PHYSICOCHEMICAL CHARACTERISTICS OF MICROENCAPSULATED NUTMEG SEED (Myristica fragrans) OLEORESIN USING FOAM MAT DRYING METHOD

Victoria Kristina Ananingsih, Bernadine Agatha, Bernadeta Soedarini

kristina@unika.ac.id

Nutmeg is a spice plant native to Maluku, Indonesia. One of the products from nutmeg processing is oleoresin. Nutmeg oleoresin is widely applied in the food and beverage industry because its flavor and aroma is easier to standardize and is more hygienic. However, oleoresin has less stable properties to heat, oxygen, and light. Therefore, an encapsulation process is needed to protect oleoresin. Oleoresin is obtained by extraction with ethanol organic solvent which has high polarity so that it can provide more extraction yields and is safe for use in food products. The extraction of nutmeg oleoresin is carried out by the ultrasonication method (Ultrasound Assisted Extraction) because of its lower energy consumption, shorter operating time, and can be carried out at low temperatures and pressures making it suitable for the extraction of compounds that are sensitive to high temperatures. Extraction was carried out at a temperature of 50˚C for 37.5 minutes at a frequency of 45 kHz and followed by separating the solvent with a vacuum rotary evaporator until oleoresin was obtained. Nutmeg oleoresin encapsulation was carried out by the foam mat drying method using two types of encapsulants (β-cyclodextrins and maltodextrin) at two treatment levels (7 grams and 10 grams) and the addition of Tween-80 emulsifier as much as 6% with two levels of stirring time (5 minutes and 15 minutes). The foam mat drying method is used because it is suitable for materials with high temperature sensitive compounds. This study aims to determine the the physicochemical characteristics of encapsulated nutmeg oleoresin using foam mat drying. The microencapsulate with the best trapped oil results were obtained in the treatment of 10 gram maltodextrin encapsulation with 5 minutes stirring time which produced 7.50% surface oil and 8.70% trapped oil. The microencapsulate with the best antioxidant activity results was obtained in the treatment of 7 grams of β-cyclodextrin encapsulant with 5 minutes stirring time which resulted in antioxidant activity of 85.40%.

RESEARCH BACKGROUND / INTRODUCTION

Nutmeg is a spice plant native from Maluku, Indonesia. Indonesia is known as the center of nutmeg plant species diversity and the biggest producer of nutmeg mace and nutmeg oil. Of the six types of nutmeg in Maluku, *Myristica fragrans* is the one that has the greatest economic value and is known to excel in the world market because of its distinctive aroma and high in oil yield (Nuryati & Yasin, 2016). Nutmeg processing into dried simplicia and powder for industrial raw materials still has various qualities and contains high microbial contamination causing the low value of nutmeg commodities. One effort that can be done is to process nutmeg into oleoresin (Rodianawati et al., 2015).

Oleoresin is pale yellow thick liquid which has distinctive aroma of nutmeg since it is a product of nutmeg processing (Nurdjannah, 2007). Oleoresin also has the similar taste and aroma as the original material which consists of a mixture of essential oils that contribute to volatile aroma and resins and other non-volatile compounds (Rodianawati et al, 2015). Oleoresin is obtained by extraction from nutmeg seeds or mace with organic solvents such as ethanol, methanol, acetone, or hexane (Gupta et al, 2013).

Nutmeg oleoresin can be applied in the food and beverage industry as flavor enhancer and aromatic agent, in the drug and pharmaceutical industry as ingredients, and in the soap and cosmetic industries (Nuryati & Yasin, 2016; Baihaqi et al, 2018). The use of oleoresin is considered superior compared to the whole form of nutmeg since oleoresin has a standardized flavor and aroma, is more hygienic, is sterile from bacteria and fungi, has small volume, is free from enzymes, and contains natural antioxidants (Nurdjannah, 2007). Antioxidant properties come from eugenol, isoeugenol, and phenolic compounds in nutmeg oil (Academic Press, 2011).

However, oleoresin is easily damaged when exposed to heat, oxygen, and sunlight, so a method to protect oleoresin is needed (Hill et al, 2013; Kfoury et al, 2015; Cevallos et al, 2010) One such method is encapsulation. Encapsulation is a coating method for sensitive and easily degraded materials. The core materials are coated with coating material or encapsulant (Ezhilarasi et al, 2013). Two types of widely used encapsulant are maltodextrin and β-cyclodextrin. Maltodextrin is widely used as a wall material because it has good stability in oil and water emulsions, good solubility in water, posseses the ability to inhibit oxidation reactions, and ease of handling during the process (Ezhilarasi et al, 2013). β-cyclodextrin is used because its ability to form a coating cavity which result in good encapsulation ability (Hadian et al, 2018). β-cyclodextrin cavity also has hydrophilic and hydrophobic properties on both sides which posseses the ability to encapsulate lipophilic core compounds in water emulsions (Crini et al, 2018).

Foam mat drying is used because it is suitable for high temperature sensitive materials (Qadri et al, 2019). Meanwhile, vacuum drying is used because it can produce better product quality since the taste and nutritional content in the core material are not damaged by high temperatures

during the drying process (Rezvankhah et al, 2019). Vacuum drying removes water by lowering the partial pressure of water vapor from the air in the drying chamber. Partial pressure has an influence on the drying speed, so the process is faster even though it uses a lower temperature compared to drying under normal pressure conditions (Sinaga, 2001, Ponciano et al., 2001, Pinedo et al., 2004 in Asgar et al., 2013). Therefore, it is necessary to conduct a research to determine the comparison of nutmeg oleoresin encapsulation with maltodextrin and β-cyclodextrin as encapsulants with encapsulant amount and stirring time variations using foam mat drying.

