

## Comparison Of Red Ginger Dehydration Process Between Oven And Freeze Drying

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**Abstract.** As the tendency towards health awareness has increased, the consumption of dried herbs has risen. The phytochemicals within the plant were known for their health benefits. However, dried herbs in the market these days seemed to lack active compounds. The deficiency of active compounds could be evolving from the dehydration process. The research was established to compare the dehydration process in the dried red ginger (*Zingiber officinale* var. *rubrum*) production between freeze drying and oven drying. The oven drying process was done by drying in the oven at 60°C for 24 hours. While the freeze-drying process was done by freezing the red ginger at -60°C for 12 hours, then dried in a vacuum at 25°C. Physical appearance, microstructure, chemical constituents, moisture loss, extraction yield, Total Phenolic Content (TPC), and antioxidant activity were analyzed. The results showed that freeze-dried seemed to have a light green color while the oven-dried had a brownish-yellow color. The freeze-dried appeared to have less volume shrinking as the cell wall in the freeze-dried was more preserved, which was shown by the SEM imaging. GCMS results conveyed that freeze-dried looked to have more abundant compounds in higher molecular weight. In comparison, the oven-dried had more abundant in lower molecular weight. Also, the results indicated that there was a significant difference between freeze-dried and oven-dried in total phenolic content. However, there were no significant differences in moisture content, extraction yield, and antioxidant activity. Therefore, further research needs to be developed to compare both dehydration processes.

**Keywords:** dehydration, freeze-drying, red ginger, oven drying

### 1. INTRODUCTION

The use of plants as medicinal drugs is increasing these days due to the unique attributes as an abundant source of therapeutic phytochemicals. Azwanida et al. (2015) reported that the high content of phenolics and flavonoids in medicinal plants had been correlated with antioxidant activities. The antioxidant is essential in restricting fats against oxidation since the oxidation of polyunsaturated fatty acids in biological membranes drives to several diseases like atherosclerosis, emphysema, cancer, and cirrhosis (Stoilova et al. 2004). Other active compounds, including curcumin within red ginger, also have been proved to have pharmacological activities. Suciayati et al. (2017) discovered that curcumin had antibacterial and able to inhibit bacterial growth.

One of the plants that are rich in phytochemicals is ginger (*Zingiber officinale*). Though ginger is widely used as spices, ginger is also commonly known as a medicinal herb since ginger contains an abundance of antioxidants, active compounds, majorly gingerol, and their derivatives. As explained by Norhidayah et al. (2013), Gingerol and its derivatives act as a cancer preventive agent,

while the antioxidant activity improved health by fighting degenerative diseases caused by oxidative stress.

The problem to be examined involves the product of ginger, specifically red ginger, known in Indonesia as “Jahe Merah” (*Zingiber officinale* var. *rubrum*). Dried red ginger in the market these days seemed to have a lack of red ginger pungency compared to the fresh red ginger. The lack of red ginger pungency in the powder form also indicates the shortage of active compounds, specifically total gingerols and total shogaols (Jayashree et al. 2014). The loss of active compounds, specifically gingerol, could be coming from the dehydration process during the production of dried red ginger. The research was aimed to compare the active compounds between freeze drying and oven drying method in the production of dried red ginger. The data of active compounds changes between both dehydration methods will be analyzed. By analyzing both dehydration processes, the research could develop dried red ginger with a better quality of active compounds.

## 2. MATERIALS

The main materials used in this research were fresh red ginger. The fresh red ginger was bought at a local market (Citymarket) in Pondok Cabe, South Tangerang.

### Pre-treatment of drying red ginger

Fresh red ginger was cleaned by freshwater to scrub the dirt and detach the soil that sticks on the skin of the fresh red ginger. The cleaned red ginger then dried inside the room without using sunlight for an hour and soaked up with paper to absorb the excess water. Next, the red ginger was peeled and sliced with a diameter of  $25.0 \pm 0.5$  mm and a width of  $3.0 \pm 0.5$  mm. Afterward, the slice of red ginger was weighed and ready to be dried.

