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Carbohydrate Polymers 62 (2005) 309–315

Carbohydrate Polymers

www.elsevier.com/locate/carbpol

The use of gum arabic and modified starch in the microencapsulation of a food flavoring agent

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Received 13 September 2004; revised 4 October 2004; accepted 10 March 2005
Available online 09 November 2005

Abstract

Solvent extracted oleoresins exhibit a flavor profile, close to the freshly ground spice in a wide spectrum of foods. In spite of their many advantages over ground spices, their sensitivity to light, heat and oxygen is a disadvantage. One approach to overcome this disadvantage is by means of microencapsulation. The present work reports on microencapsulation of cardamom oleoresin by spray drying using gum arabic, maltodextrin, and a commercially available modified starch as wall materials. The microcapsules were evaluated for the content and stability of volatiles, non-volatiles, entrapped 1,8-cineole and entrapped α-terpinyl acetate for 6 weeks. Gum arabic offered greater protection to the oleoresin than maltodextrin and modified starch, as seen from the $t_{1/2}$, time required for a constituent to reduce to 50% of its initial value.
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Keywords: Cardamom oleoresin; Gum arabic; Modified starch; Microencapsulation

1. Introduction

Spices have occupied an important place in the world of commerce for many centuries. Among the major spices, India leads in area and production of black pepper, cardamom (large), ginger, chilli and turmeric (Sasikumar & Sharma, 2001). Spice oils and oleoresins are value added products that are produced worldwide (Premi, 2000). With most spices, the total extracts or oleoresins are known to reflect the flavor quality more closely than the distilled volatile oil (Govindarajan, Narasimhan, Raghuveer, & Lewis, 1982). Oleoresins are the solvent extracts of a spice, which contain both volatile components, such as essential oils; and non-volatile components, such as fixed oils, antioxidants, and pigments. It is the balance of volatile to non-volatile resinous matter that is the best indication of quality and this should be closely similar to that in the original spice. They gained acceptability during 1970s as common ingredients for the food manufacturer and their production and consumption have been steady over the last few years (Pagington, 1983).

Cardamom is known for centuries for the enchanting taste and flavor it imparts to the food (Menon, 2000). Cardamom, known as the ‘Queen of spices’, is the dried fruit of *Elletaria cardamomum*, a perennial herbaceous plant of the order of Zingiberaceae (Mathai, 1985). The cardamom flavor is incorporated in processed foods, mainly by using the hydro distilled cardamom oil or the solvent-extracted cardamom oleoresin (Govindarajan et al., 1982). Solvent extraction of ground seeds of cardamom gives a greenish oleoresin containing about 70% volatile oil. It has the full flavor of the spice. The cardamom seeds are used in items subjected either to high temperature (above 149 °C) or to high acid concentration (below pH 4). At an elevated temperature, changes may occur in the volatile constituents. Many pastry products that utilize cardamom are exposed to oven temperatures from 149 to 205 °C for 5–90 min. Some beverages utilizing cardamom have pH values as low as 2.8 and are exposed to simmering conditions for 15–25 min. Cardamom is frequently used in the seasoning in fruit dishes that have a pH range from 2.6 to 4.6. Chemical changes, which could affect flavor, might be expected in cardamom, since the volatile oils which are credited as being responsible for the flavor of this spice consist of terpenoids. Terpenoids are, generally, unstable in the presence of acid, light, oxygen or heat, and are capable of undergoing hydrolysis, rearrangement, polymerization, and oxidative

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doi:10.1016/j.carbpol.2005.03.020

reactions. Due to such detrimental conditions there is an increase in *p*-cymene, a terpene with petroleum like aroma. Further, this increase in *p*-cymene apparently occurs at the expense of the major constituent, α-terpinyl acetate, a compound, which contributes to the desirable flavor of this spice (Brennand & Heinz, 1970). These problems are overcome by microencapsulation, which is defined as the technique of packing minute particles of a core material within a continuous polymer film that is designed to release its contents in a predictable manner under a predetermined set of conditions.

