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Microencapsulation of cardamom oleoresin: Evaluation of blends of gum arabic, maltodextrin and a modified starch as wall materials

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Abstract

Although the spice oleoresins provide complete flavour profile than their respective essential oils, their sensitivity to the light, heat and oxygen is a disadvantage. This can be overcome by effective encapsulation. The present work reports on the microencapsulation of cardamom oleoresin by spray drying using binary and ternary blends of gum arabic, maltodextrin, and modified starch as wall materials. The microcapsules were evaluated for the content and stability of volatiles, entrapped 1,8-cineole and entrapped α-terpinyl acetate for 6 weeks. A 4/6,1/6,1/6 blend of gum arabic:maltodextrin:modified starch offered a protection, better than gum arabic as seen from the $t_{1/2}$, time required for a constituent to reduce to 50% of its initial value.

Keywords: Cardamom oleoresin; Gum arabic; Modified starch; Encapsulation

1. Introduction

Spices form a major class of ingredients used in most food products today. Among the major spices, India leads in area and production of black pepper, cardamom (large), ginger, chilli and turmeric (Sasikumar & Sharma, 2001). 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 flavour is incorporated in processed foods, mainly by using cardamom essential oil or the solvent-extracted cardamom oleoresin. With most spices, the total extracts or oleoresins are known to reflect the flavour quality more closely than the distilled volatile oil. Commercial cardamom oleoresins have volatile oil contents ranging between 52 and 58% (Govindarajan, Narasimhan, Raghuveer, & Lewis, 1982). The major compounds present in the cardamom oleoresin are 1,8-cineole and α-terpinyl acetate comprising two-third of the total volatiles (Lewis, Nambudiri, & Philip, 1966). Cardamom oleoresin has a more mellow and less harsh flavour characteristic of cardamom (Krishnan, 1981; Sankarikutty, Sreekumar, Narayanan, & Mathew, 1988), and is used in the flavouring of bakery, confectionery and meat products.

These spice extracts are used either in the form of liquid concentrate or adsorbed on a carrier (Gilberston, 1971). In both cases oleoresin undergoes oxidative degradation. It was found that destruction of several pigments occur under exposure to oxygen:hydroxylic groups are converted into unstable ketones, which in turn decomposes. Apart from the fading effect, the oxidation products and intermediates (such as peroxides) may have a detrimental effect on the foodstuffs themselves. Hence, there is a need for protection of the oleoresin against environmental factors, which contributes to its deterioration, e.g. oxygen, light, moisture. Microencapsulation using carbohydrates such as hydrolysed starches (SHP), emulsifying starches and gums (especially gum acacia), serve as the most common carrier materials (Reineccius, 1988, 1989; Rosenberg, Kopelman & Talmon, 1990; Verswic, 1988) is most common approach to tackle this problem. These encapsulated flavours are prepared by locking the natural spice extractives into special matrix resulting in improved heat stability and shelf life. The spray drying technique is quite suited for the encapsulation of spice oils and oleoresins (Raghavan, Abraham, & Shankar-anarayana, 1990; Sankarikutty, Sreekumar, Narayanan,

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& Mathew, 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). Spray-dry microencapsulation has been used to improve the stability of carotenoids in carrot pulp and paprika oleoresin (Leach, Oliveria, & Morias, 1986).