METHODS

1. Extraction of Nutmeg Oleoresin (Rahardjo, 2019)

A total of 1 kg of dried nutmeg seeds are ground with herbs grinder and sieved through 80 mesh sieve. The large nutmeg seeds are mashed with blender and sifted again. A total of 20 grams of nutmeg powder was dissolved in 200 mL of 96% ethanol in erlenmeyer. The ratio of solvent:nutmeg powder is 1:10. Extraction was carried out with Ultrasonic Cleaner UC-10SD. Erlenmeyer was immersed in ultrasonic water bath at 50˚C for 37.5 minutes with frequency of 45 kHz. The extracted sample was filtered using filter paper and Whatman filter paper no. 1. Ethanol solvent in the filtrate was separated by rotary vacuum evaporator to obtain oleoresin.

2. Encapsulation of Nutmeg Oleoresin (Azari, 2020 and Santoso, 2020 with Modifications)

A total of 5 grams of nutmeg oleoresin was added with encapsulants (β-cyclodextrin and maltodextrin) each with two levels of treatment (7 grams and 10 grams). For oven drying treatment, Tween-80 emulsifier was used as much as 6% of the total ingredients (1.14 g for 7 g encapsulant and 1.32 g for 10 g encapsulant) and 7 mL of aquadest. The mixture was homogenized with medium speed mixer with two treatment stages (5 min and 15 min). For vacuum drying treatment, 4 g of Whey Protein Isolate (WPI) emulsifier and 17 mL of aquadest were homogenized with rotor-stator homogenizer at 3500 rpm with two variations of homogenization time (5 minutes and 15 minutes). The emulsion was poured evenly onto a glass pan covered with a thick plastic layer. Drying was carried out with oven for 24 hours at 50˚C for oven drying treatment and in a vacuum oven for 48 hours at 50˚C in 0.5 atm pressure for vacuum drying treatment. The encapsulates were taken from a glass pan and ground into powder with blender and stored in zipper plastic bag with silica gel wrapped in aluminum foil and stored in a closed container. Each treatment was repeated twice.

3. Moisture Content Analysis (Santoso, 2020 with Modifications)

Moisture content analysis was carried out with moisture analyzer. The moisture analyzer was turned on and an empty aluminum pan was placed inside it. A total of 1 gram encapsulate was placed on the aluminum pan and waited for 10 minutes until the moisture content value appeared on the screen.

4. Analysis of Antioxidant Activity using Free Radical DPPH (*2,2-diphenyl-1-pycrylhidrazyl*) (Adiani et al, 2013 and Ginting et al, 2018 with Modifications)

For oven drying treatment, as much as 0.025 grams of encapsulate was dissolved in 10 mL of 99.98% methanol and allowed to stand for 2 hours. For vacuum drying treatment, 0.5 grams of encapsulate was added to 5 mL of methanol and allowed to stand for 1 hour. A total of 0.1 mL of the solution was added with 3.9 mL of DPPH solution and homogenized with vortex for 30 seconds and then incubated in a dark room at 25˚C for 30 minutes. The absorbance of the sample was measured with a spectrophotometer at a wavelength of 517 nm. Blanks were made from 0.1 mL of methanol to which 3.9 mL of DPPH solution was added in the same method. The absorbance of the blank was recorded as the control absorbance. Antioxidant activity was calculated as % inhibition according to the formula.

$$Antioxidant\ Activity\ (\%inhibition) = \frac{control\ absorbance - sampel\ absorbance}{control\ absorbance} \text{ x } 100\%$$

5. *Surface Oil* Analysis (Azari, 2020)

One gram of encapsulate was put into a centrifuge tube and 5 mL of ethanol was added and centrifuged at 1700 rpm for 15 minutes. The sample was filtered with filter paper and washed with 15 ml of ethanol. The filtrate was transferred to a porcelain cup that had been dried in oven for 24 hours and the initial weight had been known. The sample in the cup was dried in the oven for 24 hours and was then put in a desiccator for 15 minutes and weighed as the final weight.

Surface oil = final weight of cup– initial weight of cup

$$\%\ Surface\ oil = \frac{surface\ oil\ weight\ (g)}{sample\ weight\ (g)} \text{ x } 100\%$$

6. *Trapped Oil* Analysis (Azari, 2020 and Jayanudin *et al*, 2017)

One gram of encapsulate was put into an erlenmeyer and added with 20 mL of ethanol and then put into a water bath of Ultrasonic Cleaner UC-10SD at 50˚C for 45 minutes at a frequency of 45 kHz. The sample was filtered with filter paper and the filtrate was transferred to a porcelain cup that had been oven-baked for 24 hours which its initial weight was known. The sample in the cup was then dried in the oven for 24 hours and palced on a desiccator for 15 minutes. The cup containing the sample was weighed and recorded as the final weight.

Total oil = final weight of cup – initial weight of cup

$$\%\ Total\ oil = \frac{total\ oil\ weight\ (g)}{sample\ weight\ (g)} \text{ x } 100\%$$

Trapped Oil = Total oil – Surface oil

$$\%\ Trapped\ oil = \frac{trapped\ oil\ weight\ (g)}{sample\ weight\ (g)} \text{ x } 100\%$$

7. Encapsulate Morphological Analysis using *Scanning Electron Microscopy* (*SEM*) (Jayanudin *et al*, 2017 with Modification)

Morphological analysis of nutmeg oleoresin microcapsules was carried out using Scanning Electron Microscopy (SEM). SEM was coated with a thin layer of platinum with a high vacuum mode of 3.0 nm (30 kV) and a low vacuum mode of 4.0 nm (30 kV), and voltage acceleration of 0.5 to 30 kV. SEM was turned on and the microencapsulate was inserted into the specimen chamber. The image display would appear on the monitor screen in the most appropriate magnification.

8. Data Analysis

Data analysis was performed using parametric statistics with SPSS (Statistical Product and Service Solutions) program version 13.0. There were three independent variables consist of encapsulants, amount of encapsulant, and stirring time. Each independent variable consisted of two levels of treatment (maltodextrin and β-cyclodextrin for encapsulant; 7 grams and 10 grams for the amount of encapsulant; and 5 minutes and 15 minutes for stirring time). Normality test and homogeneity test of variance were performed. The significance difference between treatments was carried out by independent t-test.