Microencapsulation protects the oleoresin against destructive changes, and also converts it into a free-flowing powder. Besides, it also protects the flavors from undesirable interaction with food and minimizes flavor/flavor interaction (Reineccius, 1989; Versic, 1988). The microcapsules may range from several millimeters in size (0.2–5000 μm) and have multitudes of shapes, depending on the materials and methods used to prepare them (Balassa & Fanger, 1971). The simplest of the microcapsules may consist of a core surrounded by a wall or barrier of uniform or non-uniform thickness. The core may be composed of just one or several different types of ingredients and the wall may be single or double-layered. The retention of these core is governed by factors such as its chemical nature including the chemical functionality, relative volatility and polarity (Goubet, Leouere, & Voilley, 1998). Controlled diffusion, breakage of the capsules, dissolution and solvent effects can cause losses of the encapsulate. The degree of protection is, generally, calculated as the rate of its loss from the microcapsules.

A number of methods are reported for microencapsulation of flavors, but the most popular technique employed in the industry is spray drying. Spray drying technique is quite suited in the encapsulation of oils and oleoresins (Raghavan, Abraham, & Shankaranarayana, 1990; Sankarikutty, Sreekumar, Narayanan, & Mathew, 1988). Carbohydrates such as hydrolyzed starches (SHPs), emulsifying starches and gums (especially gum acacia) are the most common carrier materials (Reineccius, 1988, 1989). Being hydrophilic in nature, SHPs have little affinity for hydrophobic flavor oils. Their hydrophilic nature can be changed by modifying them with *n*-octenyl succinic anhydride (*n*-OSA). *n*-OSA treated starch contains hydrophobic octenyl side chains, which impart emulsifying capability to the starches. Most *n*-OSA starches used for encapsulation are depolymerized to lower the viscosity (Qi & Xu, 1999). Gums are also used for secondary effect such as stabilization of emulsion, encapsulation, and film formation. The flavor industry uses gum arabic as a fixative in spray drying applications where the gum encapsulates the flavor compound, protecting it from oxidation and volatilization. Recovery and oxidation stability of orange oil encapsulated in gum arabic and emulsifying starches by spray drying has been reported (Qi & Xu, 1999).

Reports on microencapsulation of spice oleoresins in scientific literature are scant. Microencapsulation of garlic oleoresin by spray drying using edible gums as wall material has been described (Xiang, Yang, Li, Wang, & Cheng, 1997). Zilberboim, Kopelman, and Talman (1986) utilized spray-drying process to encapsulate paprika oleoresin and several volatile esters in gum arabic. Microencapsulation of capsicum oleoresin in a gum mixture composed of carrageenan and maltodextrin at a ratio of 0.5–3.5:9.5–7.0 was studied (Xiang et al., 1997). Microencapsulation of red pepper oleoresin using gum arabic and modified starch has also been tried (Jung & Sung, 2000). The present work reports on the efficacy of three carbohydrate materials viz. gum arabic, maltodextrin and a commercially available modified starch (starch octenyl succinate derivative marketed under the trade name of ‘Hi Cap’ by National Starch and Chemicals Corporation, a company of the ICI group) as wall materials for encapsulation of cardamom oleoresin.

2. Materials and methods

2.1. Materials

Gum arabic was obtained as gift sample from TIC Gums, USA. Modified starch (Hi Cap100) obtained was from National Starch Chemicals Corporation, Mumbai. Cardamom oleoresin was procured from Synthite Chemicals, Kerala. Standard 1,8-cineole was procured from Balsara Chemicals, Mumbai and standard terpinyl acetate was procured from Beta Chemicals, Mumbai. Tween-80 was procured from E. Merck (India). All chemicals used were of AR grade.