Gum arabic is a very effective encapsulating agent because of its protective colloid functionality. It produces stable emulsions with most oils over a wide pH range. It also forms a visible film at the oil interface, but the mechanism of emulsification is still not understood. Gum arabic is compatible with most gums, starches and carbohydrates and proteins. Cost and limited supply have been restricted the use of gum arabic for encapsulation. Hence, maltodextrins and modified starches were used as alternative carrier materials (Reineccius, 1988). Maltodextrins have been investigated as replacers of gum arabic in spray dried emulsions (Anandaraman & Reineccius, 1987; Bangs & Reineccius, 1988; Kenyon & Anderson, 1988; Trubiano & Lacourse, 1988). Mixture of gum arabic and maltodextrin was reported effective in microencapsulation of cardamom oil using spray drier (Sankarikutty, Sreekumar, Narayanan, Mathew, 1988). The major shortcomings of maltodextrin are its lack of emulsifying capacity and marginal retention of volatiles (Buffo & Reineccius, 2000; Reineccius, 1988). Retention of volatile flavour compounds increases with an increase of DE of the maltodextrins (Anandaraman & Reineccius, 1986; Bangs & Reineccius, 1981) suggesting the importance of DE to the functionality of wall system. The chemically modified starches most closely reproduce the functional properties of gum arabic. 1-Octenyl succinyl anhydride (*n*-OSA) starches stabilize an emulsion through a number of possible mechanisms. *n*-OSA starches are reported to be superior to gum acacia in emulsification properties and in retention of volatile flavours during spray drying (Trubiano & Lacourse, 1988). The products encapsulated in gum arabic showed a reduction in content during the shelf life study at controlled temperature (Bertolini, Siani, & Grosso, 2001). However, the modified starches have some disadvantages, they are not considered natural for labeling purposes, often have an undesirable off-taste and do not afford good protection to oxidizable flavourings (Qi & Xu, 1999). Reports on micro-encapsulation 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, & 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, Yang, Li, Wang, & Cheng, 1977). Microencapsulation of red pepper oleoresin using gum arabic and modified starch has also been tried (Jung & Sung, 2000).

There are no reports on encapsulation of cardamom oleoresin. In the light of this information, an attempt has been made to encapsulate cardamom oleoresin by spray drying using gum arabic, modified starch-HiCap100 and maltodextrin as the wall materials. Our previous work (Krishnan, Bhosale, & Singhal., unpublished work) had evaluated the different wall materials individually for encapsulation of cardamom oleoresin, and found gum arabic to be better than maltodextrin and the modified starch. The present work deals with the microencapsulation of cardamom oleoresin by spray drying using binary and ternary blends of gum arabic, maltodextrin, and modified starch as wall materials. The microcapsules were evaluated for the content and stability of volatiles, entrapped 1,8-cineole and entrapped α-terpinyl acetate.

2. Materials and methods

2.1. Materials

Gum arabic was obtained as gift sample from TIC Gums, USA. Octenyl succinylated waxy maize starch (HiCap100) obtained was from National Starch Chemicals Corporation, Mumbai. Cardamom oleoresin was gifted from Synthite Chemicals, Kerala, India. Standard 1,8-cineole was gift samples from Balsara Chemicals, Mumbai and standard α-terpinyl acetate was purchased from Beeta Chemicals, Mumbai. All chemicals used were of AR grade.

2.2. Methods

2.2.1. Preparation of microcapsules by spray drying

About 30% solution of the different blends of gum arabic, maltodextrin and the commercial modified starch, i.e. HiCap100 were dispersed in distilled water and final volume made to 100 ml. It was kept for rehydration for about 12 h at refrigerated temperature (10–12 °C). About 1.5 g (5% of the carrier used) of oleoresin was added to the mixture and emulsified in a shear homogenizer (Indofrench Industries Engineers, Mumbai, Model type, SPM-9) for 5 min at 3000 rpm until complete dispersion. Two drops of Tween 80 was added to aid emulsification. The resulting slurry was spray dried in a Büchi-190 model mini spray dryer (Büchi, 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 adjusted to 5 bar. The inlet and outlet temperature was maintained at 178 ± 2 and 120 ± 5 °C, respectively, and feed rate was 300 g/h. The microcapsules prepared were collected and filled in airtight, self-sealable polyethylene pouches and stored in a dessicator until further studies.

2.2.2. Analysis of spray dried microcapsules

2.2.2.1. Analysis of entrapped 1,8-cineole (EC), and entrapped α-terpinyl acetate (ETA). To evaluate the ability of these carrier materials as a flavour 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, & Hussong (1955) with slight modifications. About 20 mg of microcapsules were washed with 1 ml of absolute ethanol to wash the surface components. The residue left from above washing was dispersed 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 (Angadi, Ravikumar, Rajeevalochan, Kumar, & Shankaranarayana, 2002) and absorbance at 236 nm for α-terpinyl acetate estimation.

2.2.2.2. Analysis for total volatiles (TV) and non-volatile ether extract (NV). 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).

