

Preparation and Characteristics of Galangal Essential Oil/Alginate Microcapsules

Thichanee JIAMRUNGRAKSA¹ and Sireerat CHARUCHINDA^{1,2*}

¹*Center of Excellence in Textiles, Department of Materials Science, Faculty of Science, Chulalongkorn University, Bangkok 10330, Thailand*

²*National Center of Excellence for Petroleum, Petrochemicals, and Advanced Materials, Bangkok 10330, Thailand*

Abstract

Galangal essential oil/alginate microcapsules were prepared by solidifying in liquid (orifice method) between sodium alginate and calcium chloride. The structure, morphology and also the thermal stability of the microcapsules were investigated by FT-IR, optical microscope, infrared moisture determination balance (IMDB) at 120°C for 2 hours. The galangal essential oil release content of microcapsules was estimated by measuring time course of weight of microcapsules determination by isothermal TGA at 37°C and 120°C. FT-IR spectral data confirmed the presence of galangal essential oil in the prepared microcapsules. An increase of sodium alginate concentration and oil content resulted in significantly higher average size of the microcapsules ranging from 750 to 1000 µm. However, at 1% (w/v) of sodium alginate concentration, microcapsules tended to form agglomerate. IMDB data indicated that maximum oil content in the microcapsules was 40%. The galangal essential oil release content indicated that the thermal stability of galangal essential oil was less than the galangal essential oil/alginate microcapsules. Thus, it is expected that the microcapsules might be useful for improving the retention time of galangal essential oil in the capsule.

Key words : Galangal essential oil, Microencapsulation, Sodium alginate/Calcium chloride

Introduction

Health and hygiene have become essential to human being's way of life. Since cotton fabrics which are the most widely used fabrics can be easily attacked by microbes, antimicrobial finishing protecting consumers from the unfavorable effects of infections and also providing them a comfortable living is much needed.

Galangal, an abundant and low cost species of Thai's herbs, is considered as an environmental friendly antimicrobial agent to catch up awareness of the consumers' preference on green product.^(1, 3, 5-6) However, direct contact with high concentration of galangal essential oil could result in eye and skin irritation. In addition, its antimicrobial activity on cotton fabrics quickly disappears after washing due to its quick release characteristic. To solve these problems, microencapsulation using herbal oil as core and an alginate biopolymer as wall material adopted to reduce irritation, to control and control its release.⁽⁴⁾

In this study, galangal essential oil/alginate microcapsules will be prepared by solidifying in liquid (orifice process) between sodium alginate and calcium chloride.⁽²⁾ The characteristics of the microcapsules in terms of average size, loading capacity, morphology and thermal stability have been investigated.

Materials and Experimental Procedures

Materials

Galangal essential oil (Galangal EO) (extracted by steam distillation and contained 1,8-cineole 55%) was purchased from Thai China Flavours & Fragrances Industry Co., Ltd. Sodium alginate was kindly provided by August Chem Co., Ltd. Calcium chloride and 100% ethanol was purchased from TSL Chem Co., Ltd. and T.C. Sathaporn Group Ltd., Part., respectively.

Preparation of Galangal EO/Alginate Microcapsules

Alginate microcapsules containing galangal EO were prepared by solidifying in liquid (orifice process).⁽²⁾ 1, 3 and 5 mL of galangal EO were diluted

*Corresponding author E-mail : sireerat.c@chula.ac.th

with ethanol and was then mixed with 25 mL of 1 to 5% (w/v) sodium alginate aqueous solutions under continuous mechanical stirring at room temperature to make an emulsion. The obtained emulsion was injected using a syringe into a 250 mL of gel bath containing 5% (w/v) of calcium chloride solution. The obtained calcium alginate capsules suspension was equilibrated overnight and followed by washing twice with distilled water and dried at room temperature to evaporate water on the capsule surface.

