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Microencapsulation of Lemon Oil by Spray Drying Using Acacia and Maltodextrin

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ABSTRACT: The use of spray drying as a method of preparing microcapsule powder from lemon oil was studied. Acacia and maltodextrin were used as filmogenic and protective agents in preparing lemon oil microcapsule, respectively. Various ratios of acacia:maltodextrin:lemon oil were utilized in preparing feeding liquids for microencapsulation of the lemon oil by spray drying. The suitable ratio of acacia:maltodextrin:lemon oil employed in the feeding liquid and the optimum concentration of feeding liquid for producing the high percent yield of microcapsule production were identified. The highest yield of microcapsule production was obtained by using acacia:maltodextrin:lemon oil in the ratio of 15:7.5:7.5 (g%) and in the concentration of 20%. The use of lemon oil and microcapsule powder as flavoring agents in ascorbic tablet formulations were investigated. Tablet sticking was found in tablets utilizing oil but did not occur in the case of utilizing microcapsule powder as the flavoring agent in the ascorbic acid tablet formulation.

Key words: microencapsulation, spray dry, acacia, maltodextrin

INTRODUCTION

Microencapsulation has been applied in various fields of pharmaceutical industry (1). One of these applications was microencapsulation of volatile oils to produce tablet flavoring agents in powder form. In general, the use of volatile oils directly as tablet flavoring agent can result in some problems during tablet production such as tablet sticking and poor flowability of granule or powder (2). Microencapsulation of volatile oils can eliminate these problems by encapsulating the volatile oils within the thin film of polymer(s) resulting in microencapsulated powders. Various methods and polymers have been utilized in microencapsulation process to prepare microcapsules from the volatile oils (3,4). However, spray drying technique is the method of choice for a large scale production since it provides a means for a continuous production. In addition, it has been applied widely in various industries

such as chemical, food, fish, and pharmaceutical industries (1,5). During spray drying process, high temperature is needed in order to evaporate the solvent from the product. The residence time, when exchange of heat between the stream of drying air and the sample take place, is comparatively short. The material being spray dried is not heated up to the temperature of the drying air. As a matter of fact, the droplets sprayed into the hot stream of air form immediately a steam coating, protecting the core of the droplets from excessive heat exposure because the evaporation energy keeps the particles cool (6). Hence, the high temperature utilized in spray drying process does not present any problem for microencapsulation of the heat labile materials.

The polymers employed in microencapsulation process range from water-soluble polymers (e.g. acacia, gelatin) to water-insoluble polymers (i.e. ethylcellulose)

depending on the applications of microcapsules produced. For microencapsulation of volatile oils, gelatin and acacia were generally employed (7). However, there was a suggestion of using acacia and maltodextrin in preparing microcapsules of volatile oils by spray drying but the suitable conditions of the spray drying process was not specified (6). In this investigation a volatile oil, lemon oil, was used to prepare microcapsules by spray drying process using acacia and maltodextrin as a filmogenic and protective agents, respectively. Acacia provided film forming property to encapsulate lemon oil while maltodextrin added physical strength to the film. Lemon oil microcapsules were then incorporated into various ascorbic acid tablet formulations in order to studying the use of the microcapsule as a tablet flavoring agent. Both acacia and maltodextrin alone are frequently used in many tablet formulations (8), therefore the application of the spray-dried lemon oil microcapsules in tablet formulation should be acceptable.

MATERIALS AND METHODS

Materials

The following chemicals were obtained from commercial sources : lemon oil (supplied by Nutrition Ltd., Part., Thailand), acacia (Batch No. AA 23/344, supplied by Srichand United Dispensary Co., Ltd., Thailand), maltodextrin PHS 17 (supplied by Nutrition Ltd., Part., Thailand), ascorbic acid (Lot No. HM SP 922, Japan), lactose (The Lactose Company of New Zealand, Ltd., New Zealand), mannitol (Fluka Chemic, Switzerland), citric acid (Lot No. CAM

06/209, China), saccharin sodium (Lot No. SA 05, Srichand United Dispensary Co., Ltd., Thailand), sodium chloride (Ajax Chemicals, Australia), tartrazine (Lot No. C811184, Government Pharmaceutical Organization, Thailand), absolute ethanol (Government Pharmaceutical Organization, Thailand), polyvinylpyrrolidone K90, talcum, silicon dioxide (Aerosil®) and magnesium stearate (supplied by Pharmaceutical Science Ltd., Part., Thailand).

Equipment

Spray dryer (Buchi 190 Mini Spray Dryer, Buchi Co., Switzerland), single stroke tableting machine (Viuhang Engineering, Thailand), analytical balance (Sartorius, Model A200S, Germany), hot air oven (Binder, Model E115, Germany).

