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ScienceDirect
Carbohydrate Polymers 67 (2007) 536–541
Carbohydrate Polymers
www.elsevier.com/locate/carbpol

Stability of cumin oleoresin microencapsulated in different combination of gum arabic, maltodextrin and modified starch

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Received 10 May 2006; received in revised form 14 June 2006; accepted 19 June 2006
Available online 22 August 2006

Abstract

Microencapsulations of cumin oleoresin by spray drying using gum arabic, maltodextrin, and modified starch (HiCap® 100) and their ternary blends as wall materials were studied for its encapsulation efficiency and stability under storage. The microcapsules were evaluated for the content and stability of volatiles, and total cuminaldehyde, γ-terpinene and *p*-cymene content for six weeks. Gum arabic offered greater protection than maltodextrin and modified starch, in general, although the order of protection offered was volatiles > cuminaldehyde > *p*-cymene > γ-terpinene. 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 $t_{1/2}$, i.e. time required for a constituent to reduce to 50% of its initial value. However protective effect of ternary blend was not similar for the all the constituents, and followed an order of volatiles > *p*-cymene > cuminaldehyde > γ-terpinene.

Keywords: Cumin oleoresin; Gum arabic; Modified starch; Maltodextrin; Microencapsulation

1. Introduction

India is the largest producer of cumin seed in the world. Not more than 4% of the seeds produced in India are exported, though 10–15% could be spared without eroding the country's requirements. Cumin seeds yield up to 4.55% volatile oil, of which 30–60% is cuminaldehyde. The flavour is warm, heavy, spicy, and curry like, dominated by cuminaldehyde. Cumin volatile oil contains over a dozen chemical components, includes terpenes (e.g. β-pinene, *p*-cymene, γ-terpinene) aldehydes (e.g. cuminaldehyde, 1,3-*p*-mentha and 3-*p*-menthen-7-al) and terpene alcohol (cuminyl alcohol) (Verghese, 1991). With most spices, the total extracts or oleoresins are known to reflect the flavour quality more closely than the distilled volatile oil (Govindarajan, Narasimhan, Raghuveer, & Lewis, 1982). Oleoresins are also free from many of the inherent disadvantages of the corresponding ground spices; in particular they are hygienic and can be standardized for acceptable flavour levels by blending. Unlike the essential oils, oleoresin contains natural antioxidants of the corresponding spices. But spice oils and oleoresins are so concentrated that they pose a number of problems in handling and use. Being immiscible in aqueous foods, they do not disperse well into the food matrix. Also flavour loss occurs when incorporated into dry food mixes during high temperature processing. Besides, they are sensitive to light, heat and oxygen, and hence have a short storage life if not stored properly. The microencapsulation method seems to be useful to solve these problems.

Microencapsulation of flavors in carrier matrices can provide protection against the degradative reaction, prevent loss of volatile flavors and enhance stability of the flavor core materials. Various techniques are employed to form microcapsules, including spray drying, spray chilling, extrusion coating, fluidized bed coating, liposome entrapment, coacervation, liposome entrapment, centrifugal extrusion and rotational suspension separation. The most common and economical way to carry out microencapsulation is spray

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doi:10.1016/j.carbpol.2006.06.023

drying. Numerous papers have been published about the encapsulation of liquid flavors by spray drying (Bhandari, Dumoulin, Richard, Noleau, & Lebert, 1992; Buffo & Reineccius, 2000; Finney, Buffo, & Reineccius, 2002; Liu et al., 2001; McNamee, O'Riordan, & O'Sullivan, 2001; Rosenberg, Kopelman, & Talmon, 1990; Soottitantawat, Yoshii, Furuta, Ohgawara, & Linko, 2003). There are very few reports on encapsulation of spice oleoresin by spray drying. 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 Talmon (1986) utilized spray-drying process to encapsulate paprika oleoresin and several volatile esters in gum arabic. Microencapsulation of capsicum oleoresin in a 0.5–3.5:9.5–7.0 ratio of gum mixture composed of carrageenan and maltodextrin (Xiang et al., 1997) and that of red pepper oleoresin using gum arabic and modified starch has also been tried (Jung & Sung, 2000). Our earlier work showed a 4/6:1/6:1/6 blend of gum arabic, maltodextrin and modified starch to have a better encapsulation efficiency and stability for cardamom oleoresin as compared to gum arabic (Krishnan, Bhosale, & Singhal, 2005).