### Oven drying

$110.0 \pm 1.0$  g of red ginger slices were placed on a metal tray and then put at the bottom position of the oven (Ecocell 111MMM, Germany). Red ginger was dried at  $60^\circ\text{C}$  for 24 hours.

### Freeze drying

$55.0 \pm 1.0$  g of red ginger slices were spread on the metal tray and frozen at  $-60.0 \pm 5.0$  °C for 12 hours inside the cold trap of the freeze dryer (Biobase BK-FD10P, China). Then, the frozen red ginger was dried at vacuum pressure in a room temperature ( $28^\circ\text{C}$ ) for 24 hours.

Extraction of dried red ginger (An et al. 2015), (Zhang et al. 2018)

The extraction of dried red ginger was done as described by (An et al. 2015) and (Zhang et al. 2018) with modifications. The extraction of dried red ginger was done with a 1:6 (%w/v) ratio between dried red ginger powder and absolute ethanol solvent (FULLTIME, China). Dried red ginger that was extracted had been grounded with a blender (Tokebi V8000, South Korea) and filtered through a sieve 20 mesh. The extraction process with the maceration method was done using a magnetic stirrer (MSH-20D, South Korea) for 24 hours with an agitation speed of 400 rpm in a room temperature ( $28^\circ\text{C}$ ). Afterward, the solution was filtered with a Whatman 50 filter paper. Then, the solution was distilled at  $75.0 \pm 2.0^\circ\text{C}$  for an hour. The extract that was acquired was dried red ginger extract, which was used for further analysis.

## 3. METHODOLOGY

### Analysis of dried red ginger microstructure using SEM

The dried red ginger surface was observed using a scanning electron microscope (SEM Quanta FEG 650, United States). The dried red ginger with a  $5 \times 5 \times 5$  mm<sup>3</sup> was mounted on the SEM stub with

carbon tape. Then, the sample was inserted and observed in a magnification of 250, 500, and 1000x with 10 kV voltage.

### Analysis of chemical constituents within extract dried red ginger

The determination of chemical constituents was conducted in Chemical Lab at Universitas Islam Negara (UIN) Syarif Hidayatullah, Jakarta. Gas Chromatography-Mass Spectrometry (GCMS QP2010-SE Shimadzu, Japan) was utilized in analyzing the chemical constituents within the dried red ginger extract. The carrier gas that was used was helium with a flow of 0.98 mL/min. The temperature at the injector port was set at  $250^\circ\text{C}$  and utilized in split mode with a ratio of 1:100. The oven temperature was held at  $70^\circ\text{C}$  for 2 minutes, which then elevated to  $150^\circ\text{C}$  with a rate of  $12^\circ\text{C}/\text{min}$ , and raised again to  $250^\circ\text{C}$  at a rate of  $15^\circ\text{C}/\text{min}$ . The mass spectrometry was running with ion source temperature at  $250^\circ\text{C}$ , interface temperature at  $260^\circ\text{C}$ , and the acquisition mass range from 40 to 550 with time scanning of 27 minutes.

### Analysis of total phenolic content using Folin-Ciocalteu (Singleton et al. 1999)

Analysis of total phenolic content was done with Folin-Ciocalteu reagent following the method by Singleton et al. (1999) with few modifications. At first, 0.2 mL Folin-Ciocalteu reagent (Merck, Germany) was diluted with deionized water to 2.0 mL. 0.4 mL dried red ginger extract was mixed with 2.0 mL Folin-Ciocalteu solution and kept for 5 minutes in the darkness. Afterward, the Folin-Ciocalteu sample solution was added with 3.0 mL sodium carbonate solution (7.5% w/v) (Merck, Germany) and shaken vigorously. The sample solution was incubated at room temperature ( $28^\circ\text{C}$ ) for 2 hours in the darkness before analyzing the absorbance value with a Visible spectrophotometer (VWR V-1200, United States) at the wavelength of 765 nm. Total phenolic content was determined by using a gallic acid standard solution (Sigma-Aldrich, Germany). Total phenolic content was denoted as mg gallic acid equivalent (mg GAE/g extract).