Characterization of Microcapsules

The existence of galangal EO in the structure of microcapsules was characterized by ATR FT-IR (Bruker Tensor 27). The average size of the microcapsules was measured by an optical microscope (Olympus SZ40). Loading capacity of microcapsules was determined by an infrared moisture determination balance (IMDB, AND AD-4715) at 120°C for 2 hours.

$$\text{Loading capacity (\%)} = \frac{W_{m(0)} - W_0}{W_{m(0)}} \times 100 \quad (1)$$

Where $W_{m(0)}$, W_0 denote the weight of microcapsules measured before and after complete evaporation of galangal EO at 120°C for 2 hours, respectively.

The thermal stability of the microcapsules was evaluated by isothermal TGA (Perkin Elmer TGA7) at 37°C and 120°C. The galangal EO release content of microcapsules was estimated by measuring time course of weight of microcapsules determination by isothermal TGA at 37°C and 120°C.

$$\text{Oil release content (\%)} = \left(\frac{W_{m(0)} - W_{m(t)}}{W_{m(0)} - W_0} \right) \times 100 \quad (2)$$

Where $W_{m(0)}$, $W_{m(t)}$ denote the weight of the microcapsules at time 0 and t , respectively, whereas, W_0 denotes the weight of microcapsules measured after complete evaporation of galangal EO at 120°C for 2 hours, respectively.

Results and Discussion

Characterization of Microcapsules

FT-IR spectrum of galangal EO, alginate and microcapsules of galangal EO/alginate are presented in Figure 1. The spectrum revealed that

the prepared galangal EO/alginate microcapsules contained alginate due to the characteristic peak of alginate at 3400 cm^{-1} and 1600 cm^{-1} corresponding to O-H stretching and carbonyl stretching, respectively. Moreover, the characteristic peak of EO at 2900 cm^{-1} corresponding to C-H stretching was also found. Thus, the prepared microcapsules was confirmed the presence of galangal EO. This may also clarify from the SEM images of fracture surface of microcapsules with and without galangal EO as shown in Figure 2. It is obviously seen that surface of microcapsule without EO is smooth, whereas, surface of microcapsule with EO is porous due to the evaporation of the EO penetrate through the surface of microcapsules.

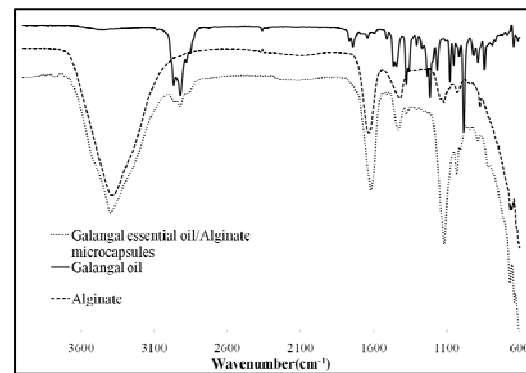


Figure 1. FT-IR spectrum of galangal EO, alginate and microcapsules of 3% (w/v) sodium alginate and 5 mL galangal EO content.

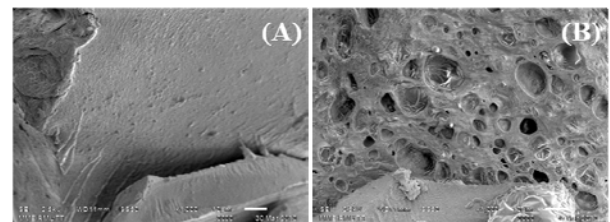


Figure 2. SEM images of fracture surface of microcapsules ($\times 1000$) (A) 3% (w/v) sodium alginate and 0 mL oil content, (B) 5% (w/v) sodium alginate and 5 mL oil content.

Effects of Concentration of Sodium Alginate and Galangal EO Contents on Size and Loading Capacity of Microcapsule

Table 1 shows that an increase of sodium alginate concentration and oil content significantly resulted in larger average size of the microcapsules ranging between 750 and 1000 μm . However, at 1% (w/v) of sodium alginate concentration, microcapsules tended to

Preparation and Characteristics of Galangal Essential Oil/Alginate Microcapsules

agglomerate (Figure 3). IMDB data indicated that maximum oil content in the microcapsules was 40% at 5 mL galangal EO content and 5% (w/v) sodium alginate concentration.

Table 1. Size and loading capacity of galangal EO/alginate microcapsules.

