Physical characteristics of cinnamon oil microcapsule

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Abstract. Cinnamon (Cinnamomum burmanii) oil products can be obtained from the bark by steam distillation. Essential oils are susceptible to high temperatures, oxidation, UV light, and humidity. Microencapsulation may change essential oils into powder, protect the sensitive core material and reduce the amount of flavor which lost during storage. In the microencapsulation, one of the important factors is the type of coating agent. The objective of this work was to characterize the cinnamon oil microcapsule. Ratio variations of coating agent maltodextrin and gum arabic were (1:0); (0:1); (1:1); (2:3). Physical characteristics such as water content, solubility, bulk density, surface oil, and microencapsulation efficiency of samples were investigated. Results showed that the ratio variations of the coating agent significantly affected the water content, bulk density, surface oil and microencapsulation efficiency but significantly affected the water solubility. Characteristics of selected microcapsule were 6.13% water content; 96.33% solubility; 0.46 g/cm³ bulk density; 2.68% surface oil; 70.68% microencapsulation efficiency and microstructures were rather good.

1. Introduction

There are 54 species of cinnamon (Cinnamomum sp.) in the world, and 12 of them are located in Indonesia. Cinnamomum Burman Nii (from Indonesia) which also known as cassia vera, Cinnamomum zeylanicum (from Sri Lanka and Seychelles) and Cinnamomum cassia (from China) are types of cinnamon with the best demand in the world market. They are also the most famous spice plants. Cinnamomum Burman Nii type Blume or cassia vera is a traditional export product that is still controlled by Indonesia as the main exporting countries in the world [1]. In the international trade, cinnamon tree products are marketed in the form of bark, essential oils, and oleoresin. Volatile oil products can be obtained from the bark, twigs, branches, or leaves of the cinnamon tree by distillation. Steam distillation produced a higher yield. Previous research [2] reported that the yield of cinnamon essential oil by steam treatment was 0.66%.

Essential oil of cinnamon has a clear yellow to brownish color and cinnamaldehyde as the main component. Essential oil of cinnamon has been widely used as an ingredient in cosmetics, perfumes, foods and beverages flavor, used as antiseptic and antimicrobial. However, essential oils are susceptible to high temperature, oxidation, UV light, and humidity. Oxidative damage caused an unpleasant flavor and also
decreased both shelf life stability and sensory properties [3]. Microencapsulation is one of the techniques that can be used to protect the core material such as essential oil by changed the materials from a liquid into solid form. The products became easy to handle, and the core material can be protected from losing flavor [4].

In the microencapsulation process, coating agent affected the quality. Maltodextrin and gum arabic are a coating agents that often used in microencapsulation. Maltodextrin was able to protect the main compounds in ginger oil during the encapsulation process. Previous research reported the production of ginger oil microcapsule and ginger oleoresin microcapsule using maltodextrin as a coating agent. Sample with a ratio of essential oil and maltodextrin in 1:25 produced the best characteristics. However, due to low viscosity emulsion during the spray drying process made the skin layer weak and caused the core material to be less protected [5].

Gum arabic has unique properties compared to the other types of gum. With a concentration of 40-50%, gum arabic does not produce too high viscosity and can create a perfect stability. Gum arabic is an effective emulsifying agent due to protective colloid ability [6]. In soybean oil emulsification and microencapsulation using gum arabic study [7], the particle size ranged from 9-17 μm and the microencapsulation efficiency decreased from 100 to 48% by the addition of oil. Essential oil addition less than 50% would produce a good solubility in water powder. Maltodextrin and gum arabic in red palm oil microcapsules showed the best formulation with a ratio of maltodextrin and gum arabic and oil in 2:3:2. The water content of microcapsule was 0.92%, carotenoids 82.63 ppm and solubility in water 66.82% [8]. Therefore, a combination of coating agents such as maltodextrin and gum arabic could produce microcapsule with good characteristics. The objective of this work was to characterize the cinnamon oil microcapsule that contained maltodextrin and gum arabic as coating agents.

2. Method

2.1 Essential Cinnamon Oil Preparation
The bark of a cinnamon tree (Cinnamomum Burman Nii) from Bubakan, Girimarto, Wonogiri (Indonesia) was air dried for 5 days until the water content decreased less than 12%. Dried cinnamon bark was milled and sieved 10 mesh. The powder cinnamon was steam distillation for 4 hours.

2.2. Microemulsion
The microemulsion is made with a ratio of essential oils and coating as 1:25. The formula is then dissolved in distilled water with a ratio of coating material and distilled water as 1:20. Meanwhile, the variation ratio of coating material maltodextrin (DE 10-15 with Associated British Budi brand from Bratachem Chemical Store Yogyakarta) and gum arabic (powder Type 4687 from Bratachem Chemical Store Surakarta) is (1:0); (0:1); (1:1) and (2:3). Homogenization process is done using a homogenizer (Ultra-Turax) for 15 minutes at 8000 rpm.