The selection of wall material for spray drying is very vital for efficient encapsulation. Most common wall materials are gum arabic, maltodextrin, emulsifying starches and others, of which gum arabic is preferred for its encapsulation efficiency and stability. Gum arabic produces stable emulsions with most oils over wide pH range, also forms a visible film at the oil interface. Cost, limited supply and quality variations have restricted the use of gum arabic for encapsulation purpose. An area of research of increasing interest is the development of an alternative and inexpensive polymer or polymer blends, which could encapsulate flavors with same or greater efficiency as gum arabic. In recent reports, gum arabic in combination with maltodextrin and modified starch or alone are known to give a better encapsulation efficiency and stability than gum arabic (Anandaraman & Reineccius, 1987; Bangs & Reineccius, 1988; Kenyon & Anderson, 1988; Trubiano & Lacourse, 1988; Reineccius, 1988; You-Jin, Vasanthan, Temelli, & Song, 2003).

The present work deals with the microencapsulation of cumin oleoresin by spray drying using gum arabic, maltodextrin, and modified starch using gum arabic modified starch (HiCap® 100) and maltodextrin as well as their ternary blends as wall materials. The microcapsules that were obtained were analyzed for the content and stability of their total cuminaldehyde (TC), γ-terpinene (TGT), *p*-cymene (TPC) and volatiles. Scanning electron microscopy was used to study the morphological characteristics of the microcapsules.

2. Materials and methods

2.1. Materials

Gum arabic was obtained as gift sample from TIC Gums, USA. Modified starch (HiCap® 100) obtained was from National Starch Chemicals Corporation, Mumbai. Maltodextrin DE-18 was procured from Raptokos Brett & Co., Mumbai. Cumin oleoresin was gifted from Kancor Flavours, Kerala, India. Standard cuminaldehyde was obtained from Quest International Limited, Mumbai. Standard γ-terpinene and *p*-cymene were obtained from Spectrum Ingredients and Balsara Chemicals, Thane, Mumbai, respectively. All other chemicals were of AR grade.

2.2. Methods

2.2.1. Preparation of microcapsules by spray drying

Thirty percent w/v solution of the different blends of gum arabic, maltodextrin and the commercial modified starch i.e. HiCap® 100 were dispersed in distilled water and final volume made to 100 ml. It was rehydrated for about 12 h at refrigerated temperature (10–12 °C). Three grams (10% based on the carrier used) of oleoresin was added to the mixture. The mixture was emulsified in a shear homogeniser (Indofrench Industries Engineers, Mumbai, Model type- SPM-9) for 5 min at 3000 rpm until complete dispersion of the oleoresin. Two drops of Tween 80 was added for proper emulsification. The resulting slurry was spray dried in JISL LSD-48 mini spray dryer (Company: Jay Instruments, Mumbai) (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 3 bar with the feed rate at 300 g/h. The inlet and outlet temperature was maintained at 160 ± 2 °C and 120 ± 5 °C, respectively. The microcapsules prepared were collected from the collecting chamber and filled in airtight, self-sealable polyethylene pouches. Theses pouches were stored in a dessicator for storage studies.

2.2.2. Analysis of spray-dried microcapsules

2.2.2.1. Analysis of total cuminaldehyde (TC), total γ-terpinene (TGT) and total p-cymene (TPC). To evaluate the ability of wall materials as a flavor carrier, the spray-dried microcapsules were subjected to analysis for total cuminaldehyde (TC), total γ-terpinene (TGT) and total *p*-cymene (TPC). 2.5 g of microcapsules was taken in 50 ml standard conical flask and 8 ml of water was added. The conical flask was shaken vigorously to dissolve in the water. Dichloromethane (12 ml) was added to this while shaking. The mixture was transferred to 125 ml separating funnel and 8 ml of ethanol was added. The separating funnel was shaken properly and allowed to stand for 5 min to separate into layers. Bottom layer (dichloromethane) was taken in 25 ml standard volumetric flask. The exact volume was measured with measuring cylinder. One gram of Na_2SO_4 was added and the extract was filtered. Ten milliliters of the extract was transferred to 10 ml standard volumetric flask in which 0.2 mg of hexadecane was added. Two microliters was injected in the column of the gas chromatography. The TC, TGT, TPC content was then estimated using their respective standard curve.

The amount of constituents in cumin oleoresin in sample was determined by gas chromatography using Chemito GC8610 model, India. Chromatography was performed using 5% SE 30 on chromosorb W-HP, 80–100 mesh (4 m, 1/8" outer diameter, S.S. column) with carrier gas nitrogen (35 ml/min) and an oven temperature programme comprising of an injector temperature of 230 °C, initial temperature of 55 °C for 0 min, temperature increase was from 55 to 90 °C at 6 °C/min, held for 1 min, then 90 to 118 °C at 6 °C/min, 118–120 °C at 0.3 °C/min, 120 to 230 °C at 10 °C/min, flame ionization detector temperature of 240 °C.