### Analysis of antioxidant activity using DPPH (An et al. 2015), (Shimamura et al. 2014), (Abdelhady et al. 2011)

The determination of antioxidant activity was done with the method developed by An et al. (2015) with modifications. Sample of red ginger extract (0.4 mL) was reacted with 3.5 mL 0.14 mM DPPH (Merck, Jerman) solution, shaken vigorously, and then incubated for 30 minutes in the darkness. Afterward, the sample was measured its

absorbance value using a Visible spectrophotometer (VWR V-1200, United States) at a wavelength of 517 nm. Absolute ethanol (FULLTIME, China) was used to create a blank solution following the same procedure. Antioxidant activity was determined by using an ascorbic acid standard solution (Merck, Germany). Antioxidant activity was denoted as  $IC_{50}$  value (mg/mL) and Ascorbic acid Equivalent Antioxidant Capacity/AEAC (mg ascorbic acid/100 g of extract). The  $IC_{50}$  value was measured according to the method developed by Shimamura et al. (2014) and Abdelhady et al. (2011) with modifications.

#### Statistical analysis

All analyses and experiments were done in triplicate. The quantitative data results were reported as the mean of triplicate  $\pm$  standard deviation. The comparison data between freeze drying and oven drying treatment was assessed by independent samples t-test using IBM SPSS Statistics Version 25. Results of data analysis were further analyzed at 5% probability ( $P < 0.05$ ) using Microsoft Excel 2019.

## 4. RESULTS AND DISCUSSIONS

Effects of dehydration process on physical appearance and microstructure

Based on the observation, the color of red ginger looked to be more deteriorate in the oven-dried than the freeze-dried. The oven-dried sample (Figures 2 and 4) exhibited a brownish yellow color as the freeze-dried sample (Figures 1 and 3) had a light green color. This outcome was in line with the report by Krokida et al. (1998) that the oven drying resulted in brownish color as the rise air temperature yields an increase of yellow and red color within the sample. While the green color of the freeze-dried sample displayed more preserved pigment and compounds within the sample, as explained by Ratti (2001) that the internal heat transfer during freeze-drying is weaker compared to the oven drying. Regarding the volume appearance, the oven-dried seemed to be more shrinking in volume compared to the freeze-dried sample. Though the volume of the sample was not calculated, this result was in line with Ratti (2001) journal discussing that oven drying could shrink the volume up to 80% of the original volume while the freeze-drying only shrinks 5-15% of its original volume.



Figure 1 Freeze-Dried Red Ginger



Figure 2 Oven-Dried Red Ginger

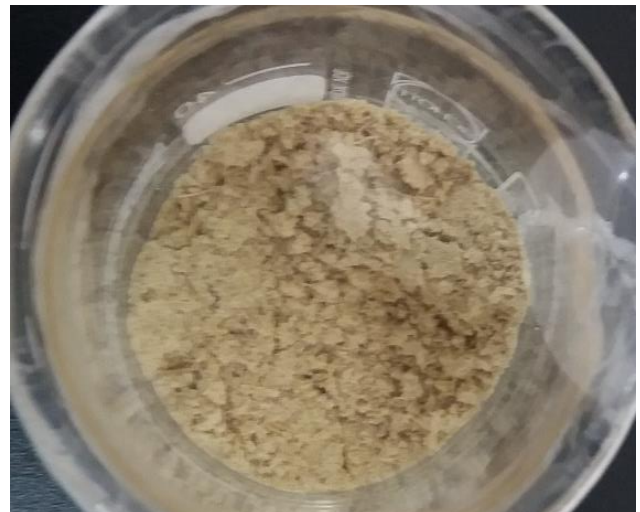


Figure 3 Freeze-Dried Red Ginger Powder



Figure 4 Oven-Dried Red Ginger Powder

Sharma (2017) mentioned that the images of red ginger microstructure taken by SEM were shown to have plant vessels indicated by cavities. Also, Buléon et al. (1998) reported that the images were shown to have starch granules in an ovoid to spherical shape. Starch granules looked like to be covered by the cell wall. The freeze-dried, which is shown in Figures 5 and 7, appeared to have a more preserved cell wall than the oven-dried sample (Figures 6 and 8). These results corresponded with the journal written by Keijing et al. (2015); this outcome could be due to the dehydration process by ice sublimation in a vacuum system, which leads to the more preserved cell wall compared to the oven-dried sample.