2.3. Microencapsulation by spray drying
Microencapsulation is done by a spray dryer (Spray Dryer, Basic Lab Plant SD) with an inlet temperature of 109°C.

2.4. Emulsion characterization
2.4.1. Emulsion stability
Immediately after the emulsion preparation, 100 mL of samples were transferred gradually to 100 ml cylinders, sealed, stored at room temperature for 24 hours. After 24 hours, the volume of the upper phase measured. The stability was measured by % of separation and expressed as:
% Separation = \[\frac{H1}{H0} \times 100\%\] where Ho represents the initial emulsion height and H1 is the phase upper height.

2.4.2. Emulsion droplet size
The droplet size distribution was measured using a microscope optic lab viewer. A small sample was added in the preparation and observed under the objective lens.

2.5. Powders analysis
2.5.1. Moisture Content
Analysis of water content was conducted using a thermogravimetric method by weighing the sample in the form of powder / material which has been refined as much as 1-2 grams in the weighing bottle. Then, dried the refined material in an oven with a temperature of 100-105°C for 3-5 hours, cooled in a desiccator and weighed. Pre-heat oven for 30 minutes longer cooled and weighed. This treatment was repeated until a constant weight [9].

2.5.2 Solubility
Solubility measurement was calculated based on the weight percentage of residues that could not go through a filter paper to the weight of sample used. Solubility analysis was performed by gravimetric method. Samples (0.75 g) was diluted in 100 ml of distilled water and filtered by vacuum filtration. Before the next step, the filter papers were dried in a 105°C oven for 30 minutes and weighed. After the filtration process, the filter papers along with the residues were dried in an oven at 105°C for approximately 3 hours, cooled in a desiccator for 15 minutes and weighed [10].

2.5.3. Surface oil and microencapsulation efficiency
Surface oil was calculated using the gravimetric method. Fifteen milliliters of hexane was added to 2 g of powder in a test tube, processed in the vortex for 2 minutes at room temperature. The mixture was filtered using Whatman No 1 filter paper and washed 3 times using 20 ml of hexane. After filtering and washing, both residue and filter paper were dried at 60°C until constant weight. The content of surface oil was calculated as a weight percentage difference of the powder, before and after the extraction and washing process. Microencapsulation efficiency (ME) was calculated using the formula: ME = \[\frac{(Total\ oil - Surface\ oil)}{Total\ oil} \times 100\%\] [11].

2.5.4. Bulk density
Bulk density was determined by the method of tapping. 2 grams of powder was put in a 10 ml cylinder. A cylinder containing the powder knocked using a tap density to a constant volume. Bulk density was calculated by dividing the sample weight by volume [12].

2.5.5. Analysis by SEM
Microstructural observations on microcapsules made with SEM (JEOL JSM-type T-300). Samples were prepared by attaching little microcapsules on a specimen that has been coated with insulating conductive. Furthermore, the specimen coated with gold as filling material by Fine Coat Ion Sputter JFC models 1100. After sampling, the gold coated specimen was included in the column injector to be fired with electrons. The electron will be reflected back by the sample and is referred to as secondary electron. The secondary electron detector will show in the form of images [13].
2.6. **Statistical Analysis**

SPSS (SPSS 16.0 for Windows) statistical software was used to calculate the analysis of variance (ANOVA). Duncan Multiple Range Test (DMRT) was used for the comparison of means, with significance assigned at P<0.05.

3. **Results and discussion**

3.1. **Emulsion characterization**

Emulsion stability was ranged from 98.18%-100%. The addition of gum arabic as coating material resulted in higher emulsion stability. Gum arabic is an effective emulsion thickening agent because of its ability to protect the colloid. This type of thickener is also heated resistant to the process that uses heat. Gum arabic has unique properties such as high solubility and low viscosity. The other characteristics of gum arabic were also the ability to form texture and film, binder ability and also as an emulsifying agent because of its protein component [14]. A mixture of gum arabic as an emulsifying agent in anise oleoresin showed the result that coating material emulsion provides the highest stability (90.12%) and a mixture of gum arabic maltodextrin coating material provides emulsion stability of 75.39% [15].

Microencapsulation is a process using a relatively thin coating on a small particles of solids or liquid droplets and the dispersion liquid, where the particle size ranged between 1-5000 μm. In the process of spray drying, the emulsion droplet size or diameter ranges between 1-100 μm [16].

