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#### MICROENCAPSULATION OF NUTMEG ESSENTIAL OIL WITH VACUUM DRYING

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#### **Abstract**

Nutmeg (MyristicafragransHoutt) is a native spice from Indonesia that has a distinctive aroma. The aroma of nutmeg seeds come from the content of oleoresin called myristicin, which contain as much as 0.5-13.5%. In the food processing, nutmeg's oleoresin are often added as a flavor. The extraction method and the type of solvent will also affect the amount of oleoresin that can be extracted. Myristicin is easily damaged by heat, bacteria, yeast, and fungi. One attempt to maintain the shelf life of this product is by microencapsulation. The aim of this study was to determine the physicochemical characteristic microencapsulated nutmeg oleoresin and nutmeg butter by vacuum drying process. Analysis of product parameters were conducted, i.e. surface oil, trapped oil, antioxidant activity and water activity. The method used in this study is vacuum drying with 60°C temperature and 0.05 MPA pressure for 24 hours. The principle of vacuum dryer is to make the product dry at a low temperature with vacuum pressure. The results of the study showed that surface oil test, trapped oil, antioxidant activity and water activity in ethanol solvent are 0,002 grams; 0,02 grams; 8,12%; 0,437 and the results of hexane solvent are 0,113 grams; 0,056 grams; 61,39%; 0,472. According to this study, the best microencapsulation is obtained by using hexane solvent.

Keywords: nutmeg, oleoresin, microencapsulation, vacuum drying

#### Introduction

Nutmeg (*Myristicafragrans*Houtt) is native spices of Indonesia from Maluku and Banda islands. Nutmeg has a distinctive aroma, useful, and has a high selling value (Atmaja *et al.*, 2017). The aromatic compounds of nutmeg are derived from myristicin and elimicin (Rahadian, 2009).

Nutmeg can be processed into essential oils, butter, and oleoresin. Oleoresin is a thick liquid extracted from spices using organic solvents. Oleoresin is composed of essential oils, resins and desired special components. Commonly used solvents in nutmeg extraction are ethanol and hexane. The solvent is widely used because it is not dangerous, non-toxic, has a boiling point that is not too high or low, and has a high solubility so that it can produce high amounts of yield. Extraction with hexane solvent (non-polar) will produce oleoresin with a high fat content and has solid form at room temperature. Extraction with ethanol solvent (polar) will produce oleoresin with a low fat content and has liquid form at room temperature (Suhirman&Balittro, 2013).

Oleoresin and nutmeg butter easily evaporate and have rancid properties at room temperature, so microencapsulation methods are needed to increase the shelf life and selling value of the product. Microencapsulation is the process of coating a solid, liquid, or gas material into microscopic particles with an appropriate coating material. Microencapsulation can increase the shelf life of oleoresin and nutmeg butter, because it is able to keep the active substance in the preparation for a long period of storage (Wahyuni, et al., 2015). The coating material that commonly used is maltodextrin. Maltodextrin is a coating and weight enhancer material, that able to hold water, increase viscosity and texture, without adding sweetness to the product (Ramadhani, et al., 2016).

Vacuum drying method is a drying method by reducing the water content of a product, using a low and constant temperature (Margana&Doni, 2017). Drying with vacuum method is able to produce high yields of product, due to radiant heat transfer which causes water vapor in the vacuum to condense without convection heat transfer. The combination of temperature and vacuum pressure that used must be directly proportional, because it is able to influence the water content of the product (Rifan, et al., 2017).

In the food industry, microencapsulated nutmeg oleoresin and nutmeg buttercan be used as flavor enhancers in bread products, cakes, candy, biscuits, and powder drinks. The aim of this study was to determine the best microencapsulation method to produce a good quality of microencapsulated nutmeg oleoresin and nutmeg butter with analysis of product parameters were conducted, i.e. surface oil, trapped oil, antioxidant activity and water activity.

#### **MATERIALS AND METHODS**

#### 2.1 Materials

The equipment used for the microencapsulated nutmeg essential oil are analytic scales, spoon, measuring cup, mixer, glass tray, blender, mesh 60, vacuum oven, Chromameter CR-400, moisture balance, Aw meters, vortex, spectrophotometer, soxhlet, oven binder, desiccator, and centrifuge. The main materials in this study were nutmeg oleoresin, nutmeg butter, maltodextrin, and water.

#### 2.2 Methods

#### 2.2.1 Microencapsulation

Microencapsulated nutmeg essential oil was madeby using the method of Ekponget al., (2016) with modification. To compare the best microcapsules between nutmeg oleoresin and butter, the formula for microencapsulationis the same. Microencapsulated nutmeg essential oil using a coating material is maltodextrin, with the ratio of nutmeg essential oil: maltodextrin: water is 3: 20: 14. The mixture nutmeg essential oil and maltodextrin was gently spread onto a glass trayand dried in oven vacuum at 60°C for 24 hours, or until the final moisture content of the mixture was less than 10%. The dried mixture was converted to powder using a blender and sieved with mesh 60, then packed in a plastic bag until further analysis.