Methods

Preparation of lemon oil microcapsules by spray drying

Various ratios of acacia:maltodextrin:lemon oil were prepared as spray feeding liquids for the production of lemon oil microcapsules by spray drying method. The ratios used were expressed in term of grams per 100 ml (g%). For example, the spray feeding liquid containing 10:10:7.5 acacia:maltodextrin:lemon oil consisted of 10.0 g of acacia, 10.0 g of maltodextrin, and 7.5 g of lemon oil in 100 ml of aqueous solution. The inlet temperature of spray drying process was set at 150°C and the feeding rate about 5 ml

Table 1. Various ratios of acacia:maltodextrin:lemon oil employed in spray feeding liquids of 100%, 50%, and 20% concentrations.

Acacia:Maltodextrin:Lemon oil (g%)	Feeding liquid concentration		
10:10:7.5	100%	50%	20%
15:7.5:15	100%	50%	20%
15:7.5:7.5	100%	50%	20%
7.5:7.5:7.5	-	50%	20%
10:7.5:7.5	-	50%	20%
15:15:7.5	-	50%	20%
7.5:15:10	-	50%	20%
15:7.5:10	-	50%	20%
20:10:10	-	50%	20%

per min. Prior to spray drying, weighed amount of acacia was dispersed in distilled water then predetermined amount of maltodextrin was added into the acacia solution. Lemon oil in the required amount was finally incorporated into the solution of acacia and maltodextrin. The ratios of acacia:maltodextrin:lemon oil utilized in the micro-encapsulation processes are listed in Table 1. The concentration of the employed spray feeding liquids were also indicated. Spray-dried microcapsules were collected from the collector of the spray drier and determined for their percent yield (by weight).

Preparation of ascorbic acid tablets using lemon oil microcapsule and lemon oil as flavoring agents

Ascorbic acid tablets were prepared by wet granulation method using lemon oil (Formulation I-III, Table 2) and

lemon oil microcapsules (Formulation IV-VI, Table 2) as flavoring agents. To prepare the ascorbic tablets; the ascorbic acid, lactose, mannitol, saccharin sodium, citric acid or citric acid and sodium chloride were mixed thoroughly. The resulting mixture was granulated by ethanolic solution of polyvinylpyrrolidone K90. Tartrazine was dissolved into the binding solution of polyvinylpyrrolidone K90 prior to granulation. For the tablets prepared by using lemon oil as flavoring agent (Formulations I-III), the oil was first dissolved in small amount of absolute ethanol and then was sprayed on the talcum powder prior to mixing with the ascorbic acid granules, lubricant and glidant. For the tablets prepared by using lemon oil microcapsules as flavoring agent (Formulations IV-VI), the lemon oil microcapsules and the lubricant and glidant were mixed to the dried granules. The resulting mixtures were compressed into the tablets of 9 mm in diameter.

Table 2. Tablet formulations (per tablet) for preparing ascorbic acid tablets containing lemon oil (I-III) or spray-dried lemon oil microcapsule (IV-VI) as flavoring agents.

Ingredient	Formulation			Formulation		
	I	II	III	IV	V	VI
Ascorbic acid (mg)	55	55	55	55	55	55
Lactose (mg)	170	170	170	170	170	66
Mannitol (mg)	50	50	50	50	83	25
Saccharin sodium	0.5%	0.5%	0.5%	0.8%	0.8%	0.8%
Citric acid	3.5%	3.5%	3.5%	3%	5%	5%
Sodium chloride	0.5%	0.5%	0.5%	-	-	-
Polyvinylpyrrolidone K90						
- 12% ethanolic solution	-	-	-	3%	-	-
- 15% ethanolic solution	-	-	-	-	3%	3%
- 20% ethanolic solution	3%	3%	3%	-	-	-
Talcum	1%	1%	1%	1%	1.5%	1%
Magnesium stearate	0.5%	0.5%	0.5%	0.8%	0.8%	0.5%
Aerosil®	-	-	-	0.5%	1%	0.5%
Tartrazine	q.s.	q.s.	q.s.	q.s.	q.s.	q.s.
Lemon oil	1%	2%	3%	-	-	-
Spray-dried lemon oil microcapsule	-	-	-	2%	2%	1.5%

RESULTS AND DISCUSSION

Acacia in a suitable amount was needed to prepare the spray feeding liquids. Too low in acacia content resulted in separation of oil phase from the feeding liquids. On the other hand, acacia in too high content caused dispersion problem and the resulting feeding liquids was too viscous to spray effectively. Lemon oil in excess concentration also induced phase separation due to an inadequate amount of acacia to cover the oil surface. Hence, the suitable ratios of acacia:maltodextrin:lemon oil employed in preparing the spray feeding liquid was needed to be determined.