About 250 mg of the sample was weighed accurately. To this 15 ml of diethyl ether and 5 ml distilled water was added, blended in a shear homogenizer at 300 rpm for atleast 30 s. The mixture was quantitatively washed with water into a separating funnel. 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 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 preweighed 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:

$$\%\mathrm{NV} = R_e/S \times 100$$

In which, S is the weight of sample, and R_e is the weight of ether soluble residue.

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

In which, S is the weight of sample, R_w is the weight of water-soluble residue, and W is the water content of sample.

$$\%\mathrm{Volatiles} = \%\mathrm{EE} - \%\mathrm{NV}$$

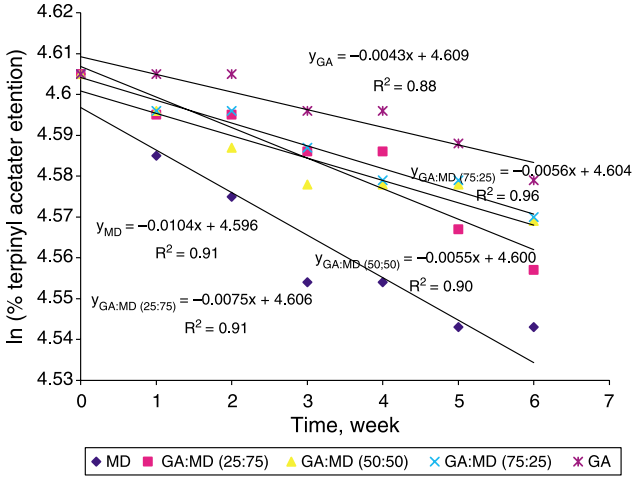

Fig. 2. Stability of entrapped α-terpinyl acetate (ETA) in microencapsulated cardamom oleoresin from gum arabic–maltodextrin blends.

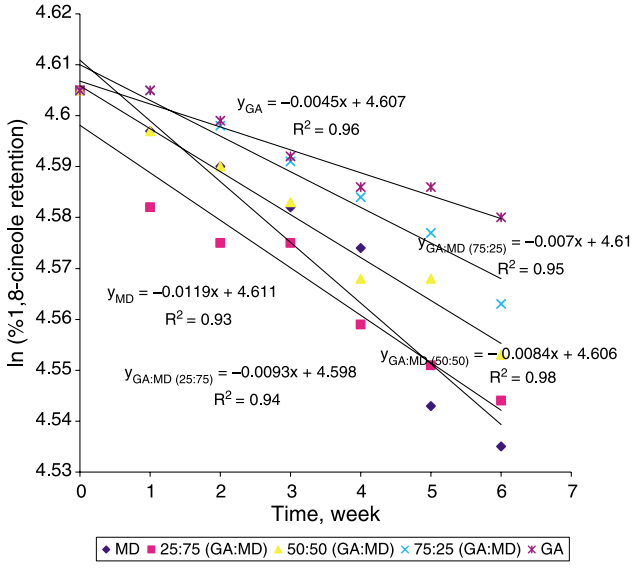

Fig. 1. Stability of entrapped 1,8-cineole (EC) in microencapsulated cardamom oleoresin from gum arabic–maltodextrin blends.

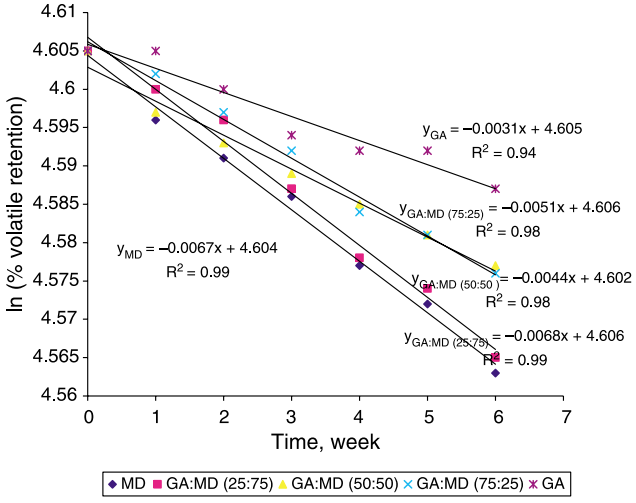

Fig. 3. Stability of total volatiles (TV) in microencapsulated cardamom oleoresin from the gum arabic–maltodextrin blends.