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).

Two-hundred milligrams of the sample was weighed accurately. To this 10 ml of diethyl ether and 3 ml distilled water was added, sonicated for 1 min. The mixture was quantitatively washed with water into a separating funnel. This was allowed to stand until the layers separated. The bottom layer was drawn into another separating funnel. To this fraction, a mixture of 8 ml diethyl ether and 2 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 2 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 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) $\%\ \mathrm{NV} = R_e/S \times 100$
In which, S = weight of sample
R_e = weight of ether soluble residue.
(ii) $\%\ \mathrm{EE} = 100 + W - (R_w/S \times 100)$
In which, S = weight of sample
R_w = weight of water-soluble residue
W = water content of sample.
(iii) $\%\mathrm{Volatiles} = \%\ \mathrm{EE} - \%\mathrm{NV}$

2.2.2.3. Stability of the TC, TGT and TPC and TV within the microcapsules. The samples were analyzed over a period of six weeks at 25 °C for TC, TGT and TPC and TV content. 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 cinnamaldehyde was calculated from the rate constant as $0.693/k$.

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

3. Results and discussion

3.1. Analysis and stability of TC, TGA, TPC and TV in free and encapsulated oleoresin

The cumin oleoresin was analyzed for total volatiles (6.78%) and non-volatiles (93.20%). It also showed 3.7% cuminaldehyde, 1.12% γ-terpinene and 0.81% *p*-cymene. This oleoresin was stored at 25 °C in a glass bottle and studied weekly for 6 weeks. The cuminaldehyde content decreased from an initial value of 3.7% to 2.99% over a period of six weeks. The γ-terpinene content decreased from 1.12% to 0.76%. Similarly *p*-cymene, decreased from 0.81% to 0.70%. The retention was in the order of *p*-cymene > cuminaldehyde > γ-terpinene. 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 cuminaldehyde, γ-terpinene, *p*-cymene and volatiles from the oleoresin, storage at low temperature and moisture free environment is recommended.

A semi-log plot of % cuminaldehyde, % γ-terpinene, and % *p*-cymene and % volatiles vs. storage time had shown a sharp decrease in all the constituents, indicating the decrease in these constituents to follow first order kinetics (data not shown). This study revealed cumin oleoresin to lose 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' of the semi-log plot as $t_{1/2} = 0.693/k$. The $t_{1/2}$ for cuminaldehyde, γ-terpinene, *p*-cymene and volatiles in cumin oleoresin stored at 25 °C was 19.74, 11.17, 28.87 and 25.66 weeks (Table 1), respectively indicating *p*-cymene to be more stable under storage condition than the others. There are also possibilities that the top note components viz. monoterpenes which are mainly responsible for the characteristic aroma of spice oils or oleoresins volatilize easily under high temperature conditions.

Ten percent of oleoresin based on carrier material was mixed separately with 30% gum arabic, maltodextrin and modified starch slurry – individually and in blends, to prepare microcapsules. The microcapsules were then analyzed for TC, TGT, TPC and TV over a period of six weeks for gum arabic, maltodextrin and modified starch as wall materials. A linear decrease in the TC, TGT, TPC and TV with storage time was observed for all the wall material used

Table 1
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 arabic (%)	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

($R^2 > 0.9$). 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 as described earlier for the free oleoresin. The $t_{1/2}$ of gum arabic microcapsules for TC, TGT, TPC and TV were 57.75, 30.13, 45.00 and 50.58 which is greater than $t_{1/2}$ evaluated for maltodextrin, modified starch and free oleoresin (Table 2). This is in agreement with the observation made by previous reports (Sharma & Arya, 1995; Raghavan, Abraham, & Shankaranarayana, 1990). This explains the use of gum arabic by flavour industries as a fixative in spray drying applications, wherein the gum encapsulates the flavour compound and protects it from oxidation and volatilization (Qi & Xu, 1999. Sharma & Arya (1995) evaluated the role of hydrocolloid concentration on retention of *p*-cymene and cuminaldehyde in the cumin oleoresin by varying gum acacia concentration from 20% to 45%, and observed the retention of cuminaldehyde and *p*-cymene to be the highest at 40% gum acacia. Cost and limited supply has restricted the use of gum arabic for encapsulation. Hence maltodextrins and modified starches were used as alternative carrier materials. Blends of gum arabic and modified starch and that of gum arabic and maltodextrin were attempted next to encapsulate cumin oleoresin. Theses microcapsules were studied for its TC, TGT, TP and TV content for the period of six weeks. In both the binary blends, half-life of the constituents increased with an increasing proportion of gum arabic in the blend (results not shown).