The percent yields of the lemon oil microcapsules obtained by using the feeding liquids containing acacia:maltodextrin:lemon oil in the ratios of 10:10:7.5, 15:7.5:15, and 15:7.5:7.5 were 23.11%, 20.61%, and 20.00%, respectively. The spray-dried lemon oil microcapsule was white fine powders with lemon odor. The spray dryer used in this investigation was a laboratory scale model having the chamber diameter of about 6 inches. Hence, high percentage of loss was expected due to narrow chamber space available for rapid drying of the feeding liquid droplets. However, the obtained yield of about 20% was considered to be too low to accept. The percent yield obtained from the feeding liquid of 15:7.5:15 acacia:maltodextrin:lemon oil was low (20.61%) while the lemon oil content in the feeding liquid was high (15 g%). Therefore, this feeding liquid of

100% concentration was not suitable for preparing the lemon oil microcapsules. Diluting the 15:7.5:15 acacia:maltodextrin:lemon oil feeding liquid to 50% and 20% concentrations prior to spray drying then were done in order to find a way to improve the percent yield. After spray drying, the percent yields obtained from the 15:7.5:15 acacia:maltodextrin:lemon oil feeding liquids of 50% and 20% concentrations were 20.00% and 18.40%, respectively. Thereby, the feeding liquid of 15:7.5:15 acacia:maltodextrin:lemon oil was not suitable to produce high yield of the microcapsules from spray drying.

The percent yield obtained from the 10:10:7.5 and 15:7.5:7.5 acacia:maltodextrin:lemon oil feeding liquids of 100% concentration were 23.11% and 20.00%, respectively. Therefore, the obtained percent yield varied from 20.00% to 23.11% after fixing the content of lemon oil at 7.5 g% and varying the ratios of acacia:maltodextrin at 15:7.5 and 10:10, respectively. In order to study the effect of acacia:maltodextrin ratio on the percent yield, the feeding liquid of fixed lemon oil content at 7.5 g% and varying the ratios of acacia:maltodextrin at 7.5:7.5, 10:7.5, 10:10, 15:7.5, and 15:15 were prepared. The effect of the feeding liquid concentration of 50% and 20% on the percent yield was also investigated. Table 3 shows the percent yields obtained from these spray feeding liquids of fixed lemon oil content.

Table 3. Percent yield of lemon oil microcapsules obtained from the feeding liquids of fixed the lemon oil content (7.5 g% and 10 g%) and varying in acacia:maltodextrin ratio.

Acacia (g%)	Maltodextrin (g%)	Lemon oil (g%)	% Yield	
			50% (conc)	20% (conc)
7.5	7.5	7.5	18.13	23.33
10	7.5	7.5	17.76	18.10
10	10	7.5	28.73	30.90
15	7.5	7.5	20.00	46.33
15	15	7.5	23.62	36.40
7.5	15	10	17.26	17.90
15	7.5	10	25.00	29.70
20	10	10	20.05	25.80

From Table 3, the percent yields obtained from the feeding liquids, having various ratios of acacia:maltodextrin while fixing the lemon oil content (7.5 g%), were between 17.76% to 28.73% and 18.10% to 46.33% for the feeding liquids of 50% and 20% concentration, respectively. The maximum percent yield of 46.33% was obtained from the 15:7.5:7.5 acacia:maltodextrin:lemon oil ratio and feeding liquid of 20% concentration. It appeared that the percent yields obtained from the feeding liquids of 20% concentration were higher than the ones obtained from the feeding liquids of 50% concentration, given the same acacia:maltodextrin:lemon oil ratios. The feeding liquids of 50% concentration were more viscous as compared to the feeding liquids of 20% concentration. Consequently, the more effective and convenient spray drying process was achieved from the feeding liquid of 20% concentration. As the result, the feeding liquid of 50% concentration provided lower percent yield of the spray-dried lemon oil microcapsules.

Table 3 also presents the percent yields obtained by the feeding liquids of fixing the lemon oil content at 10 g% while varying in the ratios of acacia:maltodextrin. It was demonstrated that the obtained percent yields ranged from 17.26% to 20.05% and 17.90% to 29.70% for the feeding liquids of 50% and 20% concentrations, respectively. Hence, by fixing the concentration of lemon oil at 10 g% in the feeding liquids was not suitable for spray drying of high percent yield. In addition, the percent yields obtained from the feeding liquids of 20% and 50% were not much different. The concentration of lemon oil at 10 g% used in the feeding liquids was shown to be too high resulting in feeding liquids that were too viscous to give the good spray drying result.