2.2. Methods

2.2.1. Analysis of the cardamom oleoresin and stability

1,8-Cineole content of cardamom oleoresin was determined using UV spectrophotometer by measuring the absorbance at 270 nm (Angadi, Ravikumar, Rajeevalochan, Kumar, & Shankaranarayana, 2002). α-Terpinyl acetate content of cardamom oleoresin was determined using UV spectrophotometer by measuring the absorbance at 236 nm (company specification). Total volatiles and non-volatiles were analyzed as per the reported method (Ranganna, 1977). Oleoresin of 0.2 g was accurately weighed in an evaporating dish in a hot air oven at 100 °C for 45 min. The evaporating dish was cooled and weighed again. The loss in the weight corresponds to volatile oil and the mass left behind to that of the non-volatiles or fixed oils. All the results were expressed as % w/w of the oleoresin sample. The stability of 1,8-cineole, α-terpinyl acetate, volatiles and non-volatiles in terms of their % retention in oleoresin was evaluated over a period of 6 weeks

2.2.2. Preparation of microcapsules by spray drying

Thirty grams of gum arabic, maltodextrin, the commercial modified starch, i.e. Hi Cap 100 were dispersed individually in distilled water and final volume made to 100 ml. It was rehydrated for about 12 h at 10–12 °C, after which 1.5 g (5% of the carrier used) of oleoresin was added. The mixture was emulsified in a shear homogenizer for 5 min at 3000 rpm until complete dispersion of the oleoresin. Two drops of Tween 80 was added for proper emulsification. The slurry of carrier materials, water, oleoresin was spray dried in a Buchi-190 model mini spray dryer (Buchi, Switzerland) (inside chamber dimension: 100 cm height, 60 cm diameter) equipped with 0.5 mm diameter nozzle. The pressure of compressed air for the flow of the spray was adjusted to 5 bar. The inlet and outlet temperature was maintained at 178 ± 2 and 120 ± 5 °C, respectively. A peristaltic pump was used to feed the spray dryer at 300 g/h. The microcapsules so prepared were collected from the collecting chamber. These powders were filled in airtight, self-sealable polyethylene pouches and stored in a dessicator containing calcium chloride at 25 °C to prevent moisture absorption until further studies.

2.2.3. Analysis of spray dried microcapsules

2.2.3.1. Analysis of entrapped 1,8-cineole (EC), and entrapped α-terpinyl acetate (ETA). To evaluate the ability of these carrier materials as a flavor carrier, the spray-dried microcapsules were subjected to analysis for entrapped 1,8-cineole and entrapped α-terpinyl acetate by method given by Fagen, Kolen, and Hussong (1955) with slight modifications. Twenty grams of microcapsules were washed with 1 ml of absolute ethanol to wash the surface components. The residue left from above washing was dissolved in hexane; volume made to 10 ml in a standard volumetric flask, sonicated for 4–5 min, filtered and used to estimate entrapped 1,8-cineole by taking absorbance at 270 nm for 1,8-cineole (Angadi et al., 2002) and 236 nm for α-terpinyl acetate (company specification).

2.2.3.2. Analysis for total volatiles (TV) and non-volatile ether extract (NV). Although most substances encapsulated by spray drying or some other techniques are volatile, the entrapped phase is sometimes wholly or partially non-volatile. The concentration of this fraction can be calculated. The method presented here for determination of this portion meets the requirement of speed and ease of operation and yields reasonable accuracy with the equipments found in the average laboratory.

The method entails organic-solvent extraction of the non-volatile material from a water solution. The non-volatile entrapped phase is separated from the encapsulating agent and the amount extracted by the organic solvent can be determined by weighing the evaporation residue. Conversely, if it is assumed that only the encapsulating agent is water soluble, the concentration of this component in the sample can be ascertained by evaporation of the water phase of the extraction (Maleeny, 1961).