Our earlier work on microencapsulation of cardamom oleoresin (Krishnan et al., 2005) and cinnamon oleoresin (Vadiya, Bhosale, & Singhal, unpublished) had clearly shown a 4/6:1/6:1/6 blend of gum arabic/maltodextrin/modified starch to be superior to gum arabic. Hence further attempts were made using ternary blends of the wall materials under study. An augmented simplex-centroid mixture design was chosen to evaluate the blends of gum arabic, maltodextrin and modified starch (Buffo & Reineccius, 2000). The design of experiment is shown in Table 1.

Four different blends of gum arabic, maltodextrin and modified starch were carried out, and the microcapsules obtained were analyzed for their TC, TGT, TPC and TV retention. A similar first order trend (semi-log plot of constituent vs. storage time) was observed for the stability of total constituents of cumin oleoresin from ternary blends. The regression analysis and $t_{1/2}$ of TC, TGT, TPC and TV retention in the said microcapsules are given in Table 3. It was observed that an increase in the proportion of gum arabic in the blend contributed significantly to increase in shelf life of the cumin oleoresin. From these results, it is evident that the GA/MD/MS (4/6:1/6:1/6) provided best protection to the cumin oleoresin, in fact even superior than the gum arabic individually, and also its combination with maltodextrin and modified starch. These results are in complete agreement with previous results on microencapsulation of orange oil, cardamom oleoresin and cumin oleoresin (Krishnan et al., 2005; Vaidya, Bhosale, & Singhal, unpublished data).

3.2. SEM of microencapsulated cinnamon oleoresin

The surface characteristics, size and shape of microcapsules of cumin oleoresin prepared using gum arabic, maltodextrin, modified starch and GA/MD/MS (4/6:1/6:1/6) were subjected to SEM analysis (Fig. 1). The microcapsules obtained from gum arabic were smooth but not uniform and had dents on the surface showing the shrinkage where

Table 2
Regression analysis of TC, TGT TPC and TV in the microcapsules[a] prepared from gum arabic, maltodextrin and modified starch as wall materials and free oleoresin

Encapsulating material	Constituent in microencapsulated cumin oleoresin							
	TC		TGT		TPC		TV	
	Regression equation	$t_{1/2}$, weeks	Regression equation	$t_{1/2}$, weeks	Regression equation	$t_{1/2}$, weeks	Regression equation	$t_{1/2}$, weeks
GA	$y = -0.0120x + 4.59$ $R^2 = 0.91$	57.75	$y = -0.0230x + 4.61$ $R^2 = 0.91$	30.13	$y = -0.0154x + 4.59$ $R^2 = 0.96$	45.00	$y = -0.0137x + 4.61$ $R^2 = 0.93$	50.58
MD	$y = -0.0471x + 4.59$ $R^2 = 0.99$	14.71	$y = -0.0536x + 4.59$ $R^2 = 0.99$	12.92	$y = -0.0316x + 4.60$ $R^2 = 0.99$	22.35	$y = -0.0724x + 4.61$ $R^2 = 0.99$	9.57
MS	$y = -0.0311x + 4.60$ $R^2 = 0.99$	22.28	$y = -0.1014x + 4.62$ $R^2 = 0.99$	6.86	$y = -0.0191x + 4.60$ $R^2 = 0.99$	36.28	$y = -0.0241x + 4.60$ $R^2 = 0.99$	28.75
Free oleoresin	$y = -0.0351x + 4.60$ $R^2 = 0.99$	19.74	$y = -0.0623x + 4.55$ $R^2 = 0.95$	11.17	$y = -0.0241x + 4.59$ $R^2 = 0.98$	28.87	$y = -0.0279x + 4.60$ $R^2 = 0.97$	25.66

[a] Oleoresin at 10% based on carrier material used.