Table 1
Regression analysis of EC, ETA and TV in the microcapsules prepared from gum arabic:maltodextrin blend as wall materials

Encapsulating material	Constituent in microencapsulated cardamom oleoresin					
	EC		ETA		TV	
	Regression equation	$t_{1/2}$, weeks	Regression equation	$t_{1/2}$, weeks	Regression equation	$t_{1/2}$, weeks
GA:MD (0:100)	$Y=-0.0119X+4.610$, $R^2=0.93$	58.23	$Y=-0.0086X+4.598$, $R^2=0.91$	80.58	$Y=-0.0067X+4.604$, $R^2=0.98$	103.43
GA:MD (25:75)	$Y=-0.0093X+4.598$, $R^2=0.94$	74.51	$Y=-0.0075X+4.606$, $R^2=0.91$	92	$Y=-0.0068X+4.606$, $R^2=0.99$	101.91
GA:MD (50:50)	$Y=-0.0084X+4.605$, $R^2=0.98$	82.5	$Y=-0.0055X+4.600$, $R^2=0.90$	126	$Y=-0.0044X+4.602$, $R^2=0.98$	130.75
GA:MD (75:25)	$Y=-0.007X+4.61$, $R^2=0.95$	99	$Y=-0.0056X+4.604$, $R^2=0.96$	123.75	$Y=-0.0051X+4.606$, $R^2=0.99$	135.88
GA:MD (100:0)	$Y=-0.0045X+4.606$, $R^2=0.96$	154	$Y=-0.0043X+4.609$, $R^2=0.88$	161.16	$Y=-0.0031X+4.605$, $R^2=0.95$	223.5

2.2.3. Stability of the EC, ETA, volatiles (TV) and non-volatile (NV) 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 & 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.

2.2.4. 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.

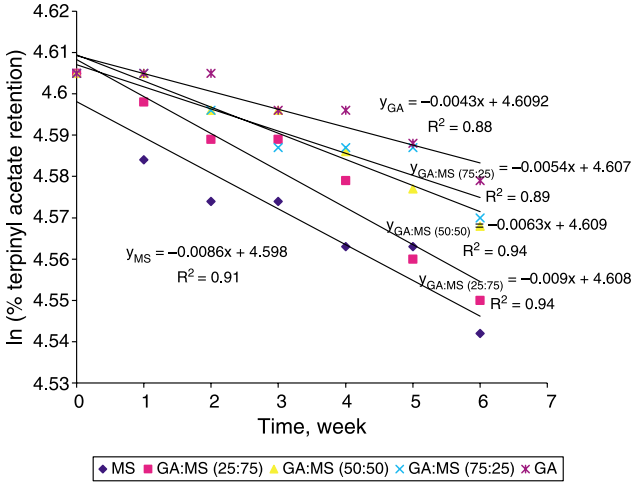

Fig. 5. Stability of entrapped α-terpinyl acetate (ETA) in microencapsulated cardamom oleoresin from the gum arabic–modified starch blends.

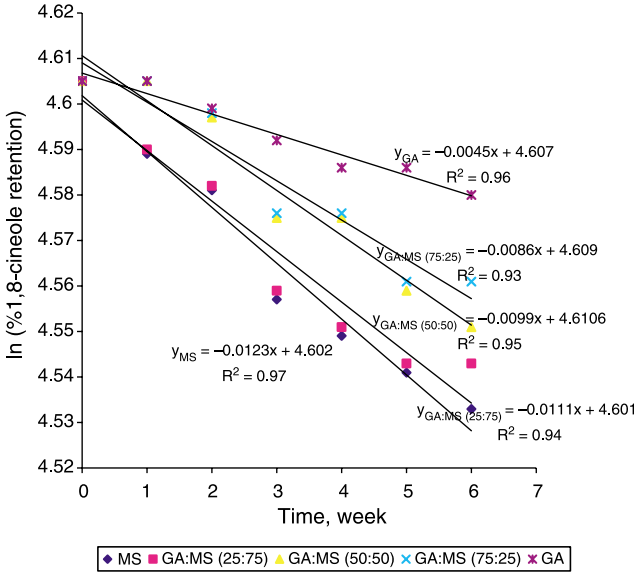

Fig. 4. Stability of entrapped 1,8-cineole (EC) in microencapsulated cardamom oleoresin from the gum arabic–modified starch blends.