Sodium alginate concentration % (w/v)	Galangal EO content (mL)	Size (μm)	Loading capacity (%)
1	1	-	15.4
	3	-	23.2
	5	-	24.6
2	1	753.5	18.2
	3	813.8	24.4
	5	852.4	28.9
3	1	806.5	23.7
	3	868.8	27.8
	5	969.0	29.7
4	1	950.3	28.3
	3	1064.3	35.3
	5	1112.9	35.9
5	1	1026.3	36.9
	3	1137.4	39.4
	5	1164.3	40.8

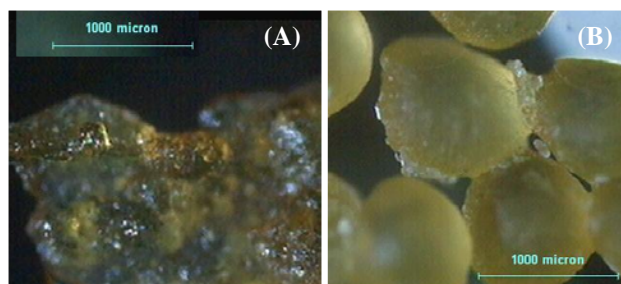


Figure 3. Microphotograph of microcapsules (x100) of 3 mL oil content and sodium.

Thermal Stability of Microcapsules and the Galangal EO Release Content of Microcapsules

Isothermal TGA provides an important tool for studying thermal stability between galangal EO and galangal EO/alginate microcapsules. The isothermal TGA curves at 37°C and 120°C shown in Figure 4 indicated that, at first ten minutes of 120°C, galangal EO exhibited a sharp weight loss when compared to the galangal EO/alginate microcapsules. Galangal EO continued extremely losing of its weight up to 60 minutes with a total weight loss of about 80%. In contrast, after 10 minutes up to 60 minutes, the weight of the microcapsules remained almost constant with a total weight loss of about only 35-40%. This may be attributed that alginate wall of the microcapsule protects the entrapped EO from the evaporation.

Thus, the microencapsulation technique could improve durability of galangal EO on application. The galangal EO release content of microcapsules was estimated by measuring time course of weight of microcapsules determination by isothermal TGA at 37°C and 120°C. The oil release content curve shown in Figure 4 indicated that, at first ten minutes of 120°C, oil content in microcapsules releases extremely about 90% and full release (100%) at 50-60 minutes. Furthermore, at 37°C, oil content in microcapsules releases about 40% and 60% at 10 and 60 minutes, respectively. This information is useful to consider the appropriate condition for processing and application of the prepared microcapsules.

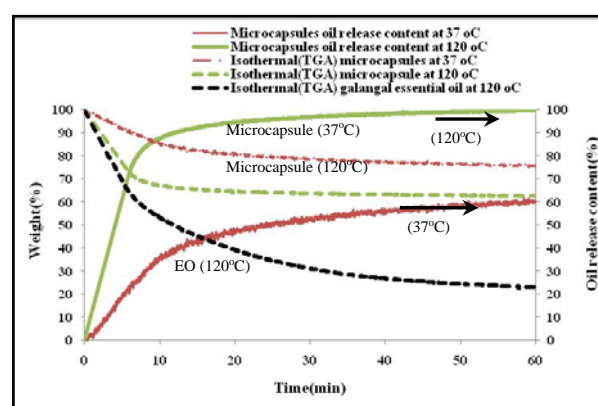


Figure 4. TGA thermogram and oil release content of galangal EO at 120°C and microcapsules of 5% (w/v) sodium alginate and 3 mL galangal EO content at 37°C and 120°C.

Conclusions

Microencapsulation of galangal EO has been done successfully by solidifying in liquid using galangal essential oil as core material and alginate as wall material. An average size of the microcapsules was in range of 750 – 1000 μm and maximum oil content in the microcapsules was 40%. Galangal EO/alginate microcapsules exhibited higher thermal stability than that of galangal EO. Finally, durability of galangal EO could be improved by microencapsulation. Furthermore, at 120°C, oil content in microcapsules releases about 90% and 100% at 10 and 60 minutes, respectively. In addition, at 37°C, oil content in microcapsules releases about 40% and 60% at 10 and 60 minutes, respectively.

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