Australian Journal of Basic and Applied Sciences, 6(12): 499-504, 2012
ISSN 1991-8178

Microencapsulation Of Peppermint Oil By Spray Drying

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Abstract: Microencapsulation is one of the quality preservation techniques of sensitive substances used in food and pharmaceutical industries. Peppermint oil was encapsulated with gum arabic, maltodextrin, and a blend,(1:1 by mass) of gum arabic and maltodextrin by spray drying. The drying gas was hot air at an inlet temperature 200^0C. No valuable changes were detected in the chemical composition of peppermint oil after encapsulation. The materials giving the highest flavor retention during drying were gum arabic and the (1:1)blend, as they provided acceptable oil retention (81% and 80%) ,respectively. The results showed the high safety of encapsulated peppermint oil, as biological evaluation showed that serum glucose, cholesterol and aminotransferases enzymes exhibited no significant change as compared to control mice. Meanwhile, creatinine (mg/dl) was significantly, but slightly, increased from 0.8 to 1.07 mg/dl after feeding diet containing encapsulated peppermint oil.

Key words: encapsulation, emulsion, peppermint oil, spray drying, gum arabic, maltodextrin and carrier

INTRODUCTION

Flavor plays an important role in consumer satisfaction and influences further consumption of foods. Most available aroma compounds are produced via chemical synthesis or extraction. Foodstuffs containing synthetic flavor are often avoided, because the consumers suspect that these compounds are toxic or harmful to their health. Recently, the market of flavors is focused in using aromatic materials coming from natural sources to replacie the use of synthetic flavors gradually Teixeira *et al*, (2004).

Flavor is indispensable ingredients of food preparations, and their compositions are often highly complex. Commercial food flavors in liquid form are difficult to handle or incorporate into food. Moreover, many flavor components exhibit considerable sensitivity to oxygen, light, and heat. In response to these difficulties, dry flavors have been developed through encapsulation and used for applications such as flavored confectionery, chewing gums, toothpastes and pharmaceutical products (Risch & Reineccius, 1995; Gibbs *et al*., 1999; Madene *et al*., 2006).

The spray drying is an economical and flexible process. This technique allows obtaining high volatile retention efficiencies and it produces powders with a satisfactory stability (Reineccius, 1991). The main factors that affect encapsulation efficiency of microencapsulated oils and flavors are: type of wall material, properties of the core materials, characteristics of the infeed emulsion and conditions of the spray drying process (Jafari *et al*., 2008).

The objective of this work was to study the influence of different carrier materials on the flavor retention of peppermint oil during spray drying and to biologically evaluate the safety of peppermint oil capsules.

MATERIALS AND METHODS

Peppermint oil was obtained from Kato Aromatic Co., Giza (Egypt) .Gum Arabic was obtained from PRS Panreac (Espania). Maltodextrin DE - 20 was obtained from the National CO., Maize Product Cairo (Egypt).

Experimental animals were obtained from the Animals House of the National Research Center, Egypt. Swiss Albino mice of five weeks age with an average weight of 25 - 30 gram were used in the experiment.

Kits for determination of glucose, cholesterol, aspartate amino transferase (AST), alanine amino transferase (ALT) and creatinine were purchased from Biodiagnostic Giza Co., Egypt.

Emulsion Preparation and Spray Drying:

Solutions of gum arabic, maltodextrin and (1:1) blend of gum arabic and maltodextrin were prepared by dispersing the solids in deionized water and heating at 60^0 C over a steam bath to facilitate solubilization. The solutions were allowed to cool to room temperature before storing at 4^0C overnight. The peppermint oil (10, 20% and 25% w/w) was added to and homogenized vigorously (10 000 rpm for 5 min.) with an Ultra Turrax M-45 homogenizer at ambient temperature. The obtained emulsion was maintained under slow agitation during spray drying.