From the investigation, most suitable feeding liquid for preparing the spray-dried lemon oil microcapsules was the 15:7.5:7.5 acacia:maltodextrin:lemon oil feeding liquid in the concentration of 20% which provided the yield of 46.33% by weight. The maximum percent yield obtained from this study was only 46.33% due to the limitation of the small chamber employed in the laboratory scale spray dryer. However, the higher percent yield of the lemon oil microcapsule was expected if the spray dryer of large scale type was applied. In such case, the more space available for rapid drying of the feeding liquid droplets was obtained and subsequently higher percent yield of microcapsule production was anticipated.

The prepared spray-dried lemon oil microcapsule then was used as flavoring agent in preparation of ascorbic acid tablets according to the formulations listed in Table 2. By comparing the ascorbic tablets prepared by using the spray-dried lemon oil microcapsule (Formulations IV-VI) and the liquid lemon oil (Formulations I-III) as tablet flavoring agents, the quality of the tablets prepared by using the spray-dried lemon oil powder was better, both in tablet physical appearances and ease of tableting process. Tablet sticking and poor flowing of granules were encountered during tableting process of the formulations using the lemon oil as the flavoring agent, while these problems did not exist in the formulations of spray-dried lemon oil powder.

The ascorbic acid tablets prepared from Formulations IV and V were sour while Formulation VI provided ascorbic tablet of satisfied taste. Formulation IV and VI yield the ascorbic tablets of better physical appearances than the tablet of Formulation V. Thereby, the best formulation for the ascorbic acid tablet prepared by using spray-dried lemon oil microcapsule as flavoring agent was Formulation VI. In addition, the spray-dried lemon oil microcapsule was found to be physically stable when kept in a desiccator. After 12 months, the spray-dried microcapsule was still usable as flavoring agent in preparing ascorbic acid tablets of good physical appearance and odor without problem in tableting process.

CONCLUSION

The application of spray drying process in preparing lemon oil microcapsule as tablet flavoring agent could be achieved with a satisfied yield by using an appropriate ratio of acacia:maltodextrin:lemon oil and a suitable concentration of the feeding liquid. Since the spray drying conditions are important factors in determining the effectiveness of spray drying process, therefore further study on spray drying conditions is recommended. In such manner the more productive means of spray drying process may be achieved, given the more appropriate conditions of spray drying such as inlet temperature, spray feeding rate, and feeding liquid concentration.

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ไมโครเอนแคปซูลชันของน้ำมันผิวมะนาวด้วยการพ่นแห้ง โดยใช้เอเคเซียและมอลโตเด็คชตริน

เพียรภิจ แดงประเสริฐ^{1,2} และศันสนีย์ พงษ์วัย¹

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²ผู้เขียนที่สามารถติดต่อสอบถามได้

บทคัดย่อ : ได้ทำการศึกษาถึงการใช้วิธีการพ่นแห้งในการเตรียมผงไมโครแคปซูลจากน้ำมัน ผิวมะนาว โดยใช้
เอเคเซียและมอลโตเด็คชตรินเป็นสารก่อฟิล์มและสารปกป้องฟิล์มตามลำดับ และใช้ของเหลวที่ทำการพ่นแห้ง
ที่ประกอบด้วยเอเคเซีย:มอลโตเด็คชตริน : น้ำมันผิวมะนาวในอัตราส่วนต่าง ๆ และเมื่อศึกษาหาอัตราส่วน
ที่เหมาะสมของเอเคเซีย :มอลโต เด็คชตริน:น้ำมันผิวมะนาวที่ใช้ รวมทั้งหาความเข้มข้นที่เหมาะสมของของ
เหลวที่ทำการพ่นแห้ง พบว่าเมื่อใช้ของเหลวที่ทำการพ่นแห้งซึ่งประกอบด้วยเอเคเซีย : มอลโตเด็คชตริน :
น้ำมันผิวมะนาวในอัตราส่วน 15 : 7.5 : 7.5 (g%) ในความเข้มข้น 20% จะได้ผลผลิตของไมโครแคปซูลที่มากที่สุด
จากนั้นทำการศึกษาถึงการนำน้ำมันผิวมะนาวและไมโครแคปซูลของน้ำมันผิวมะนาวไปใช้เป็นสารแต่งกลิ่น
ในตำรับยาเม็ดแอสคอบิคแอซิดพบว่าเกิดปัญหาการติดของยาเม็ดในกรณีที่ใช้ น้ำมันผิวมะนาวเป็นสารแต่ง
กลิ่น แต่ไม่พบปัญหาดังกล่าว ในกรณีที่ใช้ไมโครแคปซูลของน้ำมันผิวมะนาว เป็นสารแต่งกลิ่นในตำรับยาเม็ด
แอสคอบิค แอซิด

กุญแจคำ : ไมโครเอนแคปซูลชัน การพ่นแห้ง เอเคเซีย มอลโตเด็คชตริน

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