UTILIZATION OF ALGINATE AS AN ENCAPSULATION MODEL OF COCONUT SHELL LIQUID SMOKE

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ABSTRACT

Alginate extracted from brown seaweed has gelling properties that make it useful as a wall material in encapsulation systems. Liquid smoke contains the active substances, such as phenols, which can preserve food. In order to protect the active substances, liquid smoke is encapsulated by using alginate and maltodextrin. The purpose of this study was to investigate liquid smoke encapsulation technology with maltodextrin and alginate using a spray dryer, to improve the physical and chemical characteristics of the liquid smoke. The microcapsules of liquid smoke were made, using a spray dryer SD 04, by encapsulating liquid smoke with two types of wall materials, maltodextrin and the combination of alginatemaltodextrin. The ratio of liquid smoke to total solids (wall materials) was 9:1 (v/w). The alginate concentration used was 0.5 to 2% (w/v). Parameters observed in this study were phenol release, shape and morphology, encapsulant efficiency, drying yield, phenol marker and, particle size. This study used a completely randomized design with three replications. The best treatment was obtained by using the alginate with a concentration of 1% (w/v) and maltodextrin of 9% (w/v) with phenol release of 2.52% (w/w) in the 20 minute of release, encapsulant efficiency of 45.13% and drying yield of 28.74%. The particle size analyzer results showed that the particles were agglomerating. Scanning electronic microscope (SEM) observation illustrated that all treatments have a better capsule morphology than the controls, whereas Optilab image processing and analysis software results showed that phenolic compounds are encapsulated by wall materials used.

Keywords : alginate, maltodextrin, encapsulation

INTRODUCTION

Alginate has a linear polymeric structure consisting of two monomer units, β -D- mannuronic acid and α -L- guluronic acid (Pranoto, 2007). Alginate has the ability to form a viscous solution (Rasyid, 2003) and to form gel due to the protonation of carboxyl groups reducing electrostatic repulsion from carboxylate anions which will provide an opportunity for alginate molecules to interact through hydrogen bonding (Marseno, 1998). With its characteristic of forming gel, alginate can be used as an encapsulating material for active substances. The use of alginate as wall material in encapsulation is done by using a spray dryer (Kailsapathy, 2002). According to Cvitanović et al., (2011) alginate is used as a wall material because it has good biocompatibility properties so that the release of the active substances in the capsules can be controlled; it also has good and non-toxic biodegradability. Encapsulation is the process of trapping a substance in another substance (Zuidam and Shimoni, 2010) which can protect bioactive components from destructive changes and change it into the form of flour (Shaikh et al., 2006). Alginate with its ability to form a gel can build strong encapsulant walls to trap the active substances in liquid smoke.

Liquid smoke is a condensate liquid from smoke which has undergone sedimentation and filtration to separate the tar from particulate materials (Pszczola, 1995 in Sari et al., 2006), which is the result of pyrolysis and purification, a method of distillation from materials containing lignin, cellulose and hemicellulose. Liquid smoke can act as a food preservative due to its antibacterial and anti-fungal properties which can inhibit decay (Dwiyitno and Rudi, 2006), because it contains phenolic and acid compounds which play a major role in inhibiting the growth of bacteria and fungi (Dainius et al., 1979; Wendorff, 1981; Sofos et al., 1988; Swastawati and Darmanto, 2006 in Dwivitno and Rudi, 2006).

Liquid smoke encapsulation using maltodextrin has been performed by Saloko et al., (2012). But the use of maltodextrin as an encapsulating material is less effective, because it can result in caking (microcapsules sintering) and agglomeration of the product (Sansone et al., 2011), whereas the combination with alginate can better trap the active substances. The purpose of this study was to determine the effect of alginate addition on physical and chemical characteristics of the liquid smoke microcapsules.

MATERIALS AND METHODS

Materials

The materials used in this study were food grade sodium alginate (CV.Cehmix Pratama-Yogyakarta), Maltodextrin DE of 10.8% (CV.Cehmix Pratama-Yogyakarta), coconut shell liquid smoke produced through pyrolysis with two distillations.