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The emulsion was spray dried in a BUCHI 190 Spray Dryer with an evaporation rate of 1.5 kg /1 hour and a chamber diameter of 10 cm, equipped with a pressurized nozzle operating at 5 atmospheres. Feed is metered into the dryer by a peristaltic pump. Powder was collected at the bottom of dryer cyclone and kept in air tight containers at 8^0 C until analyzed

Separation And Identification Of The Chemical Components Of Peppermint Oil:

Gas chromatography technique was used to separate and identify the volatile chemical components using a Hewlett Packard 5890 series II Instrument equipped with a flame ionization detector (FID) and a carbowax fused silica column (50 m length, 0.25 mm inside diameter, and film thickness of 0.32 µm). The oven temperature was programmed from 60 ^{o}C to 200 ^{o}C at the rate of 3 ^{o}C/min. Helium (1 ml/min) was used as a carrier gas with a split ratio of 100: 1. The temperature of injection port and detector were 150^0C and 250°C, respectively. Percentages of peak area were calculated with a Hewlett Packard 3396 integrator.

Determination of Viscosity:

Viscosity was determined using Brookfield Viscometer (DV-II+, Middleboro, MA, USA) equipped with the LV spindle Brookfield at 20 rpm according to Kim *et al*.,(1996).

Determination Of Total Oil Remainder And Moisture Content:

Total oil was determined by Clevenger distillation and Moisture content was determined by the toluene distillation in the spray dried powders according to Association of Official Analytical Chemist (1990).

Determination Of Surface Oil:

The volatile compounds retained on the surface of particles were determined by washing of 5 gram of powder with 20 ml of diethyl ether. The solvent was evaporated with rotary evaporator at 25^0 C. The surface oil was determined by weighing the oil (Trubiano, & Lacourse, 1988).

Determination of Bulk Density:

Bulk density was determined by tapping method according to Hall & Hedrick (1971).

Biological Evaluation:

Ten of Swiss Albino male mice were given daily gavages doses of encapsulated peppermint oil. The dose was 1/10 of LD_{50} (LD_{50} = 2490 mg/kg body weight) which was dissolved in 10 ml water. The dose was applied 5 days per week for 30 days. The Standard Balanced Diet was consisted of casein 15 g/100g , starch 43.7 g/100g , sucrose 21.8 g/100g , fat 15 g/100g , salt mixture 3.5 g/100g and vitamin mixture 1 g/100g , according to Varna *et al*. ,1978), water were available ad .labium.

Blood samples were taken from mice after 18 hours fasting period on the 30 day. According to the method described by (Schemer,1967). The collected blood samples were put into a dry clean glass tubes, left for 15 minutes at room temperature to coagulate by itself then the tubes were centrifuged at 3000 rpm for 10 minutes. The clean supernatant serum was kept at -20°C for analysis.

The glucose content was determined according to the methods of (Barham & Trinder, 1972). The determination of total cholesterol was carried out according to the method of Richmond, 1973. The determination of Alanine aminotransferase (ALT) was carried out according to (Reitman & Frankel, 1957) .The determination of Aspartate aminotransferase (AST) was carried out according to (Reitman & Frankel, 1957). Serum creatinine was kinetically determined according to (Houot, 1985).

Statistical Analysis:

Analysis of variance (ANOVA) was processed using statistical package for social science (SPSS) software version11.0.1 (2001) and data were presented as means ± S.E.M**.** (Standard error of mean) $P < 0.05$.

RESULTS AND DISCUSSION

3.1 - Chemical Composition Of Peppermint Oil:

The chemical composition of peppermint oil was identified. Table (1) indicated that the major two components of peppermint oil (more than 10%) are menthol (39.03%) and menthone (25.19%).Simultaneously, ten minor components (1 - 10%) were identified, namely Alph pinene, Beta pinene, Camphene, Myrcene, Limonene, Octanal, Isomenthone, Linalool, Methyl acetate and Piperitone.