#### 2.3. Analysis

#### 2.3.1 Color Analysis

Color analysis done using MINOLTA Chromameter 400 series (CR-400). Chromameter calibrated in advance by firing on a white plate. Once calibrated, chromameter is used to measure the color samples. Microencapsulated nutmeg essential oil is placed in the clear plastic then measured with a chromameter. Units of color emerges is L\*, a\*, dan b\*. L value indicates the level of 0 means the absolute black and 100 means white. The value of b is one attribute that indicates with a scale (-70) to 70. The value of b is negative indicates the degree of bluish while the positive values of b indicates the degree of yellowish. The value of a shows a reddish or greenish sample (Pomeranz &Meloan, 1987).

#### 2.3.2 Water Activity

Water activity (Aw point) becoming one of parameters in the analysis of product stability. Measuring water activity is very important to predict and knowing the existence of microorganisms that usually can spoilage on product. Water activity analysis done using Aw meters. Microencapsulated nutmeg essential oil were put into clean and dry containers. Samples are put in

about half of the Aw meter container (approximately 15 grams). Then the sample is measured with Aw meter for 15 minutes and the results obtained are recorded.

#### 2.3.3 Surface Oil Analysis

Microencapsulated samples about 2 gramsplaced in a centrifuge tube and dissolved in 10 ml of. Then centrifuged at 1700 rpm for 15 minutes. Furthermore, the sample is filtered with filter paper and then washed with 15 ml of hexane for 2 times. The filtrate obtained was displaced in a porcelain cup of known weight and then dried in oven binder for 24 hours. After that, the porcelain cup were put into the desiccator for 15 minutes then weighed as the final weight.

Surface oil yield = the final weight of the porcelain cup - the weight of the empty porcelain cup

#### 2.3.4 Trapped Oil Analysis

Microencapsulatedsamplesabout 2 grams and then wrapped in filter paper 2 times. The Soxhlet is filled with hexane up to half a tube. The sample is put into Soxhlet and then switch on at number 1 for 3 hours. Make sure the hexane in the tube does not run out. After that, the filtrate obtained was displaced in a porcelain cup of known weight and dried in oven binder for 24 hours. After that the porcelain cupwere put in a desiccator for 15 minutes then weighed as the final weight.

Total oil = the final weight of the porcelain cup - the weight of the empty porcelain cup

Trapped oil = total oil - surface oil

#### 2.3.5 Antioxidant Activity Analysis

Microencapsulatedsamples about 0,5 grams extracted with 5 ml of methanol for 2 hours in the dark room. 1,1 ml of extract were taken and treated with 3,9 ml of DPPH solution made by dissolving 2,4 mg of DPPH in 100 ml of methanol. Samples were incubated in the dark for 30 minutes and absorbance was measured at a wavelength of 517 nm. As a control, use 0,1 ml of methanol were reacted with 3,9 DPPH solution and methanol used as a blank solution. Antioxidant activity is expressed as %inhibition with the following equation:

%antioxidant activity =  $\frac{absorbance\ control-absorbance\ of\ the\ sample}{absorbance\ control} x100\%$ 

(Williams et al., 1995)

#### **RESULTS AND DISCUSSION**

#### 3.1 The Color Analysis of Microencapsulated Nutmeg Essential Oil

Table 1. The Color Analysis of Microencapsulated Nutmeg Essential Oil

<u> </u>			
Sample -	Color		
	L*	a*	b*
Malto_Oleoresin	83.39	0.86	6.63
Malto_Butter	95.18	-0.64	3.69

Microencapsulated nutmeg butter has a lighter color than microencapsulated nutmeg oleoresin, with a greenish yellow color. Where the L\* notation indicates the lighting value with susceptibility from 0 (dark) to 100 (bright). The a\* negative notation shows green, while the a\* positive notation shows red. The b\* negative notation shows blue and the b\* positive notation shows yellow. Color is an important parameter in determining food quality. In addition, color is able to influence the consumer acceptance of a product (Mella, 2016).

#### 3.2 Water Activity of Microencapsulated Nutmeg Essential Oil

Table 2. Water Activity of Microencapsulated Nutmeg Essential Oil

Sample	Aw point
Malto_Oleoresin	0.437
Malto_Butter	0.472

Microencapsulated nutmeg oleoresin has a lower AW value than microencapsulatednutmeg butter, although in susceptible that is not too far. Water activity (AW) shows the amount of free water in food, that used by microbes to grow. High AW value allows microbial growth and the shelf life of the product becomes shorter. Thus, the higher the AW value of a product, the lower shelf life of the product due to microbial growth factors (Ariani, *et al.*, 2016).

#### 3.3 Surface Oil of Microencapsulated Nutmeg Essential Oil

Surface oil shows the amount of oil that is on the surface of the microcapsules, which will affect the stability of the active compound from the essential oil during the storage process. The more surface oil values, the lower the encapsulation efficiency value. So that the amount of active ingredients wrapped is reduced because a lot of oil is attached to the surface. As a result, the oxidative stability of the microcapsules will be damaged (Pourashouri *et al.*, 2014).