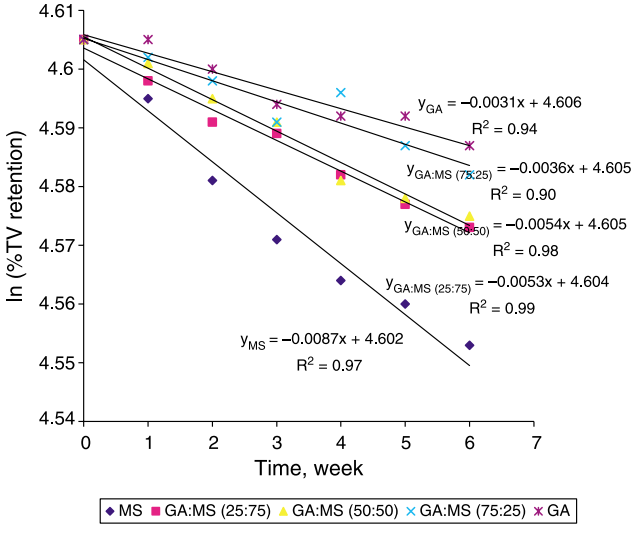

Fig. 6. Stability of total volatiles (TV) in microencapsulated cardamom oleoresin from the gum arabic–modified starch blends.

Table 2
Regression analysis of EC, ETA and TV in the microcapsules prepared from gum arabic:modified starch blend as wall materials

Encapsulating material	Constituent in microencapsulated cardamom oleoresin					
	EC		ETA		TV	
	Regression equation	$t_{1/2}$, weeks	Regression equation	$t_{1/2}$ weeks	Regression equation	$t_{1/2}$ weeks
GA:MS (0:100)	$Y=-0.0123X+4.601$, $R^2=0.97$	56.34	$Y=-0.0104X+4.596$, $R^2=0.91$	66.63	$Y=-0.0087+4.601$, $R^2=0.97$	79.65
GA:MS (25:75)	$Y=-0.0111X+4.600$, $R^2=0.94$	62.43	$Y=-0.009X+4.608$, $R^2=0.94$	77	$Y=-0.0053X+4.603$, $R^2=0.99$	130.75
GA:MS (50:50)	$Y=-0.0099X+4.610$, $R^2=0.95$	70	$Y=-0.0063X+4.609$, $R^2=0.94$	110	$Y=-0.0054X+4.605$, $R^2=0.98$	128.33
GA:MS (75:25)	$Y=-0.0086X+4.609$, $R^2=0.93$	80.58	$Y=-0.0056X+4.604$, $R^2=0.89$	123.75	$Y=-0.0036X+4.605$, $R^2=0.90$	192.5
GA:MS (100:0)	$Y=-0.0045X+4.606$, $R^2=0.96$	154	$Y=-0.0043X+4.609$, $R^2=0.88$	161.16	$Y=-0.0031X+4.605$, $R^2=0.95$	223.5

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 analysed for total volatiles (60.71%) and non-volatiles (39.28%). It also showed 28.58% of 1,8-cineole and 50.80% of α-terpinyl acetate. This oleoresin evaluated for six weeks for the levels of percentage retention of 1,8-cineole, terpinyl acetate, total volatiles and non-volatiles. A semi-log plot of %1,8-cineole, %α-terpinyl acetate and %volatiles vs. storage time showed a sharp linear decrease in all the cases, indicating the decrease in these constituents to follow first-order kinetics. The $t_{1/2}$ for 1,8-cineole, α-terpinyl acetate and volatiles in the cardamom oleoresin stored at 25 °C was 27.72, 60.78 and 47.79 weeks, respectively, indicating α-terpinyl acetate to be more stable as compared to 1,8-cineole (Krishnan, Bhosale, & Singhal, unpublished work).

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 maltodextrin, gum arabic, modified starch and the different blends of these three.