![Figure 1. Cinnamon Essential Oil Emulsion (A) MD:GA 1:0, (B) MD:GA, (C) MD:GA 1:1, and (D) MD:GA 2:3](image)

The emulsion droplet size was ranging from 2.1 μm - 65.3 μm. The size of the microcapsules proved that the product will be made to microscopic size. Figure 1 showed that the core material essential oil of cinnamon is covered by the coating material. Homogenizing linseed oil with different wall materials can be combined to produce an emulsion with droplet diameter ranging from 0.6 to 26 μm [17].

3.2. **Powder characterization**

| Table 1. Physical Properties of Microcapsule |
|---------------------------------------------|
| Characteristic                              | Variation Ratio of Coating Agents (MD:GA) |
|                                             | (1:0) | (0:1) | (1:1) | (2:3) |
| Water Content (%)                           | 5.691±0.14<sup>a</sup> | 7.715±0.61<sup>b</sup> | 6.502±0.44<sup>a</sup> | 6.134±0.24<sup>a</sup> |
| Solubility (%)                              | 97.002±1.42<sup>a</sup> | 91.393±6.55<sup>a</sup> | 97.467±0.39<sup>a</sup> | 96.333±0.69<sup>a</sup> |
| Bulk density (g/ml)                         | 0.426±0.001<sup>a</sup> | 0.491±0.011<sup>c</sup> | 0.453±0.006<sup>b</sup> | 0.456±0.007<sup>b</sup> |
| Surface oil (%)                             | 4.262±0.03<sup>c</sup> | 4.099±0.12<sup>c</sup> | 3.447±0.39<sup>b</sup> | 2.675±0.15<sup>a</sup> |
| EM (%)                                      | 59.859±5.75<sup>a</sup> | 66.164±2.01<sup>a</sup> | 65.811±5.54<sup>a</sup> | 70.684±6.23<sup>a</sup> |
Water content is an important parameter that can affect the stability of the product during storage and distribution. The water content of the microcapsules was ranging between 5.691% - 7.715% (Table 1). The lowest water content (5.691%) was performed by the formula with a coating ratio of (MD: GA) (1:0), and the highest (7.715%) was showed by the formula with a coating ratio of (MD: GA) (0:1). Those range of water content was a typical water content of the microcapsule products obtained from spray drying (2-6%) [18]. Microcapsules analysis with the same coating material showed a range of 5.9-8.1% [19]. The result indicated that the water content of cinnamon essential oil microcapsules was still in accordance with the typical water content in microcapsule products.

The addition of maltodextrin can reduce water content because maltodextrin has a low viscosity that made the solution could heat at high concentration, so water less absorbed and microcapsule were easily dried [20]. High viscosity indicates that the water trapped in the structure were high, so it will be difficult to be separated during the drying process. The increasing of maltodextrin has been used and the decreasing gum arabic has been used, decreased the viscosity. It can also be explained that if less water trapped inside the structure, it means that the water content was low. Previous research [8] reported that red palm oil microcapsule with the lowest water content (0.62%) was showed by the formula with a coating ratio of (MD: GA) and oil in (3:3):2.

Solubility in water of microcapsule with gum arabic and maltodextrin as the coating material ranged from 94-98% [19]. The solubility value is determined by the ability of the coating material to dissolve water. The solubility of cinnamon essential oil microcapsules ranged from 91.393% - 97.467% (Table 1). The highest solubility of MD: GA (1:1) coating material was equal to 97.467%. Maltodextrin is soluble in cold water perfectly so that it can release the flavor rapidly in certain applications [21]. Gum arabic also has a high solubility in water. So, a combination of both can produce microcapsule with high solubility in water [22]. The high solubility of maltodextrin will increase the solubility of microcapsule [23]. Compared to gum arabic that will form an irregular pattern of oil droplets that can affect the solubility, maltodextrin can improve solubility because it is more soluble in water. The formula of (MD:GA): Oil in (2:3):2 has a higher solubility (66.82%) due to the amount of maltodextrin (MD:GA):Oil(1:3):1. Besides, the size of droplet relatively more uniform or more stable when compared with the other formula, which is (MD: GA): Oil (3:3): 2 [8].

Bulk density analysis in the food industry is usually performed to help determine the capacity of the powder in packaging, storage and distribution process. Bulk density is the mass of the particle that is measured from a container per unit volume [23]. In this study, powder density ranged from 0.426 g/cm³ - 0.491 g/cm³ and increased gradually in line with the decline of maltodextrin (Table 1). Microcapsules with a coating ratio of MD: GA (1:0) showed the smallest bulk density (0.426 g/cm³) while the other values are MD:GA (1:1) 0.453 g/cm³; MD:GA (2:3) 0.456 g/cm³. The largest bulk density (0.491 g/cm³) performed by MD: GA (0:1). Small particle size would form a larger mass density as a result of a reduction in the cavities between the particles. High water levels will increase the weight of the material in the same container volume, and cause the increasing of bulk density [24].