RESULTS

1. Moisture Content (%)

Table 1. Moisture Content (%) of Microencapsulated Nutmeg Oleoresin

Treatments		Oven Drying	
Encapsulant Amount (g)	Stirring Time (minute)	β-Cyclodextrin	Maltodextrin
7	5	11.84 ± 1.81	3.09 ± 0.83
10	5	10.24 ± 0.28	4.31 ± 1.49
7	15	10.83 ± 4.85	3.21 ± 1.41
10	15	8.11 ± 1.62	4.42 ± 1.70

The results comprise mean ± standard deviation of two replicates.

According to the independent sample t-test, there was a significant difference between treatments of maltodextrin and β-cyclodextrin encapsulant on the moisture content of microencapsulates (see Table 4). Maltodextrin encapsulant results in lower water content. In oven drying, the moisture content of maltodextrin microencapsulation was lower (3.09% to 4.42%) than β-cyclodextrin (8.11% to 11.84%).

2. Trapped Oil (%)

Table 2. Trapped Oil (%) of Microencapsulated Nutmeg Oleoresin

Treatments		Oven Drying	
Encapsulant Amount (g)	**Stirring Time (minute)**	**β-Cyclodextrin**	**Maltodextrin**
7	5	4.30 ± 0.57	7.75 ± 2.05
10	5	3.38 ± 2.66	8.70 ± 2.69
7	15	2.30 ± 2.83	6.10 ± 1.27
10	15	3.40 ± 0.57	6.15 ± 0.92

The results comprise mean ± standard deviation of two replicates.

According to the independent sample t-test, there was a significant difference between treatments of maltodextrin and β-cyclodextrin encapsulant on the trapped oil of microencapsulates (see Table 4). The microencapsulates with maltodextrin had a higher trapped oil. In oven drying, the trapped oil of maltodextrin microencapsulation was higher (6.10% to 8.70%) than β-cyclodextrin (2.30% to 4.30%).

3. Antioxidant Activity (%)

Table 3. Antioxidant Activity (%) of Microencapsulated Nutmeg Oleoresin

Treatments		Oven Drying	
Encapsulant Amount (g)	Stirring Time (minute)	β-Cyclodextrin	Maltodextrin
7	5	85.40 ± 5.59	79.26 ± 2.86
10	5	73.03 ± 4.70	52.55 ± 4.63
7	15	77.67 ± 2.77	82.24 ± 2.93
10	15	68.90 ± 8.69	60.35 ± 3.28

The results comprise mean ± standard deviation of two replicates.

In oven drying, there was a significant difference between the treatment of encapsulant amount (7 g and 10 g) on the antioxidant activity of microencapsulates (see Table 4). The use of 7 grams encapsulant showed higher antioxidant activity than 10 grams, both on maltodextrin and β-cyclodextrin encapsulant in oven drying treatment

4. Microencapsulates Morphological Analysis Using SEM

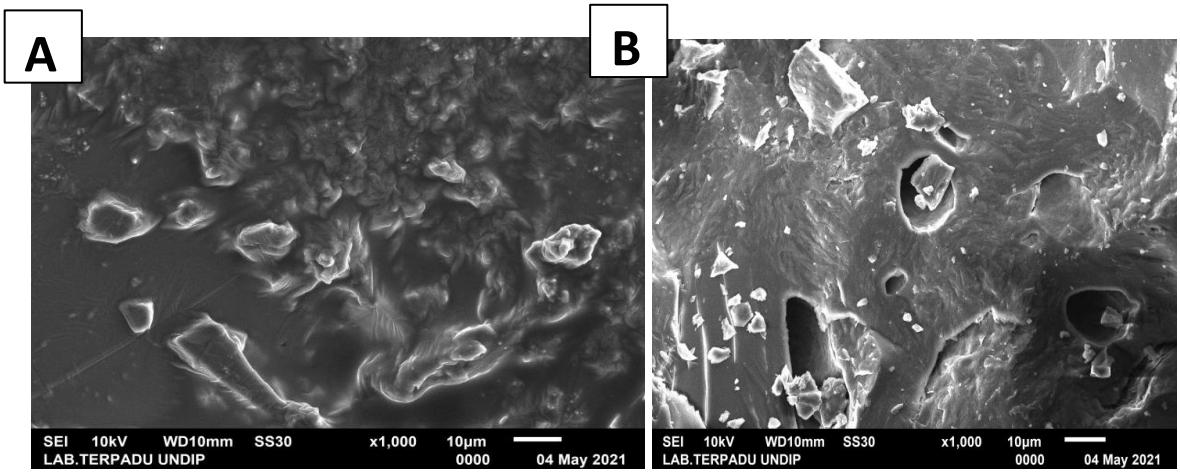

Figure 1. SEM micrograph of microencapsulated nutmeg oleoresin. (A) oven drying of maltodextrin, and (B) oven drying of β-cyclodextrin. The samples of 7 grams maltodextrin with 5 minutes stirring time and 7 grams of β-cyclodextrin with 15 minutes stirring time were used for morphological analysis. 500 magnification was used in vacuum drying sample while 1000 magnification was used in oven drying sample.

In vacuum drying, cracks were observed on the walls of maltodextrin microencapsulation (Fig. 1A). Cracks were also seen on the surface of β-cyclodextrin microencapsulation (Fig. 1B). In β-cyclodextrin with oven drying, a smoother wall surface without shrinkage with few cavities and few fractions in the wall was observed (Fig. 1D).

DISCUSSION

1.Moisture Content

Moisture content is an important factor which determine the quality and stability of food products during handling, processing, and storage (Vikas et al, 2018). The moisture content indicates the percentage of water contained in food. The lower the water content, the slower the growth of bacteria and fungi in food product (Qadri et al, 2019; Sangamithra et al, 2014). The low water content also prevents the hydrolysis which causes a decrease in the quality of oil (Vikas et al, 2018). Based on Indonesian National Standard (SNI 01-3709-1995), the maximum moisture content allowed for spice powder is 12%. In this study, the nutmeg oleoresin microencapsulates has met the requirements of moisture content based on SNI.