Preparation of Liquid Smoke Microparticles

In this study, the liquid smoke undertaken encapsulation was using encapsulating 10% materials (w/v)maltodextrin (control), 9.5% (w/v)maltodextrin and 0.5% (w/v) alginate, 9%

(w/v) maltodextrin and 1% (w/v) alginate, 8.5% (w/v) maltodextrin and 1.5% (w/v) alginate, 8% (w/v) maltodextrin and 2% (w/v) alginate. Liquid smoke microparticles were made with 10% of the total weight of the solids in 250 ml of microparticle solution (liquid smoke with encapsulating materials). Each of encapsulating material treatments was added into a liquid smoke solution of 225 ml. The encapsulating materials mixture was homogenized with liquid smoke, this process used a stirrer for 30 minutes, heated in a water bath (shaking waterbath, Julobo SW 23) of 100 rpm up to a temperature of 45°C. The subsequent homogenization process used turrax (homogenizer ultra turrax T50) for 2 minutes at 4000 rpm. The homogenous microparticles solution was filtered using a vacuum filter with Whatman filter paper no.41.

Preparation of Liquid Smoke Microcapsules

The microparticles that were made were put into a spray dryer (spray dryer SD 04, nozzle 5mm) with an inlet temperature of $130^{\circ}C\pm1^{\circ}C$, an outlet temperature of $79^{\circ}C\pm1^{\circ}C$, flow rate of 5ml/min, pump of 7%, compressor of 60%. The microcapsules were collectedfrom the chamber of the dryer.

Chemical Analysis

The chemical analysis performed was phenol release referring to Cvitanović *et al.*, (2011) which has been modified, followed by phenol analysis referring to Senter *et al.*, (1989).

Morphological Examination and Physical Analysis

The morphology of the microcapsules was examined using a transmission electron microscope (TEM JEM-1400, Jeol, Japan) and scanning electron microscope (SEM FEI, Belanda). The microcapsules sizes formed can be determined by using a particle size analyzer.

A physical analysis in this study measured encapsulation efficiency as described by Cvitanović et al. (2011), drying



Fig.1. Liquid smoke microcapsules phenol release

yield referring to Sansone et al. (2011). And the last physical analysis was of phenol markers to ensure the phenol particles had been encapsulated referring to Mohrig et al., (2003). A phenol marker used FeCl₃ solution of 5% (w/v) with a ratio of 3 drops of $FeCl_3$ solution: 1 ml of liquid smoke. The FeCl₃ solution was added to liquid smoke, and then mixed with the encapsulating materials and homogenized, and examined under a microscope (Olympus CX21LED) with magnification up to 100x which has been connected to Optilab software (Advance, Indonesia) on the computer. If the particle was phenol, then it would be visible with a particular color as mentioned by Soloway et al. (1952).

RESULTS AND DISCUSSION

Phenol release

The phenol release analysis showed the percentage of active substances (phenol) released from the capsule. It was most important to determine the extent of any release of active substances from the encapsulation system, to ensure the active substances could still function. The phenol release with the addition of various concentrations of alginate can be seen in Figure 1, which showed that the largest percentage of phenol release resulted from the concentration of 1% (w/v) alginate of 2.52% (w/w) with a 20 minute long release time (time of phenol release maximum percentage). The lowest phenol percentage released in the control treatment (10% (w/v) maltodextrin) was 2.04% (w/w) with a release time of 10 minutes. The results above illustrated that 1% (w/v) alginate trapped phenol better than other treatments.

Alginate concentration above 1% (w/v) has a higher viscosity (data not shown) so that the percentage of phenol release is lower than 1% (w/v) alginate. High viscosity would make the capsule walls thicker, making it more difficult to release the active substances trapped by the encapsulating materials, while alginate concentrations below 1% (w/v) have a lower viscosity, making the walls of the capsule weaker in trapping the active substances. This causes the trapped active substance to be released during the spray drying process and the impact was less phenol in the phenol release test using distilled water. The study results of Patsialas et al., (2011) suggested that alginate of more than 1% (w/v) has a high solution viscosity so it is difficult to extrude and form beads on peptide encapsulation using alginate.



Fig 2. Liquid smoke microcapsules phenol drying yield and encapsulant efficiency

A high viscosity occured due to a strong interaction between encapsulating materials and active substances, whereas a low viscosity indicated a weak interaction. The control treatment (10%)(w/v)maltodextrin) has a quicker release maximum time of 10 minutes, the addition of alginate slowing down the release time, because phenolic compounds can enter into the alginate matrix network gel (Cvitanović et al., 2011). This can occur due to the interaction between the encapsulating materials (maltodextrin and alginate) and the trapped active substances (phenolic components of liquid smoke) (Ahmed et al., 2010). This interaction occurs because a phenolic reaction has the ability to enter into the polysaccharide network by means of penetrating the polymer membrane (Popa et al., 2000).

