins and 660 mins, giving the product the lowest hardness of 13.16 N/cm² and the highest 23.24 N/cm². Corresponding to this time point, the total polyphenol content of the sample reached the highest value of 175.1 mg GAE/100 g dry matter (360-mins drying sample) and the lowest value was 122.64 mg GAE/100 g dry matter

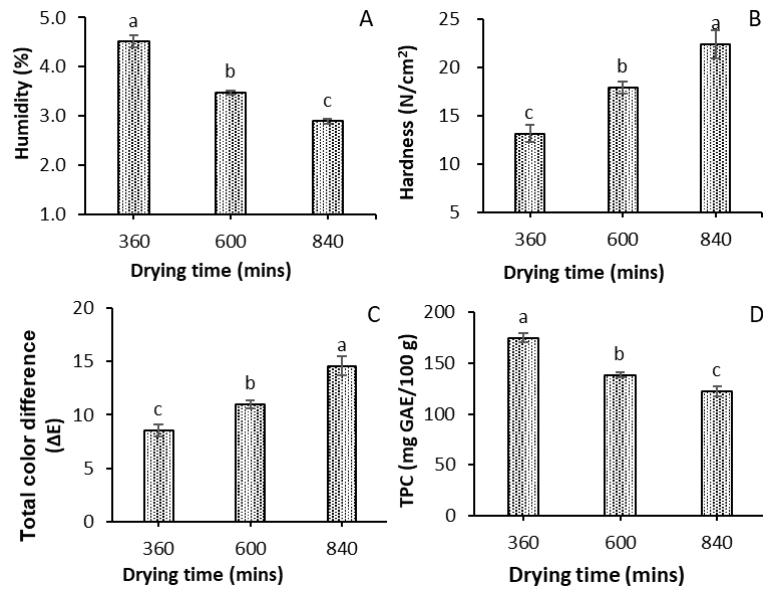


Figure 6. Effect of drying time on (A) moisture content, (B) hardness, (C) color, (D) total polyphenol content in freeze-dried durian. Bars with different notations are statistically significantly different ($p < 0.05$).

(sample drying 660 mins). Figure 6 also shows the value of ΔE of durian dried at 3 levels of surveying time ranged from 8.54-14.56, corresponding to drying time from 360 mins to 840 mins. From the analytical results, the study noted that the longer the drying time, the lower the moisture content and total polyphenol content of the sample, and the higher the hardness and ΔE value. The analysis results shown in the ANOVA analysis and the LSD classification test also show that different drying times have an effect on all the analytical parameters at the 95% confidence level.



Figure 7. Appearance of freeze-dried durian at different drying times (A) 360 mins, (B) 600 mins, and (C) 840 mins.

According to Antal *et al.* (2016), the longer the drying time, the greater the loss of water in the sample, leading to the lower moisture content of the sample. A study on dried apple products also showed the same trend that the moisture content of the samples decreased gradually with drying time. Beta carotene and polyphenols are temperature-sensitive compounds. During the sublimation drying process, although the sample is dried under vacuum conditions; however, the prolonged drying time at 50°C may have reduced the content of β -carotene and polyphenols in the durian flesh. This may have led to a reduction in the intensity of the yellow flesh of the fruit, increasing the color difference between the pre- and post-drying samples; as well as change the polyphenol content in the product after drying. In terms of organoleptic quality, samples dried at 600 mins received many good reviews from laboratory members, with better brittleness than other samples. In general, samples dried at 600 mins had better

total polyphenol content, moisture content as well as organoleptic results than the rest of the experimental samples.

4. Conclusion

Owing to the tropical monsoon climate, the fruit trees grown in Vietnam are diverse and regionally specific. A vast majority of fruit and agricultural products are cultivated in the Mekong Delta region. Therefore, post-harvest preservation and processing technology have been developed to meet the increasing needs of consumers. This study has proposed a process of the freeze-drying method with a total drying time of 32 hrs, which was 6 hrs less than the proposed process using the machine supplier. The specific drying conditions were carried out as follows: -20°C for 540 mins, 40°C for 600 mins, 45°C for 360 mins, and 50°C for 420 mins. The freeze-dried durian products have an average moisture content of 2.87% and a hardness value of 20.76 N/cm². The yellow color of the post-drying product is brighter than that of the pre-dried sample, which is shown by the L* and the b* values. The results of these surveys will help in the process of increasing the industrial scale for freeze-dried durian products at food factories, especially in the situation where durian products are popular and the durian is widely grown everywhere.

Conflict of interest

The authors declare no conflict of interest.

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