Table 1: Chemical Composition of peppermint oil before and after encapsulation

Peak	RRT	Component	%*	
			before	after
1	0.189	Alph pinene	3.32	3.29
2	0.239	Beta pinene	2.44	2.42
3	0.248	Camphene	1.37	1.07
4	0.280	Myrcene	1.29	1.59
5	0.318	Limonene	5.37	5.16
6	0.576	Octanol	1.89	1.52
7	0.698	Menthone	25.08	24.19
8	0.741	Isomenthone	5.54	5.56
9	0.870	Linalool	5.64	5.63
10	0.918	Menthyl acetate	6.46	6.67
11	1.0	Menthol	39.01	40.30
12	1.139	Piperitone	2.61	2.60

RRT: is the relative retention time of menthol (22.139 min) which used as standard retention time equal one
*% equal the average values before and after encapsulation

The principal components of the Pharmaceutical grade peppermint oil are menthol (29%), menthone (20-30%), and menthyl acetate (3-10%) (European pharmacopeia, 1997).

Same table indicated that no valuable changes could be detected in the chemical composition of peppermint oil before and after encapsulation process.

3.2 The Relationship Between Three Carriers And Their Viscosity:

The viscosity of different solutions obtained using three selected carriers namely, maltodextrin (MD), blend mattodextrin and gum Arabic (1:1) and gum arabic (GA) at eight different concentrations were detected.

Table 2: Effect of the concentration of the three selected wall materials on their viscosity (by centipoises cp at 25^0C)

Carrier concentration %W/W	Viscosity of the Selected Wall Materials (CP) at 25^0C		
	MD	blend GA:MD (1:1)	GA
5	11.5	13.2	16.1
10	15.6	17.3	21.2
15	22.0	30.4	75.2
20	30.2	66.5	143.6
25	39.4	98.1	179.2.
30	47.1	138.7	256
35	56.8	205	345
40	64.5	275	467

MD: Maltodextrin GA: Gum Arabic blend GA: MD (1:1)

From Table (2), It could be noticed that the lowest viscosity values were that of MD, while blend GA: MD (1:1) and GA carries had the higher values at different used concentrations.

The maximum infeed viscosity that can be efficiently atomized in the spray dryer was 250 cp. Thus, optimum chosen concentration for GA was 30% and for GA: MD blend was up to 40%, while MD carried was used at over 40%. These results are in harmony with the work of Reineccius (1991).

3.3 Effect Of Carrier To Oil Ratio On The Percentage Of Oil Retention:

The following experiment was conducted to study the effect of three carrier-oil ratios, namely 9:1, 4:1 and 3:1 on the oil retention%.

Table 3: Effect of Carrier: oil ratio on the retention

Ratio carrier : Oil	Starting (goil/100g powder)	MD Total oil remainder (g oil/100g Powder)	Retention %	MD:GA (1:1) Total oil remainder (g oil/100g Powder)	Retention %	GA Total oil remainder (g oil/100g Powder)	Retention %
9:1	10	5.5	55	7.9	79	8.1	81
4:1	20	11.2	56	16	80	16.2	81
3:1	25	13	52	18	72	18.8	75

MD: Maltodextrin GA: Arabic gum blend MD: GA (1:1)

The data in Table (3) reveal that the ratios 9:1 (10%) and 4:1 (20%) had the highest retention %, but when the ratio 3:1 (25%) was utilized, a lower retention percentages was obtained. These results are in agreement with those obtained by Sankarikutty *et al*., (1988) and Risch (1995), who mentioned that the ratio 4:1 is usually adopted in most of published reports. The ratio 4:1 has been reported optimal for encapsulating materials like gum arabic, and for other carbohydrate derivatives (Reineccius, 1988).

It might be clear that, as the oil concentration increased the encapsulation efficiency gets lower. This can be attributed to the greater amount of core material close to the drying surface, which makes the diffusion path length short to the air/particle interface, thus increasing the surface oil content.

3.4 The Properties Of Encapsulated Peppermint Oil Powder:

The properties of peppermint oil powder largely affect its application. Therefore, the effect of these properties were experimentally studied.