Two-hundred and fifty milligram of the sample was weighed accurately. To this 15 ml of diethyl ether and 5 ml distilled water was added, blended in a waring blender at 300 rpm for at least 30 s. The mixture was quantitavely washed with water into a separating funnel. This was allowed it to stand until the layers separated. The bottom layer was drawn into another separating funnel. To this fraction, a mixture of 10 ml diethyl ether and 5 ml ethyl alcohol was added, shaken well, and the phases were allowed to separate. The bottom water layer was drained into a weighed flask. The ether extracts were combined and washed with 10 ml portion of water to ensure complete

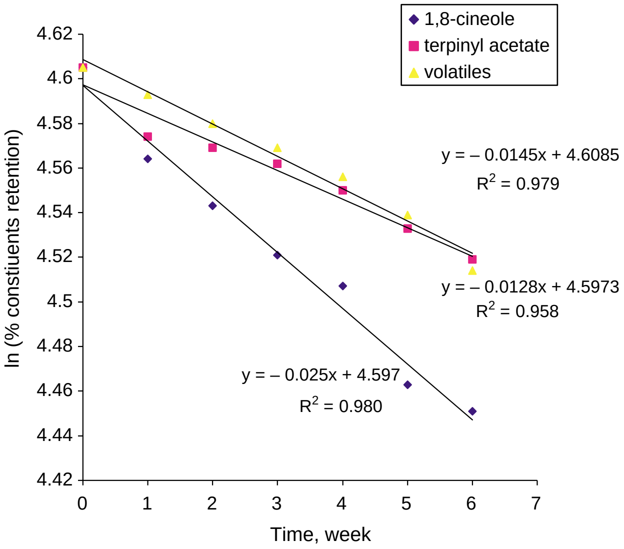

Fig. 1. Stability of cardamom oleoresin constituents stored at 25 °C.

Table 1
%Retention[a] of 1,8-cineole, α-terpinyl acetate, total volatiles and non-volatiles in oleoresin stored at 25 °C

Time (weeks)	1,8-Cineole	α-Terpinyl acetate	% TV	% NV
0	28.59	50.80	60.71	39.28
1	27.44	49.25	60.02	39.97
2	26.87	49.01	59.24	40.75
3	26.29	48.67	58.58	41.41
4	25.92	48.09	57.83	42.16
5	24.80	47.28	56.87	42.94
6	24.51	46.62	55.45	43.12

[a] Results are the mean of two individual determinations

Table 2
Half-life of 1,8-cineole, α-terpinyl acetate and volatiles in cardamom oleoresin stored at 25 °C

Constituents	Half-life, $T_{1/2}$ (weeks)
1,8-Cineole	27.72
α-Terpinyl acetate	60.78
Volatiles	47.79

Table 3
Recovery of constituents from cardamom oleoresin entrapped in microcapsules prepared from various wall materials

Carrier materials	% Recovery[a]													
	0 week		1 week		2 week		3 week		4 week		5 week		6 week	
	A	B	A	B	A	B	A	B	A	B	A	B	A	B
Gum arabic	52.59	62.09	52.59	62.09	52.59	62.09	52.01	61.57	52.01	61.57	51.96	61.05	51.96	60.54
Maltodextrin	42.39	52.26	42.07	51.22	41.76	50.70	41.44	49.67	41.12	49.67	39.84	49.15	39.52	49.15
Modified Starch	41.44	51.22	40.80	50.17	40.48	49.66	39.52	49.66	39.21	49.15	38.89	49.15	38.57	48.11

[a] Results are the mean of two individual determinations, where A, entrapped 1,8-cineole; B, entrapped α-terpinyl acetate. Oleoresin at 5% based on carrier material used.