Different proportions of gum arabic and maltodextrins were tried such as 25:75 (GA:MD), 50:50 (GA:MD), 75:25 (GA:MD) as wall materials and microcapsules obtained were compared to those prepared with the individual wall material. Figs. 1–3 give the results over a 6-week storage for 1,8-cineol, α-terpinyl acetate and total volatiles, respectively. The half-life of retention in the said microcapsules (Table 1) is higher in blends containing higher proportions of gum arabic. Mixture of gum arabic and maltodextrin was reported effective in microencapsulation of cardamom oil using spray drier (Sankarikutty, Sreekumar, Narayanan, & Mathew, 1988). It was seen that as the concentration of the gum arabic increased, the half-life of the constituents increased.

The same procedure was followed for the modified starch. Different proportions of gum arabic and modified starch were tried such as 25:75 (GA:MS), 50:50 (GA:MS) and 75:25 (GA:MS), and the microcapsules so prepared were studied for EC, ETC and TV for a period of 6 weeks (Figs. 4–6). It was seen that the combination containing higher proportions of gum arabic provided better entrapment of the constituents. The half-life of retention in the said microcapsules are given in Table 2. Again, blends containing higher proportions of gum arabic provided maximum protection. It was also seen that as the concentration of

Table 3
Design centrum for blending gum arabic, maltodextrin and modified starch for effective encapsulation

Resulting coordinates of mixture experiment in the sub-region defined by pseudo-components			
Coordinates	Gum acacia (%)	Maltodextrin (%)	Modified starch (%)
(1/3,1/3,1/3)	49.75	25.125	25.125
(4/6,1/6,1/6)	74.50	12.75	12.75
(1/6,1/6,4/6)	36.25	12.75	51
(1/6,4/6,1/6)	36.25	51	12.75

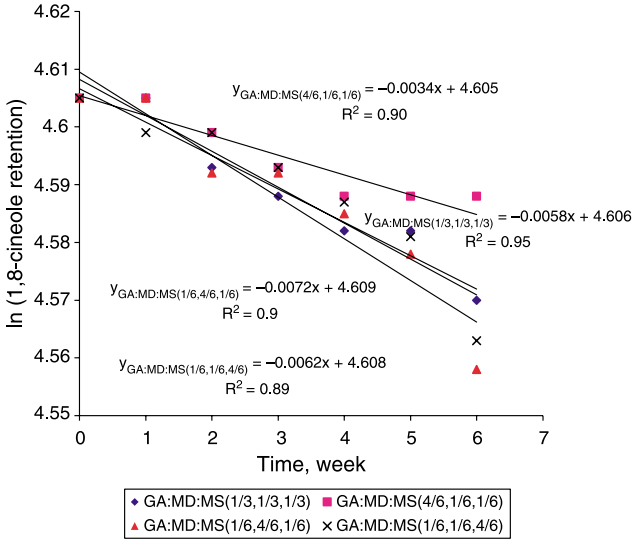

Fig. 7. Stability of 1,8-cineole in microncapsulated cardamom oleoresin from gum arabic–maltodextrin–modified starch blends.

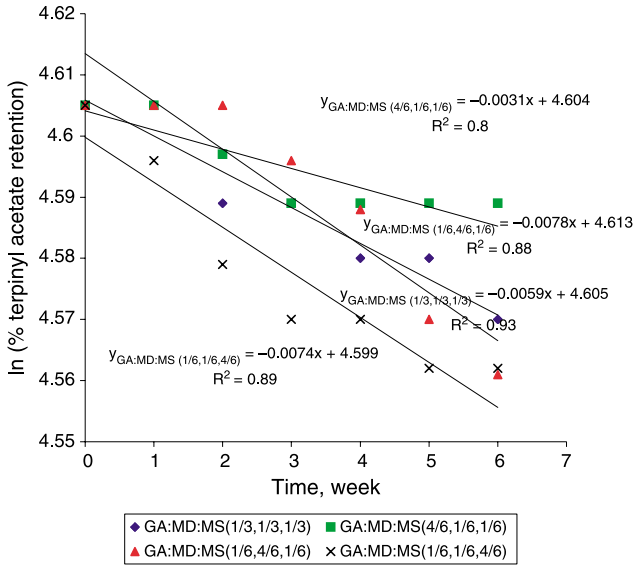

Fig. 8. Stability of 1,8-cineole in microncapsulated cardamom oleoresin from gum arabic–maltodextrin–modified starch blends.