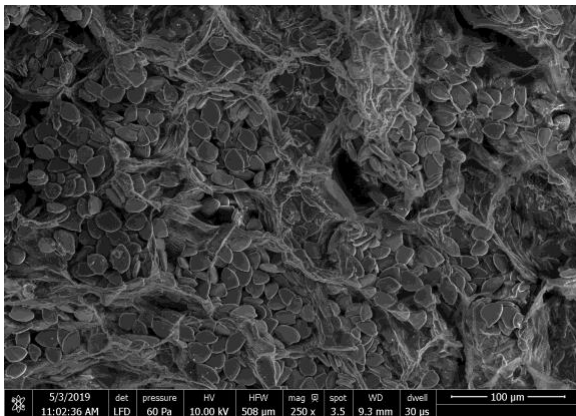


Figure 5 Secondary Electron Image of Freeze-Dried Red Ginger (250x)

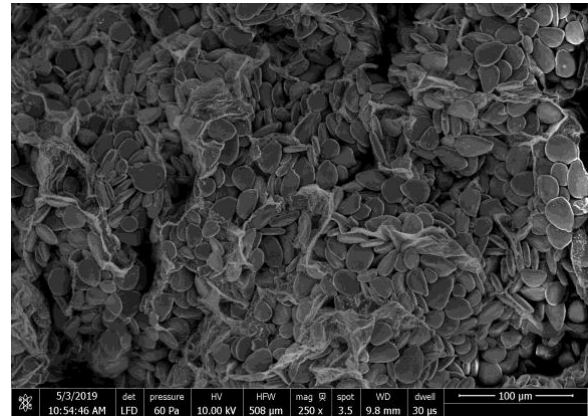


Figure 6 Secondary Electron Image of Oven-Dried Red Ginger (250x)

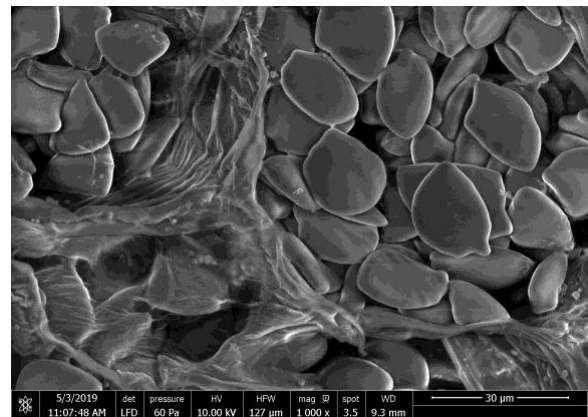


Figure 7 Secondary Electron Image of Freeze-Dried Red Ginger (1000x)



Figure 8 Secondary Electron Image of Oven-Dried Red Ginger (1000x)

Effects of dehydration process on the chemical constituents.

Oven-dried red ginger had slightly different chemical constituents compared to freeze-dried (Table 1). The black circles in Figures 9 and 10 show differences of compounds with lower molecular weight while the blue circles show a higher molecular weight. The red circles reveal the

compounds that were found in the freeze-dried but not in the oven-dried. The green circles show the compounds that were found in the oven-dried but not the freeze-dried. The results from GCMS showed that there were 16 peaks within each dried red ginger. There were 15 compounds recognized in the freeze-dried red ginger (Figure 9), while oven-dried red ginger (Figure 10) exhibited 16 compounds identified by GCMS. Freeze-dried and oven-dried red ginger had 14 identical compounds with one additional compound found in the freeze-dried only and two additional compounds found in the oven-dried only. Both dried red ginger extracts had zingiberene as the highest content, where freeze-dried had 28.28%, and oven-dried had 26.36%. Borneol and aromadendrene were not found in the freeze-dried sample but included as a part of oven-dried ginger extract. In contrast,  $\epsilon$ -muurolene was lost in the oven-dried but not in the freeze-dried.