Table 4
Recovery of total volatiles (TV) and non-volatiles (NV) from cardamom oleoresin entrapped in microcapsules prepared from various wall materials

Carrier Materials	% Recovery[a]													
	0 week		1 week		2 week		3 week		4 week		5 week		6 week	
	TV	NV	TV	NV	TV	NV	TV	NV	TV	NV	TV	NV	TV	NV
Gum Arabic	22.98	16.45	22.98	16.45	22.88	16.55	22.83	16.60	22.73	16.70	22.73	16.70	22.58	16.85
Maltodextrin	11.05	17.75	10.85	17.95	10.75	18.05	10.60	18.2	10.55	18.25	10.70	18.75	10.60	18.95
Modified Starch	14.92	16.45	14.77	16.60	14.57	16.80	14.42	16.95	14.32	17.05	14.27	17.10	14.17	17.20

[a] Results are the mean of two individual determinations.

removal of the water-soluble components. These materials were then transferred to the weighed flask to determine the water-soluble fractions. The combined ether extract was transferred to another weighed boiling flask, the solvents evaporated and the flask was dried for 2–4 h in an oven at 105 °C. The results were reported as non-volatile ether extract (NV) and % ether extract (EE) as follows:

(i) $\%\text{NV} = R_e/S \times 100$

In which, S, weight of sample; R_e, weight of ether soluble residue.

(ii) $\%\text{EE} = 100 + W - (R_w/S \times 100)$

In which, S, weight of sample; R_w, weight of water-soluble residue; W, water content of sample.

(iii) $\%\text{Volatiles} = \%\text{EE} - \%\text{NV}$

2.2.4. Stability of the EC, ETA, volatiles and non-volatile within the microcapsules

The samples were analyzed over a period of 6 weeks for entrapped 1,8-cineole (EC), entrapped terpinyl acetate (ETA), and also for total volatiles (TV) and non-volatiles (NV). The percentage retention of all these analytes was calculated by the formula (analyte at 'X' storage time) × 100/ (analyte at zero storage time). A semi-log plot of percentage retention of all these analytes vs. time according to Cai and Corke (2000) was done to obtain the rate constant (k) as the slope of the graph. Half-life ($t_{1/2}$) for the retention of 1,8-cineole and α-terpinyl acetate was calculated from the rate constant as $0.693/k$.

2.2.5. Scanning electron microscopy (SEM)

Particle size and structure of spray-dried microcapsules were evaluated with scanning electron microscope, Philips XL 30, (Netherlands). The microcapsules were mounted on specimen stubs with double sided adhesive carbon tapes.

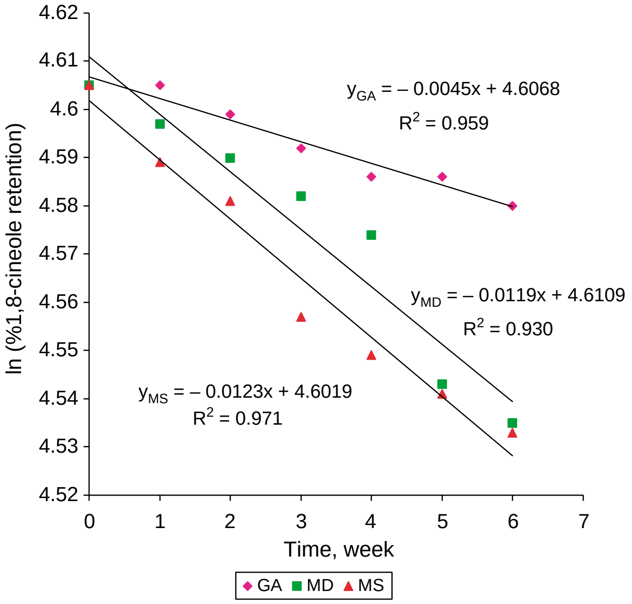

Fig. 2. Stability of entrapped 1,8-cineole (EC) in microencapsulated cardamom oleoresin prepared from carrier materials under study stored at 25 °C.