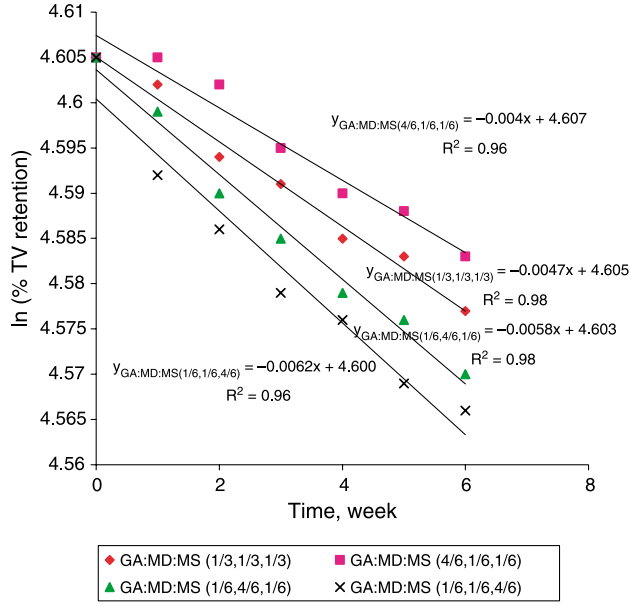

Fig. 9. Stability of total volatiles in microncapsulated cardamom oleoresin from gum arabic–maltodextrin–modified starch blends.

the gum arabic increased, the half-life of the constituents increased showing that the stability of the microcapsules increased.

In spite of trying so many combinations of the GA:MD and GA:MS, none of these combinations matched the protection offered by gum arabic. Blends of gum acacia, maltodextrins and modified starches may represent an another encapsulating matrix with improved properties regarding flavour retention, emulsion stability and protection against oxidation. Here, an augmented simplex–centroid mixture design was chosen to evaluate the blends of gum acacia, maltodextrin and modified starch as encapsulation carriers (Buffo & Reineccius, 2000). The design (centrum) for the experiment is shown in Table 3.

Here, four different blends of the gum arabic, maltodextrin and modified starch were carried out, and the microcapsules obtained were analysed for their EC, ETC and TV (Figs. 7–9). The half-life of retention in the said microcapsules are given in Table 4. From these results, it is evident that the GA:MD:MS (4/6,1/6,1/6) provided best protection to the cardamom oleoresin, in fact even much better than that provided by gum arabic alone. Other blends also performed equally quite well, suggesting the possibility of replacement of gum arabic partly by maltodextrin and modified starch. Here, half-life improved by adding both maltodextrin and modified starches.

3.2. SEM of microencapsulated cardamom oleoresin

Microcapsules of cardamom oleoresin using gum arabic, maltodextrin, modified starch and GA:MD:MS (4/6,1/6,1/6) blend as wall materials were observed for size and shape by SEM (Fig. 10). Microcapsules obtained from GA:MD:MS (4/6,1/6,1/6) were spherical and had a smooth surface. Microcapsules 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 partially disrupted (Buffo, Probst, Zehentbauer, Luo, & Reinneccius, 2002; Rosenberg, Koeplman, & Talmon, 1985; Varavinit, Chaokasem, & Shobsngob, 2001). The spherical and smooth surface show that suitability of the mixture for encapsulation. This was also confirmed by analysis of the stability results.

Table 4
Regression analysis of EC, ETA and TV in the microcapsules prepared from the blends of gum arabic, maltodextrin and modified starch