Table 1 Peak Table of GCMS

No	Chemical Constituents	Retention Time Freeze Dried (min)	Retention Time Oven Dried (min)	Freeze Dried Plot Area (%)	Oven Dried Plot Area (%)
1	Borneol	-	7.948	-	1.11%
2	Beta-citral	8.706	8.708	2.31%	5.06%
3	Geraniol	8.833	8.831	3.16%	3.96%
4	Trans-citral	9.081	9.083	3.51%	7.53%
5	-(Unknown)	9.406	-	1.24%	-
6	Geranyl Acetate	10.761	10.765	5.51%	3.98%
7	Curcumin	13.185	13.192	5.46%	6.49%
8	Zingiberene	13.473	13.475	28.28%	26.36%
9	alpha-Farnesene	13.601	13.608	8.87%	8.73%
10	beta-Bisabolene	13.736	13.741	6.66%	6.77%
11	beta-Sesquiphallandrene	14.05	14.055	11.54%	10.98%
12	Aromadendrene	-	15.133	-	0.64%
13	Farnesol	15.478	15.481	0.88%	0.87%
14	Zingerone	15.8	15.804	4.36%	4.12%
15	$\epsilon$ -Muurolene	16.395	-	1.16%	-
16	6-Gingerol	21.833	21.833	9.68%	7.16%
17	8-Gingerol	22.989	22.992	4.07%	3.20%
18	10-Gingerol	24.697	24.692	3.31%	3.04%

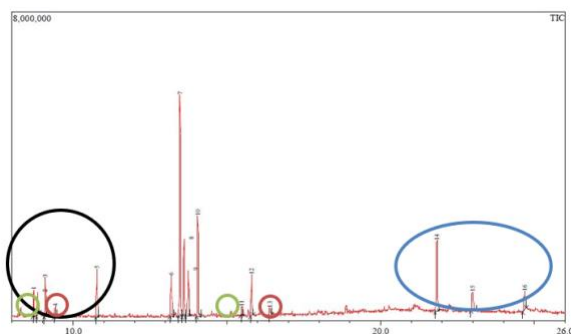


Figure 9 Freeze-Dried Red Ginger Spectra

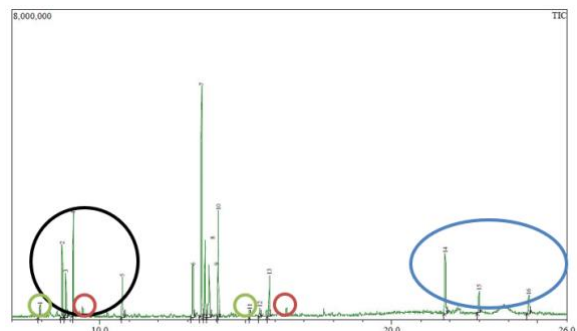


Figure 10 Oven-Dried Red Ginger Spectra

As it could be seen, most of the compounds that were detected in both extract were considered phenolics. Saranraj et al. 2019 described that sesquiterpenes, monoterpenes, and aromatic ketone were a part of phenolics since the compounds were aromatic hydrocarbons bonded with a hydroxy group (-OH). The chemical group of each constituent can be seen in table 2. In general, monoterpenes were higher in the oven-dried sample than the freeze-dried as Yadav et al. (2014) explained that there was a possibility of thermal degradation from sesquiterpenes to monoterpenes and most of the cases produce isoprene as the byproduct of the degradation. However, it is believed the borneol, in this case, did not generate in the oven-dried.