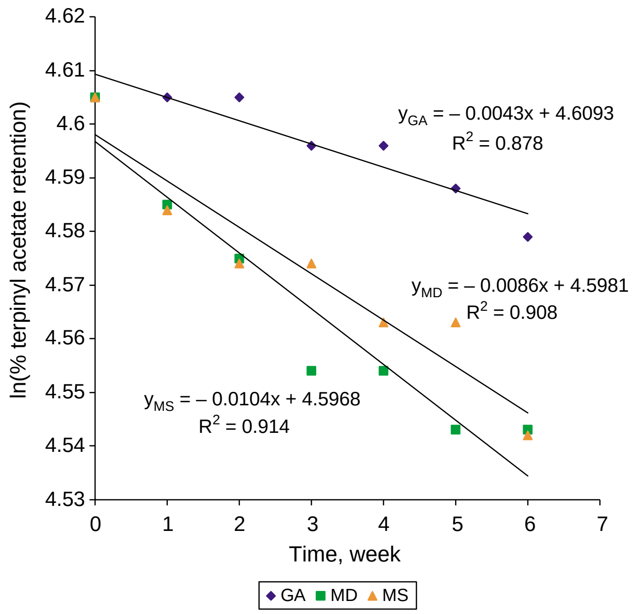

Fig. 3. Stability of entrapped α-terpinyl acetate (ETA) in microencapsulated cardamom oleoresin prepared from carrier materials under study.

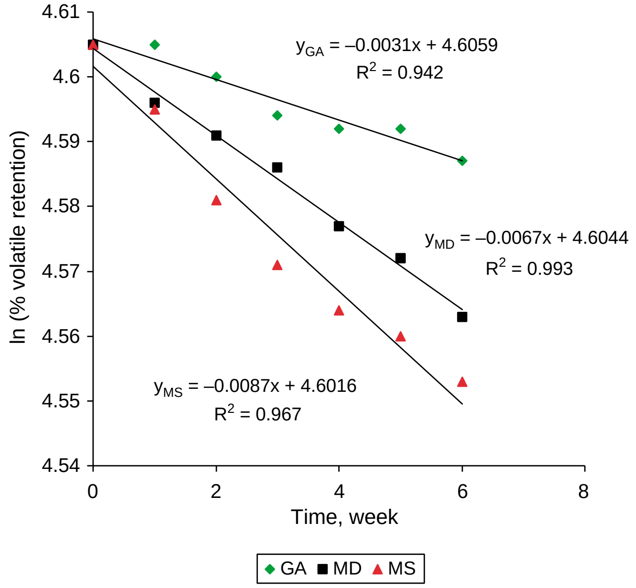

Fig. 4. Stability of total volatiles (TV) in microencapsulated cardamom oleoresin prepared from carrier materials under study.

The specimen was coated with gold–palladium and examined at 10 kV.

3. Results and discussion

3.1. Analysis and stability of EC, ETA, NV and TV in free and encapsulated oleoresin

The cardamom oleoresin was a commercial sample obtained from Synthite Chemicals, Kerala. It was analyzed for total volatiles (60.71%) and non-volatiles (39.28%). The oleoresin showed 28.58% of 1,8-cineole and 50.80% of α-terpinyl acetate. This oleoresin was stored at 25 °C in a glass bottle and studied weekly for 6 weeks. Table 1 represents the % retention of 1,8-cineole, α-terpinyl acetate, total volatiles and non-volatiles at 25 °C. The 1,8-cineole content decreased from an initial value of 28.58–24.50% over a period of six weeks. The α-terpinyl acetate content decreased from an initial value of 50.80–46.61% over a similar period. Table 1 also represents the % retention of total volatiles and total non-volatiles over this period. Higher retention of α-terpinyl acetate and lower retention of cineole have been indicated, as cineole is more volatile when compared to α-terpinyl acetate (Sankarikutty et al., 1988). The results showed that there was an increase in the concentration of total non-volatiles as a result of the decrease in the concentration of total volatiles. To reduce the losses of volatiles from the oleoresin, storage under cold condition in a moisture free airtight container and encapsulation or adsorption on inert carrier material has been recommended (Gilbertson, 1971).