Encapsulant Efficiency

Figure 2 showed the encapsulant efficiency value of liquid smoke microcapsule phenol with the greatest value, in the 1% (w/v) alginate addition treatment of 45.13%, while the smallest value was in the control treatment which was 36.47%.

The greatest efficiency was in line with the value of phenol release which was the greatest value of all treatments, this was because the concentration of 1% (w/v) alginate was the best in trapping the phenolic compounds in liquid smoke, therefore 1% (w/v) alginate also has the greatest phenol value of all treatments, Habib *et al.*, (2011) stated that encapsulant efficiency is strongly influenced by the affinity between the core (active substances) and wall materials (encapsulating materials), so that the interaction between wall materials and active substances affects phenol release value and its efficiency.

Drying Yield

The highest drying yield (Figure2) also can be seen in the 1% (w/v) alginate treatment with 28.74%, and the lowest was in the control treatment with 20.94%. This was due to the growing viscosity which results in the smaller drying yield, and therefore alginate with a concentration more than 1% (w/v) will result in a smaller drying yield. This was also due to the alginate forming a gel, resulting in caking or microcapsules sintering, which can be seen







Fig 3. Scanning electron microscope images of maltodextrin-alginate 1 % (w/v) liquid smoke microcapsules (a), control (b) and Transmission electron microscope images of maltodextrin-alginate 1 % (w/v) liquid smoke microparticles (c)

by the number of microcapsules attached to the chamber spray dryer wall, resulting in a lower drying yield. Patsialas *et al.*, (2011), confirmed caking occured when the encapsulant solution sprayed has a high viscosity, thus resulting in agglomeration. This caking can be seen in the morphology of the microcapsules (Figure 3). Caking was also seen in the 1% (w/v) alginate treatment, however this treatment still has the greatest drying yield.

The Best Product

Based on the analysis of phenol release, encapsulant efficiency and drying yield, the best treatment was maltodextrin with the addition of 1% (w/v) alginate.



Fig 4. Phenol was encapsulated by maltodextrin-alginate 1 % (w/v) which marked with FeCl₃ solution (a) and without FeCl₃ solution (b)



Fig 5. Liquid smoke microcapsules size (maltodextrin-alginate 1 % (w/v)

Analysis of the morphology, phenol marker, and particle size determined the physical characteristics of the best treatment.

The morphology of liquid smoke microcapsules

TEM analysis was used to determine the shape of the microcapsule particles, while the SEM was used to determine the morphology of the microcapsule particle shapes. Figure 3 showed that the occurrence of agglomeration (stacked cell particles) was due to caking. SEM analysis of the control treatment (maltodextrin 10 % (w/v)) (Figure 3b) showed a ruptured capsule, indicating that the capsule wall was fragile, since the maltodextrin was not strong enough to trap the active substances during spray drying. Using 1% (w/v) alginate and 9 % (w/v) maltodextrin treatment there were no ruptured capsules. This suggested that the 1% (w/v) alginate and 9 % (w/v) maltodextrin as wall materials can better trap the liquid smoke phenolic compounds.

Phenol marker

Phenol marker analysis was used to determine that phenolic compounds, which were the liquid smoke's active substances, were trapped by encapsulant, as shown in Figure 4 which shows phenolic compounds trapped by maltodextrin and alginate encapsulating materials (Figure 4). Figure 4 shows that alginate can be used as an encapsulating material in trapping phenol active substance compounds. The orange color formed, indicated phenol components with the type of chlorohydroquinone, 2.4 dinitrophenol and hydroxybenzoic acids according to Soloway and Samuel (1952).

The orange color was due to the interaction of FeCl3 and the phenol compounds to form a complex between phenols and FeCl3 (Mohrig et al., 2003). The orange color, according to Soloway and Samuel (1952) is a phenolic compound encircled by a black line, which is the encapsulating material. This is consistent with the function of the encapsulant material used as a barrier between the core materials (encapsulated active substances) and the surrounding environment.

Particle Size

The microparticle sizes can be seen in Figure 5, which showed that the presence of several peaks showing that the particle sizes range was 16,56 nm-1224 micrometer. The sizes varied because the microparticle cells were agglomerating.

CONCLUSION

Alginate can be used as a wall material for encapsulating liquid smoke, despite agglomeration occuring, even in its optimum concentration, based on the results of TEM and SEM analysis. The results of the analysis of phenol release, encapsulant efficiency, and drying yield showed that the addition of 1% (w/v) alginate to maltodextrin was the best treatment. This is proven by the analysis of the phenol marker which indicated that phenolic compounds were trapped by this wall material.

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