In contrast, the borneol could vaporize in the freeze dryer because the freeze dryer was operated in the vacuum condition (almost 40 Pa). The MSDS from National Center for Biotechnology Information (2019) showed vapor pressure of borneol is almost 4.5 kPa at 25°C. Thus, the borneol in the freeze-dried could be vaporized. Subsequently, the borneol was not a part of the freeze-dried extract. The GCMS results showed that gingerols and zingerone content were higher in the freeze-dried sample. Based on the GCMS results, zingerone appeared to be higher in the freeze-dried sample. It seemed that zingerone synthesized from gingerol is more favorable in the freeze dryer rather than within the oven. As for shogaols, they were not recognized in both samples. Most of the gingerols had higher content in the freeze-dried than the oven-dried. As Govindarajan (1982) stated, gingerols are easy to be degraded and converted into other products. For instance, gingerol can be dehydrated to form shogaols and zingerone, and another reaction is the retro-aldol condensation reaction from gingerol to zingerone (Figure 11). Gingerols at high temperature are dehydrated to shogaol, and the reason [6]-gingerol had the highest loss between all of the gingerols is that [6]-gingerol

has the lowest boiling point and it is much easier to be converted to shogaol compared to other gingerols which had been proven by Ghasemzadeh et al. (2018) study.

Table 2 Group of Chemical Constituent within Red Ginger Extract

No	Chemical Constituents	Chemical Group
1	Borneol	Monoterpene
2	Beta-citral	Monoterpene
3	Geraniol	Monoterpene
4	Trans-citral	Monoterpene
5	-(Unknown)	-(Unknown)
6	Geranyl Acetate	Sesquiterpene
7	Curcumin	Sesquiterpene
8	Zingiberene	Sesquiterpene
9	alpha-Farnesene	Sesquiterpene
10	beta-Bisabolene	Sesquiterpene
11	beta-Sesquiphallandrene	Sesquiterpene
12	Aromadendrene	Sesquiterpene
13	Farnesol	Sesquiterpene
14	Zingerone	Aromatic Ketone
15	ε-Murolene	Sesquiterpene
16	6-Gingerol	Aromatic Ketone
17	8-Gingerol	Aromatic Ketone
18	10-Gingerol	Aromatic Ketone

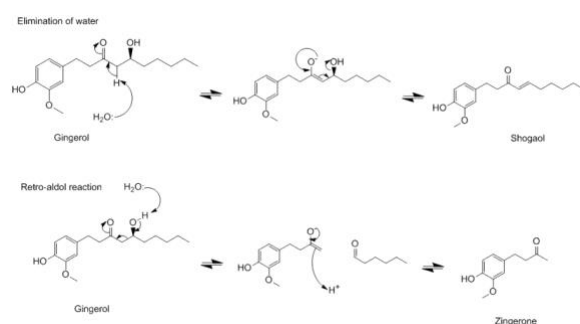


Figure 11 Reaction of Gingerol to Shogaol and Zingerone (Morgan, 2019)

Effects of the dehydration process on the moisture loss

Within 24 hours, the oven drying could provide moisture content loss  $82.49 \pm 0.43\%$  and the freeze-drying could dehydrate  $81.66 \pm 0.59\%$  moisture content within the red ginger. Nonetheless, there was not any significant difference between the freeze-drying and oven, as proven by independent samples t-test.

Effects of dehydration process on the extraction yield. Based on the extraction that had been done, the extraction yield of freeze-dried red ginger was  $9.15 \pm 0.53\%$ , while the oven-dried sample was  $7.87 \pm 3.93\%$ . However, corresponding to the independent samples t-test, the study found that there were no significant differences between dehydration processes. The high standard deviation of the oven-dried sample also designated that the data were spread out over a broader range of values. The issue could occur due to the entrapment of the solvent within the sample. Though the time duration of the filtration of red ginger ethanol solution was controlled, the rate of filtration before

distillation looked to be different between the freeze-dried and oven-dried red sample. This led to exhibit a different volume between dehydrated red ginger samples and caused the data to be not reliable.

Effects of dehydration process on the Total Phenolic Content (TPC)

The different treatment of the dehydration process showed to have a different total phenolic content of the red ginger extract between the oven-dried sample and freeze-dried sample. The TPC of the freeze-dried sample was shown to be  $79.60 \pm 0.55$  mg GAE/g extract, while the oven-dried sample was  $69.16 \pm 1.02$  mg GAE/g extract. The results were also showing a significant difference between the freeze-dried red ginger and oven-dried red ginger.