Table 3. Surface Oil of Microencapsulated Nutmeg Essential Oil

Sample	Surface Oil (grams)
Malto_Oleoresin	0.002
Malto_Butter	0.113

Microencapsulated nutmeg oleoresin produced surface oil values that are smaller than microencapsulated nutmeg butter with the same coating material and concentration, is maltodextrin with a comparison of samples: coating is 3:20. Microencapsulation process using a coating material will increase the viscosity which will give the thickness of the wall in encapsulates. The thickness of the encapsulated wall will reduce the amount of essential oil that will come out of the microcapsules during the hardening process of the microcapsule wall (Jayanudin et al., 2017). Uncapsulated butter or oleoresin will be more susceptible to damage, evaporation, and oxidation which will reduce the quality of microcapsules (Shahidi & Han, 1993). The results of

microencapsulated nutmeg oleoresin means that maltodextrin shows a better ability to stabilize emulsions in nutmeg oleoresin. The amount of maltodextrin used is sufficient to form a layer on the surface of oleoresin droplets.

#### 3.4 Trapped Oil of Microencapsulated Nutmeg Essential Oil

Table 4. Trapped Oil of Microencapsulated Nutmeg Essential Oil

Sample	Trapped Oil (grams)	
Malto_Oleoresin	0.020	
Malto_Butter	0.056	

Trapped oil is the amount of essential oil trapped inside a microcapsule. Microcapsule products with a high percentage of trapped oil show that much oil can be protected by coating agents (Djafar, 2019). The results of trapped oil in microencapsulated nutmeg essential oil are presented in Table 4. The results of microencapsulatednutmeg oleoresin show that the trapped oil is low indicating that the amount of nutmeg oil protected by the polymer is also low, so it can be concluded that the greater amount of trapped oil shows the amount of oil present on the surface to be decreasing.

#### 3.5 Antioxidant Activity of Microencapsulated Nutmeg Essential Oil

Table 5. Antioxidant Activity of Microencapsulated Nutmeg Essential Oil

Sample	Antioxidant Activity
Malto_Oleoresin	8.117%
Malto_Butter	61.385%

Microencapsulated nutmeg butter has a higher antioxidant activity than microencapsulated nutmeg oleoresin. Nutmeg seeds contain carotene and terpenoid compounds that have the potential as antioxidants and antibacterial. Antioxidants are compounds that can inhibit or slow down the oxidation process by free radicals. Terpenoid compounds are generally non-polar, so non-polar solvents are needed to extract these compounds (Ginting, *et al.*, 2017). Example of non-polar solvent that commonly used is hexane (Suhirman & Balittro, 2013). This is why the butter microencapsulation has higher antioxidant activity than oleoresin microencapsulation.

#### CONCLUSION

Microencapsulated nutmeg oleoresin has antioxidant activity 8,117%, trapped oil 0,02 grams, which surface oil is not too high about 0,002 grams, water activity 0,437, and the color is reddish yellow color. Microencapsulated nutmeg butter has antioxidant activity 61,385%, trapped oil 0,056 grams, which surface oil is not too high about 0,113 grams, water activity 0,472, and the color is greenish yellow color and almost close to white which when added to food and drinks will not affect the color. Depending on the analysis results, it could be concluded that the best microencapsulated nutmeg essential oil by the physicochemical characteristic was microencapsulated nutmeg butter.

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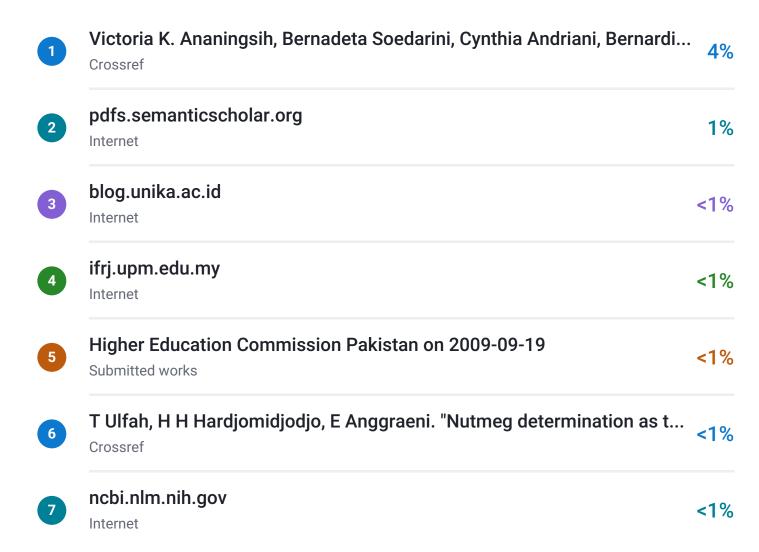
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