	GA:MD:MS (1/3,1/3,1/3)	$t_{1/2}$, weeks	GA:MD:MS (4/6,1/6,1/6)	$t_{1/2}$, weeks	GA:MD:MS (1/6,4/6,1/6)	$t_{1/2}$, weeks	GA:MD:MS (1/6,1/6,4/6)	$t_{1/2}$, weeks
EC	$Y=-0.0058X+4.606$, $R^2=0.89$	119.48	$Y=-0.0034X+4.605$, $R^2=0.90$	231	$Y=-0.0072X+4.609$, $R^2=0.90$	96.25	$Y=-0.0062X+4.608$, $R^2=0.95$	111.77
ETA	$Y=-0.0059X+4.605$, $R^2=0.93$	117.45	$Y=-0.0031X+4.604$, $R^2=0.80$	223.54	$Y=-0.0078X+4.613$, $R^2=0.89$	88.84	$Y=-0.0074X+4.599$, $R^2=0.88$	93.64
TV	$Y=-0.0047X+4.605$, $R^2=0.98$	147.44	$Y=-0.004X+4.607$, $R^2=0.96$	173.25	$Y=-0.0058X+4.603$, $R^2=0.96$	119.48	$Y=-0.0062X+4.600$, $R^2=0.99$	111.77

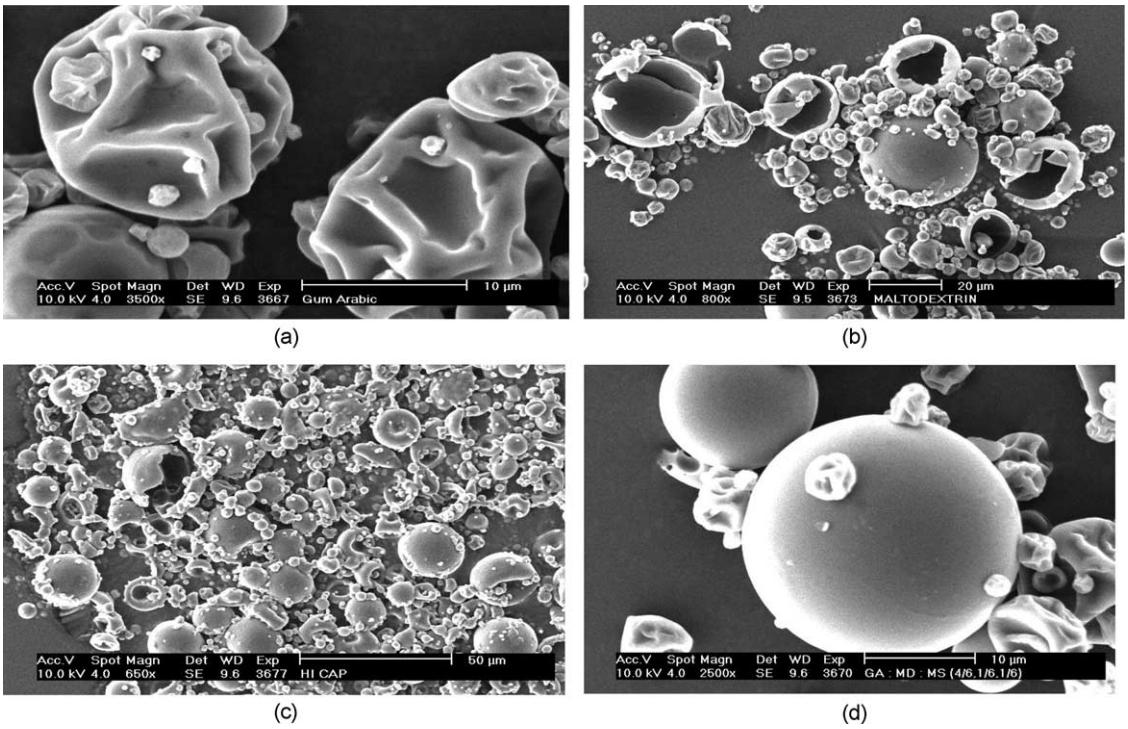

Fig. 10. SEM of the microcapsules prepared from (a) gum arabic, (b) maltodextrin, (c) modified starch HiCap100 and (d) the blends of all the three GA:MD:MS (4/6,1/6,1/6).

4. Conclusion

The results obtained in the present work indicate gum arabic to be a better wall material for encapsulation of cardamom oleoresin as compared to the other wall materials. As far as the blends were concerned, the stability of the cardamom oleoresins decreased as the quantity of gum arabic decreased in its blend with maltodextrin and HiCap100. GA:MD:MS (4/6,1/6,1/6) blend proved to be more efficient than the other blends even better than 100% gum arabic.

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