According to Asami et al. (2003), the freeze-drying may lead to higher TPC because of the ice crystal within the plant matrix. Considering ice drying developed in a more prominent rupturing of plant cell structure, this result to the better solvent access toward the sample. The better solvent access lead to a higher extraction yield. As the oven drying does not provide ice crystals, and the utilization of high temperature might cause thermolabile condensation. Thus, heat can induce phenolic losses.

Effects of dehydration process on the antioxidant activity

It was indicated that the IC<sub>50</sub> of the oven-dried sample was higher than the freeze-dried sample. The IC<sub>50</sub> of the oven-dried sample was shown to be  $750.85 \pm 29.40$  mg/g extract and AEAC of  $4.87 \pm 0.04$  mg ascorbic acid/100g of the sample. While the IC<sub>50</sub> of the freeze-dried sample was  $718.63 \pm 39.19$  mg/g extract with AEAC of  $4.96 \pm 0.05$  mg/100 g of the sample. Huyut et al. (2017) explained that Lower IC<sub>50</sub> means DPPH inhibition is more efficient with a lower amount; in other words, antioxidants within the freeze-dried sample was higher than the samples within the oven-dried. Despite that, there was no significant difference, as proven by the independent samples t-test.

As the journal written by Asami et al. (2003), the explanation of why the freeze-dried sample had higher antioxidant activity than the oven-dried sample might be due to ice crystal formation within the plant matrix that rupture the cell structure. The rupture led to a smoother diffusion of solvent to the dried red ginger. Thus, the solute is much more uncomplicated to be extracted means antioxidant is much easier to be extracted from the samples that processed through the freeze dryer compared to the oven drying. Therefore, extraction yield has a

significant role in the antioxidant activity within the sample.

## 5. CONCLUSION

To sum up, freeze-drying and oven drying have their uniqueness and advantages. The freeze-dried red ginger appeared to have a light green color while oven-dried red ginger looked to have a brownish yellow color. Also, the physical volume appearance of the oven-dried red ginger gave an impression to be more shrinking than the freeze-dried red ginger. The more shrinking in the volume of oven-dried red ginger could come from the collapse of the cell wall in the oven-dried red ginger, which was shown by the SEM images. The cell wall is related to the volume shrinking since the cell wall strengthens and preserves the structure of the plant.

Based on the GCMS results, the freeze-dried red ginger suggested having compounds that contain higher molecular weight. The spectra of oven-dried red ginger are displayed to hold compounds that exhibit lower molecular weight. The drying of the red ginger by using a freeze dryer lost  $81.66 \pm 0.59\%$  of its moisture. At the same time, the drying of red ginger by the oven lost  $82.49 \pm 0.43\%$  of its moisture. The independent samples t-test showed that there was no significant difference in the moisture loss between freeze drying and oven drying. As for the extraction yield, freeze-drying yielded  $9.15 \pm 0.53\%$  red ginger extract while the oven drying yielded  $7.87 \pm 3.93\%$  red ginger extract. The results indicated that the analysis of extraction yield had no significant difference between freeze-dried red ginger and oven-dried red ginger.

About the phenolic content of dried red ginger, the results from the Folin-Ciocalteu assay present that the freeze-dried red ginger contained  $79.60 \pm 0.55$  mg GAE/g extract. In comparison, the oven-dried contained  $69.16 \pm 1.02$  mg GAE/g extract, and there were significant differences between freeze drying and oven drying. However, the DPPH assay revealed that there was no significant difference between both dehydration processes even though the  $IC_{50}$  of freeze-dried was  $718.63 \pm 39.19$  mg/g extract as the  $IC_{50}$  of oven-dried was  $750.85 \pm 29.40$  mg/g extract.

Thus, the analysis of extraction yield, moisture content, and antioxidant activity between freeze-dried red ginger and oven-dried red ginger need further research and evidence to signify the difference between both dehydration processes.

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