According to independent sample t-test, there was significant difference between treatments of encapsulant types (maltodextrin and β-cyclodextrin) on the moisture content of vacuum-dried and oven-dried microencapsulates (see Table 4). The maltodextrin encapsulation resulted in lower water content. In vacuum-dried microencapsulates, the use of maltodextrin encapsulants showed lower moisture content (between 5.4% to 6.1%) than β-cyclodextrin (between 8.04% to 9.31%). In oven-dried microencapsulates, the moisture content of maltodextrin encapsulant was also lower (between 3.09% to 4.42%) than β-cyclodextrin (between 8.11% to 11.84%).

The higher moisture content of β-cyclodextrin microencapsulates is caused by the cyclodextrin structure. Cycodextrin has characteristic in forming cavity which is hydrophobic in the inner side and hydrophilic in the outer side (Crini et al, 2018). The hydrophobic part of β-cyclodextrin cavity will be occupied by less polar guest molecules so that the water inside the cavity becomes more difficult to evaporate (Duchene & Bochot, 2016). Moreover, cyclodextrin also easily absorbs water from the atmosphere which may lead to higher moisture content (Mahmudah, 2015). In contrast, maltodextrin has a lower hygroscopicity (Azari, 2020). Therefore, the moisture content of maltodextrin microencapsulates could be maintained during the drying process and storage which are susceptible to air exposure. Furthermore, maltodextrin also has higher water evaporation rate because the water inside maltodextrin is easier to evaporate (Santoso et al., 2020). This property contributes to the lower mositure content of maltodextrin microencapsulates.

In both vacuum-dried and oven-dried microencapsulates, encapsulant amount and stirring time variations did not show significant difference in moisture content. This result is supported by the research of Muchtadi et al (2015) which mentioned that moisture content of palm oil microencapsulates did not show any significant difference between the homogenization treatments. Homogenization and stirring treatment aimed to perform reduction and uniformity of oil droplet size to be encapsulated (Kaushik & Roos, 2007). However, the moisture content of microencapsulates was more affected by the encapsulants treatment rather than stirring time

variation (Muchtadi et al, 2015). Another factor which affect the moisture content of microencapsulates is the drying process and the storage conditions (Muchtadi et al, 2015).

2.Trapped Oil (%)

Trapped oil denote the amount of oil covered in the encapsulates. Trapped oil is an important factor of microencapsulation efficiency. The higher the amount of trapped oil, the better the microencapsulation efficiency.

Trapped oil is obtained from the subtraction of total oil and surface oil. Total oil shows the total amount of oil in the microencapsulates whilst surface oil represents the amount of oil on the surface of microencapsulates. Surface oil affects the stability of microencapsulates during storage. Oil on the surface is not covered by encapsulant thus it is more susceptible to oxidation which cause the oil to be more easily damaged (Pourashouri et al., 2014 in Jayanudin et al., 2017). Thus the higher the percentage of surface oil, the lower the encapsulation efficiency.

Independent sample t-test showed significant difference between treatments of maltodextrin and β-cyclodextrin encapsulant on the trapped oil of vacuum-dried and oven-dried microencapsulates (see Table 4). Maltodextrin encapsulation has higher value of trapped oil with a range of 12% to 15.7% for vacuum drying and 6.10% to 8.70% for oven drying, while β-cyclodextrin encapsulation results in lower trapped oil in the range of 3.3% to 11.7% and 2.30% to 4.30% for vacuum and oven drying respectively.

Maltodextrin encapsulation results in higher trapped oil than β-cyclodextrin due to the higher viscosity of maltodextrin (Akhilesh et al., 2012 in Yonata, 2020). The high viscosity of maltodextrin emulsion causes the formation of thick microencapsulates walls. The thickness of the walls can prevent the migration of oleoresin to the outside of microencapsulates so that the amount of trapped oil is greater (Jayanudin et al., 2017). On the other hand, the drying rate on low-viscosity materials occurs more slowly so that the wall formation process takes longer (Layuk et al, 2018). The longer process of wall formation causes the oil can not be completely encapsulated so that the amount of surface oil is higher. In both vacuum and oven drying, stirring time variations did not show significant differences in trapped oil.

3.Antioxidant Activity (%)

Antioxidants are the chemical substances which donate one or more electrons to stabilize or prevent the formation of free radicals (Karadag et al, 2009; Ginting et al, 2018). Analysis of antioxidant activity in nutmeg oleoresin microencapsulates is very important since nutmeg possesses some natural antioxidant compounds. Eugenol, isoeugenol, and phenolic components are antioxidant compounds found in nutmeg seed oleoresin (Academic Press, 2011). Eugenol and β-caryophillene have hydrogen atoms in benzylic and allylic functional groups that can neutralize peroxyl radicals (Kumaravelu et al, 1996). The antioxidant activity occurs due to interactions between components in nutmeg essential oil (Adiani et al, 2013).

Phenolic compounds also contribute to antioxidant activity of nutmeg. Antioxidant capacity is reported to have a positive correlation with the amount of polyphenolic compounds (Tan et al, 2013). Essential oils and lignans in nutmeg are the two largest components that contribute to polyphenolic compounds (Tan et al, 2013). Myrisfragransin is nutmeg lignan which consists of meso-dihydroguaiaretic acid, erythro-austrobailignan-6, and argenteane as the main components (Academic Press, 2011). Lignans are phenolic compounds that act as antioxidants due to the structure of two cinnamic acids (Tan et al, 2013). Caffeic acid and catechins are other two main phenolic components in nutmeg oil (Shan et al, 2005). The catechol structure of catechin and caffeic acid compounds donates electrons to free radicals of reactive oxygen species or lipid peroxyl groups which resulting in nutmeg antioxidant activity (Shan et al, 2005).

During incubation of oleoresin microencapsulates extract, methanol was used as a solvent. Methanol is a polar solvent which can bind polyphenol compounds from the nutmeg matrix more effectively (Tan et al, 2013). Antioxidant activity is represented as % inhibition (Ginting et al, 2018). The greater the value of % inhibition, the greater the antioxidant activity. DPPH solution (2,2-diphenyl-1-picrylhydrazyl) in methanol acts as free radical which will be stabilized by antioxidant compounds in nutmeg oleoresin through a reduction reaction (Karadag et al, 2009).