Surface oil content is observed from the outer surface of the microcapsule wall. The existence of this oil is undesirable because it comes in contact with the surrounding environmental conditions so that it can be damaged. The percentage of surface oil is calculated by comparing the oil that is found in the surface with the oil that is purposely added so that it can also be seen from the total oil content. The result showed that surface oil of microcapsules ranged from 2.675% - 4.262% (Table 1). The high addition of maltodextrin tends to increase the amount of oil encapsulated. Increasing the number of maltodextrins (a decrease in the amount of gum arabic) will increase the unencapsulated oil content. Maltodextrin is lipophobic so that it cannot bond with the oil molecules. In addition, the decreasing of gum arabic also reduced the oil coating ability of gum arabic as an emulsifier. Maltodextrin does not have lipophilic properties. Therefore, maltodextrin on lipid encapsulation by spray dryer method caused the
emulsion stability and low oil retention, but the oil encapsulated has a resistance to oxidation [25]. Microcapsules with coating material combination of MD: GA (2:3) showed the lowest value of surface oil (2.675%). The higher oil on the surface (2.2%) resulted by GA compared to the combination of MD /GA (1.9%) [26].

Microencapsulation efficiency of cinnamon essential oil ranged between 59.841%-70.702% (Table 1). The highest efficiency in microcapsules was reached by a combination of coating material MD:GA (2:3). In the case of flavor encapsulation by spray drying, the retention of flavor depends on the ratio between maltodextrin and gum acacia with optimal retention at a ratio of 2/3 and 3/2 [27]. Besides, another study [26] showed that the GA coating material showed 90.5% efficiency while the coating material MD/GA generated microencapsulation efficiency of 91.4%. Increasing total content of maltodextrin in the emulsion coating material caused the decreasing of surface oil and increasing of microencapsulation efficiency although a recent study reported that increasing concentrations of total solids had little effect on the efficiency of microencapsulated powder [28]. Microcapsules with coating material MD:GA (1:0) showed the lowest microencapsulation efficiency (59.841%). Using various concentrations of maltodextrin and gum acacia in the microencapsulation of ethyl butyrate with the spray drying method, proved that the increasing concentration of maltodextrin (10-30%) was accompanied by an increase in the efficiency of the microencapsulation process [28].

Figure 2. Scanning Electron Microscopy (SEM) analysis of cinnamon essential oil microcapsules (A) MD:GA (1:0) with 1000x magnification; (B) MD:GA (0:1) with 1000x magnification; (C) MD:GA (1:1) with 1000x magnification; (D) MD:GA (2:3) with 1000x magnification

Figure 2 showed that in terms of size, although the microcapsules are relatively larger in figure D, but there is no significant different between the size of A, B and C microcapsules. The size of cinnamon essential oil microcapsule ranged from 1.92 - 30.8 μm. Particles of good quality, which size are less than 40 μm [29]. Characteristics of good microcapsules were thin wall thickness, large particle size and high capable of carrying components [30]. Microcapsules processed from spray drying which used polysaccharide as the coating material showed a typical surface. Those because the effects of coating agents composition, the process of atomization and drying, irregular wrinkles at the start of the drying process, and the effect of surface tension of the solution. Development of air or moisture in the dried particles can not also be perforated. The effect of this development is commonly called ballooning. Ballooning will affect the quality of the microcapsules. Ballooning was heavily influenced by the speed of drying and viscoelastic properties of the matrix coating material. Ballooning can also occur due to too high drying process pressure, thus made the hydrophobic interaction or also called as hydrophilic occurs between the carrier amphipathic, water, and substances that are encapsulated so that it would make it into the micelle. The small droplets of micelles formed (average diameter of 100 m) making it easily dried, forming a hard coating around the encapsulated substances [31].
4. Conclusion
The selected essential oil microcapsule was determined by looking at the results of the physical characteristics analysis. In bulk density, the best result was obtained by microcapsules with the coating material of MD:GA (0:1). In surface oil, the best results are microcapsules with coating material variation of MD:GA (2:3). While in microstructural parameter, good results are reported by microcapsules with coating material variations of MD: GA (0:1) and MD:GA (2:3) which also formed a spherical microstructure somewhat wrinkled. By some parameters from physical characteristics analysis, microcapsules with coating material variations of MD:GA (0:1) and MD:GA (2:3) have the same trend. However, oil surface was more important parameter due to an undesirable high oil surface could contact with the surrounding environmental conditions that can damage the microcapsule quality. Sample with the lowest content of surface oil was microcapsule with a coating material variation of MD:GA (2:3). In conclusion, this formulation was chosen as the best microcapsule. Characteristics of selected microcapsule were 6.13% water content; 96.33% solubility; 0.46 g/cm³ bulk density; 2.68% surface oil; 70.68% microencapsulation efficiency and microstructures were rather good.

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