Fig. 1 shows a semi-log plot percentage of 1,8-cineole, percentage of α-terpinyl acetate and percentage of total volatiles vs. storage time at 25 °C for the free oleoresin. A sharp decrease in all the cases was observed. The linear nature of the graph indicated the decrease in these constituents to follow first order kinetics. This study reveals that oleoresins are sensitive to heat, and loses volatiles and characteristic components under the reported storage conditions. The half life, $t_{1/2}$, that is the time required for the reduction of a value to 50% of its original was calculated from the slope 'k' as $t_{1/2}=0.693/k$. Table 2 shows $t_{1/2}$ for 1,8-cineole, α-terpinyl acetate and volatiles in the cardamom

Table 5
Regression analysis of EC, ETA and TV in the microcapsules prepared from microcapsules prepared from various wall materials

	Gum arabic	$T_{1/2}$ weeks	Maltodextrin	$T_{1/2}$ weeks	Modified starch	$T_{1/2}$weeks
EC	$Y=-0.0045X+$ 4.606, $R^2=0.959$	154	$Y=-0.0119X+$ 4.610, $R^2=0.930$	58.23	$Y=-\ 0.0123X+$ 4.601, $R^2=0.971$	56.34
ETA	$Y=-0.0043X+$ 4.609, $R^2=0.878$	161.6	$Y=-0.0086X+$ 4.598, $R^2=0.908$	80.58	$Y=-0.0104X+$ 4.596, $R^2=0.914$	66.63
TV	$Y=-0.\ 0031X+$ 4.605, $R^2=0.948$	223.5	$Y=-0.0067X+$ 4.604, $R^2=0.983$	103.43	$Y=-0.0087+$ 4.601, $R^2=0.967$	79.65

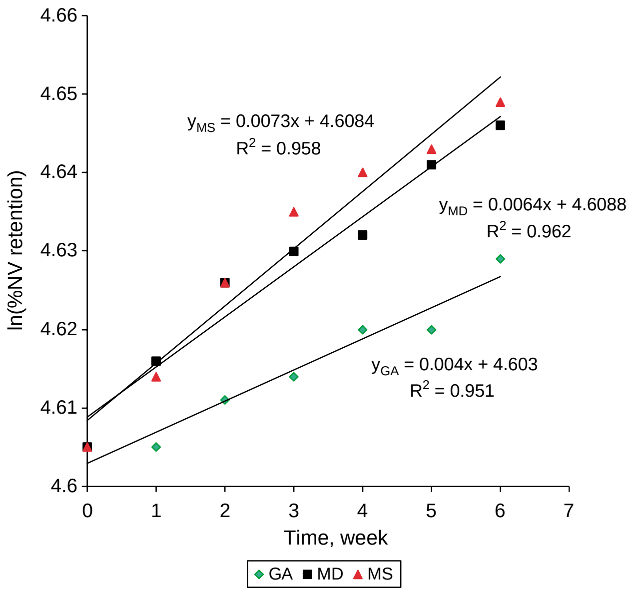

Fig. 5. Stability of non-volatiles (NV) in microencapsulated cardamom oleoresin prepared from carrier materials under study.

oleoresin stored at 25 °C. As seen, α-terpinyl acetate is more stable as compared to that of 1,8-cineole.