In vacuum-dried microencapsulates, significant difference was observed between maltodextrin and β-cyclodextrin encapsulants on antioxidant activity of microencapsulates, while significant difference between 7 g and 10 g of encapsulant amount was observed on antioxidant activity of oven-dried microencapsulates (see Table 4).

In oven drying, the use of 7 grams encapsulants showed higher antioxidant activity compared to the use of 10 grams encapsulants. This happens because the amount of encapsulants affect the thickness of microencapsulates wall. The bigger the amount of encapsulants, the higher the emulsion viscosity which causes the encapsulates walls become thicker (Jayanudin et al, 2017). The thicker the wall, the more difficult it is for the oleoresin to migrate from the microencapsulate to dissolve in methanol extract (Santoso, 2020).

In vacuum drying, the value of antioxidant activity was significantly different between treatment of encapsulation type. Antioxidant activity with β-cyclodextrin encapsulation was higher

(87.06% to 88.42%) than maltodextrin (35.47% to 47.94%). This happens because β-cyclodextrin forms cavities in microencapsulates (Crini et al, 2018). β-cyclodextrin protects oleoresins by forming covalently stable complex bonds (Yonata, 2020). A better properties of thermal stability of β-cyclodextrin also supports the protection of antioxidant compounds, where the encapsulation process involves drying at 50˚C (Vikas et al, 2018). Anggrahini et al (2007) in Sembiring et al (2020) explained that the quality of the chemical content in nutmeg will decrease when the drying temperature exceeds 45˚C.

The lower antioxidant activity of maltodextrin encapsulation may occurred due to the thickness of microencapsulates wall which could affect the oil extraction from the microencapsulates. Assagaf (2013) stated that WPI (Whey Protein Isolates) forms wall that coats the oleoresin droplet on the lipophilic side, while the hydrophilic side will bind to maltodextrin. According to the research by Rahardjo (2019), the antioxidant activity of nutmeg seed oleoresin is in the range of 60.55%-98.36%. The results obtained were consistent, especially in β-cyclodextrin encapsulation. Meanwhile both in vacuum drying and oven drying, stirring time variation did not have a significant effect on the antioxidant activity.

4.SEM

Using SEM (Scanning Electron Microscope), the morphology of nutmeg oleoresin microencapsulate was observed in the size between 10 µm to 50 µm, thus it can be classified as microencapsulates since the particle size was less than 100 µm (Huang et al, 2020). In vacuum-dried microencapsulates (Figures 1A and 1B) and oven-dried microencapsulates with maltodextrin (Figure 1C), cavities and pores were observed in the microencapsulates wall. This occurs due to the process of water evaporation during drying which causes shrinkage on the microencapsulate walls (Huang et al, 2020).

In vacuum-dried microencapsulates with maltodextrin (Figure 1A), cracks and crumbs were observed on the surface of the microencapsulates. These cracks may be formed as the result of size reduction of dried encapsulates at the late stage of encapsulation. During the process of encapsulation, the dried encapsulates were mashed with blender to reduce and uniform the size of encapsulates. The blender may cause cracks on some microencapsulates walls.

Cracks on the microencapsulates wall may also be formed due to the low foam stability during drying. Low foam stability can not maintain its structure during drying and results in broken morphological structure which looks like flakes as also reported in freeze-drying foam mats encapsulation of blueberry juice (Darniadi et al, 2020). The presence of cracks may increase the oxygen permeability of the encapsulates wall which can oxidize and degrade the core material of microencapsulates during storage (Dadi et al, 2020). Meanwhile in β-cyclodextrin encapsulation (Figure 1B), cracks on the wall were also observed but there were no crumbs at the microcapsule wall. This may happen due to the rigid structure of β-cyclodextrin as the coating material. β-cyclodextrin consists of seven hydrogen bonds which can produce complete hydrogen bond on the secondary side or the outer side and results in forming the rigid structure (Duchene & Bochot, 2016).

In oven-dried microencapsulates with β-cyclodextrin (Fig. 1D), smoother surface without shrinkage of microencapsulates wall was observed. The smooth surface supports better protection againts oxidation of oleoresin as the core material since smooth surface has smaller surface area than irregular and non-uniform shape surface (Dadi et al, 2020). However, foam mat drying tends to produce larger powder size than other drying methods such as spray drying (Kanha et al, 2020). This happens because foam mat drying emphasizes the formation of foam to increase the surface area so that the drying process can run faster at the temperatures that are not too high (Iqbal et al, 2018; Sangamithra et al, 2014). This is an advantage of foam mat drying method which can be applied to materials containing bioactive compounds that are susceptible to high temperatures such as oleoresin (Iqbal et al, 2018).

In oven-dried microencapsulates with β-cyclodextrin, cavities and pores on the wall were also observed (Fig. 1D). This morphology is in accordance with the results obtained from foam mat drying of yacon tuber juice (Franco et al, 2016). Cavities are also seen in the encapsulation of

blueberry juice with foam mat freeze drying (Darniadi et al, 2020). Cavities may be formed from air bubbles in the foam when it was dried (Franco et al, 2016). When water vapor is released from the foam during drying, foam bubbles with high stability and low density will form cavities with the same shape and size as the foam (Darniadi et al, 2020). The formation of cavities in the encapsulates was also seen in the results of encapsulation with foam mat drying of carotenoid extracts (Pinto et al, 2018). Although cavities are formed, the carotenoid extract remains encapsulated inside the wall granules so that the formation of cavities on encapsulates wall does not necessarily indicate a low protection ability (Pinto et al, 2018).

CONCLUSIONS

- Encapsulant types showed significant difference between treatments on moisture content and trapped oil of oven-dried microencapsulates.
- Encapsulant amount showed significant difference only in the antioxidant activity of oven-dried microencapsulates.
- Stirring time variations showed no significant difference in parameters.