Table 4: Properties of powder encapsulated peppermint oil (Total oil, Surface oil, Moisture content and Bulk density)

Carrier material	Starting(g oil/100g powder)	Total oil (g oil/100g Powder)	Retention %	Surface oil (g oil/100g powder)	Moisture Content %	Bulk density (g/ml)
MD	20	11.2	56	0.9	4.0	0.55
MD:GA (1:1)	20	16.0	80	0.7	4..5	0.49
GA	20	16.2	81	0.4	4.6	0.46

MD: Maltodextrin GA: Arabic gum blend MD: GA (1:1)

Table (4) shows that total oil was ranged from 11.2-16.2 % w/w. GA had the highest volatile oil retention (81%), while MD had the lowest volatile oil content retention (56%). MD typically do not produce good retention; this could be explained by their poor film – forming ability (Reineccius, 1991). The wet encapsulation matrix must form a film around the flavor compounds during the drying process, while losing the water. Since MD have no emulsification properties they produce coarse emulsions and poor flavor retention during drying.

Since moisture content affecting powder stability and caking properties, the obtained data showed that MD had the lowest moisture content (4.0%), while GA had the highest moisture content (4.6%).

In addition, the highest surface oil content was found in MD, i.e. 0.9 g/100g, while GA had the lowest surface oil content, i.e. 0.4 g/100g. Surface oil content is strongly related to the emulsion droplet size (Risch & Reineccius, 1988), and low surface oil content is important for providing storage stability to the encapsulated oil (Anandaraman & Reineccius, 1987).

Bulk density is important in packing and shipping considerations .This value tells one how much material, by weight, will fit into a container of specified volume (Finney *et al*., 2002). Bulk density results of this study ranged between 0.46-0.55g/ml

3.5 The Biological Effect Of Encapsulated Peppermint Oil:

The safety of encapsulated peppermint oil before use in human diet .was checked. The changes in some clinical pathological parameters

Table 5: Changes in clinical pathology parameters of mouse (young Adult) feed diet containing encapsulated peppermint oil

Group	AST, U/L	ALT, U/L	Glucose, mg/dl	Cholesterol, mg/dl	Creatinine, mg/dl*
Control	35.0±2.1	35±3.0	165.0±1.0	116.0±1.0	0.8±0.02
Peppermint oil	34.0±1.4	32.75±5.0	165.0±3.5	140.0±3.9	1.07±0.04*
Normal range	<30- 40	<30- 40	80 - 160	90 -170	0.3 - 0.8
Reference	(Harris,2005)	(Harris,2005)	(Gad & Chengelis 1992)	(Gad & Chengelis,1992)	(Gad & Chengelis,1992)

Values are expressed as means ± S.E.M
* Vaues are statistically significant at

Table (5) showed that feeding of mouse in diet containing encapsulated peppermint oil did not significantly affected liver function, since no significant changes were detected in AST and ALT before and after feeding on encapsulated peppermint oil .In addition, glucose and cholesterol followed the same above mentioned trend, as the changes in both after feeding peppermint oil were not significant. On the other hand, creatinine content (mg/dl) was slightly but significantly increased from 0.8 (in control) to 1.07 mg/dl after feeding on peppermint oil. The results proved the high safety of encapsulated peppermint oil in human diet.

Conclusion:

1- The concentration of Gum Arabic should not be increased beyond 30%, as higher gum concentration produces solution which is very viscous to be pumped through the atomizer of the spray drier.

2-The highest retention of flavor (81%) was detected with gum Arabic.

3-The concentration of core (peppermint oil) at 1:9 or 1:4 had the highest retention (81%) on gum Arabic.

4- The data showed that feeding of mouse in diet containing encapsulated peppermint oil did not significantly affected liver function, since no significant changes were detected in AST and ALT before and after feeding on peppermint oil diet .In addition, glucose and cholesterol followed the same above mentioned trend, as the changes in both after feeding peppermint oil were not significant. On the other hand ,creatinine content (mg/dl) was significantly increased from 0.8 (in control) to 1.07 mg/dl after feeding on pepermint oil .These results prove the complete safety of using encapsulated peppermint oil in human diet. Further studies are essential to detect the actual effect on renal parameters.

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