Oleoresin at 5% based on carrier material was mixed separately with 30% gum arabic, maltodextrin and modified starch slurry to prepare microcapsules. Microcapsules were prepared by spray drying using, gum arabic, maltodextrin and the modified starch (Hi-Cap 100, a starch octenyl succinate). The microcapsules that were obtained were analyzed for entrapped 1,8-cineole, entrapped α-terpinyl acetate (Table 3), volatiles and non-volatiles (Table 4). Gum arabic provided the maximum entrapment in terms of 1,8-cineole, α-terpinyl acetate and volatiles. The extent of entrapment of these constituents were almost similar for maltodextrin and modified starch. Figs. 2–4 gives the semi-log plot of percentage of retention of 1,8-cineole, α-terpinyl acetate and volatiles over a period of 6 weeks for the microcapsules prepared with the three wall materials under study. Again $t_{1/2}$ for each constituent in each of the wall materials was calculated and the results are seen in Table 5. Among these, the microcapsules obtained using gum arabic as carrier material provided greater protection as compared to that of maltodextrin and modified starch, which was seen from the half-life calculated. This was in agreement with the observation made by Reineccius (1989) and Raghavan, Abraham & Shankaranarayana (1990). The efficient entrapment of the constituents and volatiles in gum arabic is due to good film forming capability and their plastic rather than a glassy property. Plasticity is known to prevent cracking of the protection matrix (Bertolini, Siani, & Grosso, 2001; Sheu & Rosenberg, 1995; McNamee, O'Riordan, & O'Sullivan, 1998; Rosenberg, Kopelman, & Talmon, 1990; Sankarikutty et al., 1988). This explains the use of gum arabic by flavor industries as a fixative in spray drying applications, wherein the gum encapsulates the flavor compound and protects it from oxidation and volatilization (Qi & Xu, 1999) (Fig. 5).

3.2. SEM of microencapsulated cardamom oleoresin

Microcapsules prepared by spray drying of cardamom oleoresin using gum arabic, maltodextrins and the modified starch as wall materials were observed for size and shape from the SEM (Fig. 6). Microcapsules obtained from gum arabic were found to be nearly spherical but had many dents on the surface whereas the microcapsules obtained from maltodextrins and the modified starch were broken and not complete. A comparative study on gum arabic and modified starch for encapsulation of flavors showed a similar pattern of microcapsules (Buffo, Probst, Zehentbauer, Luo, & Reineccius, 2002; Rosenberg & Kopelman, 1985; Varavinit, Chaokasem, & Shobsngob, 2001). The SEM also indicated the suitability of gum arabic as a wall material for encapsulation of cardamom oleoresin as compared to maltodextrin and modified starch.

4. Conclusion

Gum arabic was found to be better wall material for encapsulation of cardamom oleoresin as compared to maltodextrins and modified starch. The protection offered to cardamom constituents was very clear from the experiment carried out in this work, although the same cannot be said for total volatiles. The free flowing nature of all these microcapsules is of advantage to the food processing

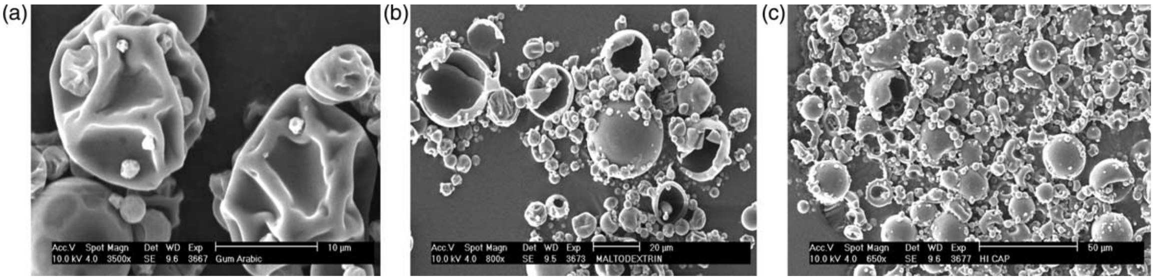

Fig. 6. SEM of the microcapsules prepared from (a) gum Arabic, (b) maltodextrin and (c) Hi-Cap.

industry. Work with other carrier materials-alone and in blends, and their physicochemical nature effecting the entrapment of such complex flavor systems need to be evaluated.

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