- The moisture content of maltodextrin microencapsulates was lower (3.09% and 5.4% for oven) than β-cyclodextrin (8.11% and 8.04% - for oven).
- Trapped oil was higher on maltodextrin encapsulation (6.10% and 12% for oven and vacuum drying repectively) than β-cyclodextrin (2.30% and 3.3% for oven and vacuum drying repectively).
- In oven drying, the antioxidant activity of microencapsulates was higher at 7 g (77.67%) compared to 10 g (52.55%).

REFERENCES

Academic Press. (2011). *Nuts and Seeds in Health and Disease Prevention* Chapter 98: Antioxidant and Antimicrobial Activity of Nutmeg (*Myristica fragrans*). London: Gupta, A. D. & D. Rajpurohit. https://www.researchgate.net/publication/264972570_Antioxidant_and_Antimicrobial_Activity_of_Nutmeg_Myristica_fragrans

Adiani, V., S. Gupta, and S. Chatterjee. (2013). Activity Guided Characterization of Antioxidant Components From Essential Oil From Nutmeg (Myristica fragrans). Journal of Food Science and Technology Vol. 52: 221-230. Retrieved from https://link.springer.com/article/10.1007/s13197-013-1034-7

Asgar, A., Zain S., Widyasanti A., dan Wulan A. (2013). Characteristics Study of Drying Process of Oyster Mushrooms (Pleurotus sp.) Using Vacuum Dryer). Jurnal Hortikultural Vol. 23 (4): 379 – 389. Retrieved from http://hortikultura.litbang.pertanian.go.id/jurnal_pdf/234/Asgar_A.pdf

Assagaf, Muhammad, Pudji H., Chusnul H., Sri Y., Supriyadi. (2013). Characteristics of Microencapsulated Nutmeg Oleoresin: Determination of Encapsulant Whey Protein Concentrate (WPC): Maltodextrin (MD) Ratio. Agritech Vol. 33 (1): 16-23. Retrieved from https://journal.ugm.ac.id/agritech/article/download/9562/7137

Azari, Ineke Loan. (2020). Optimizing The Concentration Of Maltodextrin, Tween 80, And Stirring Time In The Physicochemical Characteristics Of Nutmeg Seed Oleoresin Encapsulate (*Myristica fragrans*) Using Foam Mat Drying Method. Other thesis, Unika Soegijapranata Semarang. Retrieved from http://repository.unika.ac.id/22980/

Baihaqi, I. W. Budiastra, S. Yasni, dan E. Darmawati. (2018). Improvement of Oleoresin Extraction Effectivenesss in Nutmeg by Ultrasound Assisted Method. Jurnal Keteknikan Pertanian Vol. 6 No. 3: 249-254. Retrieved from https://journal.ipb.ac.id/index.php/jtep/article/view/25524/16574

Cevallos, P. A. P, M. P. Buera, and B. E. Elizalde. (2010). Encapsulation of Cinnamon and Thyme Essential Oils Components (Cinnamaldehyde and Thymol) in β-Cyclodextrin: Effect of Interaction with Water on Complex Stability. Journal of Food Engineering Vol. 99: 70-75. Retrieved from https://www.sciencedirect.com/science/article/abs/pii/S0260877410000634

Crini, G., S. Fourmentin, E. Fenyvesi, G. Torri, M. Fourmentin, and N. Morin-Crini. (2018). Cyclodextrin, From Molecules to Applications. Environmental Chemistry Letters Vol. 16: 1361–1375. Retrieved from https://link.springer.com/article/10.1007/s10311-018-0763-2

Dadi, D.B., S.A., Emire, A.D.Hagos, and J.B.Eun. (2020). "Physical and Functional Properties, Digestibility, and Storage Stability of Spray- and Freeze-Dried Microencapsulated

Bioactive Products from *Moringa stenopelata* Leaves Extract. Industrial Crops and Products Vol. 156: 1-10. Retrieved from https://www.sciencedirect.com/science/article/abs/pii/S0926669020308086

Darniadi, S., I. Ifie, P. Luna, P. Ho, and B.S. Muray. (2020). "Foam-Mat-Dryig of Blueberry Juice by Using Trehalose-β-Lactoglobulin and Trehalose-Bovine Serum Albumin as Matrices. Food and Bioprocess Technology: 1-10." Retrieved from https://link.springer.com/article/10.1007/s11947-020-02445-6

Duchene, D. & A. Bochot. (2016). Thirty Years With Cyclodextrin. International Journal of Pharmaceutics Vol. 514 No. 1: 58-72. Retrieved from https://www.sciencedirect.com/science/article/abs/pii/S0378517316306603

Ezhilarasi, P. N., D. Indrani, B. S. Jena, and C. Anandharamakrishnan. (2013). Freeze Drying Technique for Microencapsulation of Garcinia Fruit Extract and Its Effect on Bread Quality. Journal of Food Engineering Vol. 117: 513-520. Retrieved from https://www.sciencedirect.com/science/article/abs/pii/S0260877413000228

Franco, T.S., C.A. Perussello, L.N. Ellendersen, and M.L. Masson. (2016). "Effects of Foam Mat Drying on Physicochemical and Microstructural Properties of Yacon Juice Powder. LWT – Food Science and Technology Vol. 66: 503-513." Retrieved from https://www.sciencedirect.com/science/article/abs/pii/S0023643815303005

Hadian, Z., M. Maleki, K. Abdi, F. Atyabi, A. Mohammadi, and R. Khaksar. (2018). Preparation and Characterization of Nanoparticle β-Cyclodextrin: Geraniol Inclusion Complexes. Iranian Journal of Pharmaceutical Research Vol. 17 No. 1: 39-51. Retrieved from https://www.researchgate.net/publication/323186482_Preparation_and_Characterization_of_Nanoparticle_b-CyclodextrinGeraniol_Inclusion_Complexes

Hill, L. E., C. Gomes, and T. M. Taylor. (2013). Characterization of Beta-cyclodextrin Inclusion Complex Containing Essential Oils (trnas-cinnamaldehyde, eugenol, connamon bark, and clove bud extracts) for Antimicrobial Delivery Applications. LWT – Food Science and Technology Vol. 51: 86-93. Retrieved from https://www.sciencedirect.com/science/article/abs/pii/S0023643812004598

Huang, Y., M. Liang, L. Sun, C. S. Brennan, and D. Liu. (2020). Effect of Microencapsulation on Morphology, Physicochemical Properties and Flavour Profiles of Solid Yoghurt-Flavoured Bases. International Journal of Food Science and Technology. Retrieved from https://ifst.onlinelibrary.wiley.com/doi/abs/10.1111/ijfs.14896

Iqbal, M. J., A. Abbas, H. Rafique, M. F. Nawaz, A. Rasool. (2018). "A Review Paper on Foam Mat Drying of Fruits and Vegetables to Develop Powders. MOJ Food Processing & Technology Vol 6 No. 6: 465-467." Retrieved from

https://www.researchgate.net/profile/Muhammad-Iqbal-191/publication/329701165_A_review_paper_on_foam-mat_drying_of_fruits_and_vegetables_to_develop_powders/links/5c1653bd4585157ac1c69344/A-review-paper-on-foam-mat-drying-of-fruits-and-vegetables-to-develop-powders.pdf

Jayanudin, Rochmadi, M. K. Renaldi, dan Pangihutan. (2017). The Influence Of Coating Material On Encapsulation Efficiency of Red Ginger Oleoresin. Jurnal Penelitian Kimia, Vol. 13 No. 2: 275-287. Retrieved from https://www.researchgate.net/profile/Jayanudin-Jayanudin/publication/325573589_THE_INFLUENCE_OF_COATING_MATERIAL_DIFFERENCE_AGAINST_ENCAPSULATION_EFFICIENCY_OF_RED_GINGER_OLEORESIN/links/5b441ea9a6fdcc66191406bf/THE-INFLUENCE-OF-COATING-MATERIAL-DIFFERENCE-AGAINST-ENCAPSULATION-EFFICIENCY-OF-RED-GINGER-OLEORESIN.pdf

Kanha, N., J.M. Regenstein, and T. Laokuldilok. (2020). "Optimization of Process Parameters for Foam Mat Drying of Black Rice Bran Anthocyanin and Comparison with Spray- and Freeze Dried Powders. Drying Technology: 1-15." Retrieved from https://www.tandfonline.com/doi/abs/10.1080/07373937.2020.1819824

Karadag, A., B. Ozcelik, & S. Saner. (2009). Review of Methods to Determine Antioxidant Capacities. Food Anal. Methods Vol 2 No. 1: 41–60. Retrieved from https://www.researchgate.net/publication/225182465_Review_of_Methods_to_Determine_Antioxidant_Capacities

Kaushik, V. & Y. H. Roos. (2007). Limonene Encapsulation in Freeze-Drying of Gum Arabic-Sucrose-Gelatin Systems. LWT Vol. 40 No. 8: 1381-1391. Retrieved from https://www.sciencedirect.com/science/article/abs/pii/S0023643806002829

Kfoury, M., L. Auezova, S. Ruellan, H. Greige-Gerges, and S. Fourmentin. (2015). Complexation of Estragole As Pure Compound and As Main Component of Basil and Tarragon Essential Oils With Cyclodextrin. Carbohydrate Polymers Vol. 118: 156-164. Retrieved from https://www.sciencedirect.com/science/article/abs/pii/S0144861714011187

Kumaravelu, P., Subramaniyam, S., Dakshinamoorthy, D. P., & Devaraj, N. S. (1996). "The Antioxidant Effect of Eugenol on CCl4-induced Erythrocyte Damage in Rats. The Journal of Nutritional Biochemistry Vol. 7 No. 1: 23–28. Retrieved from https://www.sciencedirect.com/science/article/abs/pii/095528639500162X

Layuk, P., M. Lintang, dan H. J. Motulo. (2018). Effect of Coating Materials on Encapsulation Quality of Nutmeg Essential Oil. Repositori Publikasi Kementrian Pertanian. Retrieved from

http://repository.pertanian.go.id/bitstream/handle/123456789/8863/PENGARUH%20BAHAN%20PENYALUT%20TERHADAP%20KUALITAS%20ENKAPSULASI%20MINYAK%20ATSIRI%20PALA%20-%20Payung%20Layuk%2c%20M.%20Lintang%20dan%20H.J.%20Motulo.pdf?sequence=1&isAllowed=y

Maesaroh, K., D. Kurnia, dan J. A. Anshori. (2018). Comparison of Antioxidant Activity Test Methods of DPPH, FRAP, and FIC against Ascorbic Acid, Gallic Acid and Quercetin. Chimica et Natura Acta Vol. 6 No. 2: 93-100. Retrieved from http://jurnal.unpad.ac.id/jcena/article/view/19049/8961

Mahmudah, Nur Laila. (2015). Encapsulated Rose Oil (Rosa damascenaMill.) With Acetylated β-Cyclodextrin and β-Cyclodextrin Coatings. Semarang: Universitas Negeri Semarang. http://lib.unnes.ac.id/21964/1/4311411041-s.pdf

Muchtadi, T. R., A. N. Ilma, D. Hunaefi, dan S. Yuliani. (2015). Homogenization Technique And Preliminary Study Of Scaling Up Microencapsulation Of Palm Oil. Jurnal Teknologi Industri Pertanian Vol. 25 No. 3: 248-259. Retrieved from https://journal.ipb.ac.id/index.php/jurnaltin/article/view/11792

Nurdjannah, Nannan. (2007). Nutmeg Processing Technology. Jakarta: Badan Penelitian dan Pengembangan Pertanian. Retrieved from https://d1wqtxts1xzle7.cloudfront.net/38888733/HPSP-12-Ollop-Teknologi_Pengolahan_Pala-with-cover-page-v2.pdf?Expires=1633610809&Signature=TmEjP8N~H5vmQNVmmafdCKQIsX~NwVGOZxUxU4TE2IVsB4J45rS~LPPTMtuZ13H6bxF7C74Mo4p~26oZ7VZTGARbUvU-r1Qx5sRedc1rV4W7~wNgiL9GgGJppZ-UjF-hClqYzVBtFvwjtMKhEz1jwlFph14mt6LZ3EuQY7~RNHVvOjHQ4RQB8CUvfobOt1jy073mEGMU3ba1KHBI-0-VHRHuCHF5gfUZZ7VArjBQoEvUatwLAzt6roZOa9QJbr1i0DEfgYz~~HqS7OtWvMbqME3DdD9QZ~c3kSOwy1FBQbi1LjRra0H9dbBpCsqDUD-xSatfoPd-nl~QlUQDU6aiAg__&Key-Pair-Id=APKAJLOHF5GGSLRBV4ZA

Nuryati, Leli & Akbar Yasin. (2016). Nutmeg Outlook: Agricultural Commodities Plantation Subsector. Jakarta: Kementerian Pertanian. Retrieved from http://epublikasi.setjen.pertanian.go.id/epublikasi/outlook/2016/Perkebunan/OUTLOOK%20PALA%202016/files/assets/common/downloads/OUTLOOK%20PALA%202016.pdf

Pinto, M.R.M.R., D.A. Paula, A.I. Alves, M.Z. Rodrigues, E.N.R. Vieira, E.A.F. Fontes, and A.M. Ramos. (2018). "Encapsulation of Carotenoid Extracts from Pequi (*Caryocar brasiliense* Camb) by Emulsification (O/W) and Foam Mat Drying. Powder Technology Vol. 339: 10-38. Retrieved from https://www.sciencedirect.com/science/article/abs/pii/S0032591018307149

Qadri, O. S., A. K. Srivastava, and B. Yousuf. (2019). Trends in Foam Mat Drying of Foods: Special Emphasis on Hybrid Foam Mat Drying Technology. Critical Review in Food Science and Nutrition Vol. 60 No. 10: 1-10. Retrieved from https://www.tandfonline.com/doi/abs/10.1080/10408398.2019.1588221

Rahardjo, Evita Nia. (2019). Optimization of Temperature, Time, and Redudancy on The Ultrasound-Assisted Extraction of Nutmeg Oleoresin (Myristica fragrans) Using Ethanol Solvent. Semarang: Universitas Katolik Soegijapranata. http://repository.unika.ac.id/21545/

Rezvankhah, A., Z. Emam-Djomeh, and G. Askari. (2019). Encapsulation and Delivery of Bioactive Compounds Using Spray and Freeze-Drying Techniques: A Review. Drying Technology: An International Journal Vol. 38 No. 1-2: 1-25. Retrieved from https://www.tandfonline.com/doi/abs/10.1080/07373937.2019.1653906

Rodianawati, I., P. Hastuti, dan M. N. Cahyanto. (2015). "Nutmeg's (Myristica fragrans Houtt) Oleoresin: Effect of Heating to Chemical Compositions and Antifungal Properties. Procedia Food Science Vol 3: 244-254. Retrieved from https://core.ac.uk/download/pdf/82430861.pdf

Sangamithra, A., S. Venkatachalam, S. G. John, and K. Kuppuswamy. (2014). Foam Mat Drying of Food Materials: A Review. Journal of Food Processing and Preservation Vol. 39 No. 6: 3165-3174. Retrieved from https://ifst.onlinelibrary.wiley.com/doi/abs/10.1111/jfpp.12421

Santoso, Birgitta Devina. (2020). Optimization of The Amount Variation of Maltodextrin, Whey Protein Isolate, and Stirring Speed Based On To Physicochemical Characteristics of Encapsulated Nutmeg Oleoresin (Myristica fragrans) Using Vacuum Drying Method. Semarang: Unika Soegijapranata. Retrieved from http://repository.unika.ac.id/22995/

Sembiring, Bagem B.R., Supriadi, and Ediningsih. (2020). Effectiveness of Drying Methods to Minimize Aflatoxin of Dried Nutmeg. Jurnal Litri Vol.26 (1): 1 – 10. Retrieved from http://ejurnal.litbang.pertanian.go.id/index.php/jptip/article/download/10233/8973

Shan, B., Cai, Y. Z., Sun, M. and Corke, H. (2005). "Antioxidant Capacity of 26 Spice Extracts and Characterization of Their Phenolic Constituents. Journal of Agricultural and Food Chemistry Vol. 53 No. 20: 7749–7759." Retrieved from https://pubmed.ncbi.nlm.nih.gov/16190627/

SNI 01-3709-1995. (1995). Rempah-Rempah Bubuk. Badan Standardisasi Nasional Indonesia. Retrieved from http://repository.unika.ac.id/6790/8/03.70.0020%20Arief%20Nugroho%20Suanto%20LAMPIRAN.pdf

Tan, K. P., H. E. Khoo, & A. Azrina. (2013). Comparison of Antioxidant Components and Antioxidant Capacity in Different Parts of Nutmeg (Myristica fragrans). International Food Research Journal Vol. 20 No. 3: 1049-1052. Retrieved from http://www.ifrj.upm.edu.my/20%20(03)%202013/3%20IFRJ%2020%20(03)%202013%20Azrina%20(381).pdf

Vikas, Y. K. Sandeep, D. Braham, C. Manjusha, and V. Budhwar. (2018). Cyclodextrin Complexes: An Approach to Improve the Physicochemical Properties of Drugs and Applications of Cyclodextrin Complexes. Asian journal of Pharmaceutics Vol. 2 No. 2: 394-409. Retrieved from http://asiapharmaceutics.info/index.php/ajp/article/view/2367

Yonata, Diode. (2020). Microencapsulation Studies In The Process Of Making Seaweed Flavoring: Effect of Seaweed Types and Coating Materials on Physical and Chemical Characteristics Flavoring. Master Thesis Fakultas Teknologi Pertanian Unika Soegijapranata. http://repository.unika.ac.id/23404/