Table 3
Regression analysis of TC, TGT, TPC and TV in the microcapsules[a] prepared from the blends of gum arabic, maltodextrin and modified starch

Encapsulating material	Constituent in microencapsulated cumin oleoresin							
	TC		TGT		TPC		TV	
	Regression equation	$t_{1/2}$, weeks	Regression equation	$t_{1/2}$, weeks	Regression equation	$t_{1/2}$, weeks	Regression equation	$t_{1/2}$, weeks
(GA,MD,MS) (1/3,1/3,1/3)	$y = -0.0251x + 4.60$ $R^2 = 0.99$	27.60	$y = -0.0701x + 4.63$ $R^2 = 0.98$	9.88	$y = -0.0305x + 4.61$ $R^2 = 0.97$	22.72	$y = -0.024x + 4.61$ $R^2 = 0.96$	28.87
(GA,MD,MS) (4/6,1/6,1/6)	$y = -0.0124x + 4.60$ $R^2 = 0.99$	55.88	$y = -0.0195x + 4.59$ $R^2 = 0.96$	35.53	$y = -0.0114x + 4.60$ $R^2 = 0.98$	60.78	$y = -0.0081x + 4.60$ $R^2 = 0.99$	85.55
(GA,MD,MS) (1/6,4/6,1/6)	$y = -0.0439x + 4.60$ $R^2 = 0.99$	16.11	$y = -0.0764x + 4.59$ $R^2 = 0.99$	9.07	$y = -0.0438x + 4.60$ $R^2 = 0.99$	15.82	$y = -0.0203x + 4.60$ $R^2 = 0.98$	34.13
(GA,MD,MS) (1/6,1/6,4/6)	$y = -0.0353x + 4.60$ $R^2 = 0.99$	19.63	$y = -0.0623x + 4.61$ $R^2 = 0.99$	11.12	$y = -0.0256x + 4.61$ $R^2 = 0.93$	27.07	$y = -0.0277x + 4.60$ $R^2 = 0.98$	25.01

[a] Oleoresin at 10% based on carrier material used.

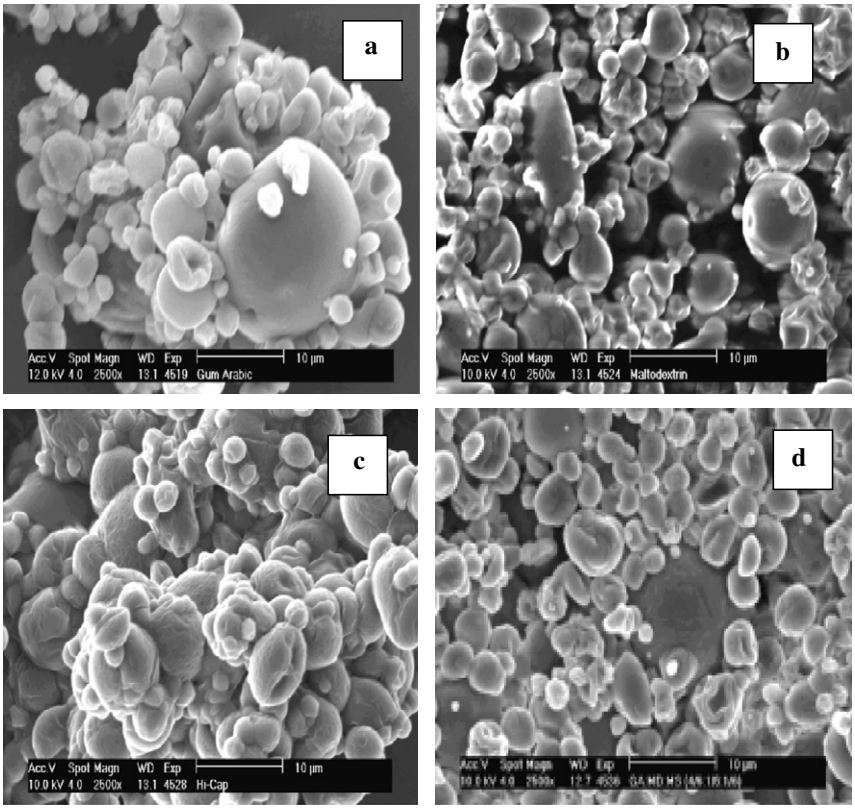

Fig. 1. SEM of the microcapsules prepared from gum arabic (a), maltodextrin (b), modified starch (c) and the blend of GA/MD/MS (4/6:1/6:1/6) (d).

as microcapsules of maltodextrin and modified starch were in broken form indicating the poor encapsulating properties of the carrier materials. This result was in confirmation with earlier reports (Buffo, Probst, Zehentbauer, Luo, & Reineccius, 2002; Varavinit, Chaokasem, & Shobsngob, 2001). The microcapsules obtained from GA/MD/MS (4/6:1/6:1/6) were slightly circular uniform showing minimum cracks and dents on the surface confirming this blend to be the best encapsulating material.

4. Conclusion

The results obtained in the present work indicate gum arabic to be a better wall material for encapsulation of cumin oleoresin as compared to the other wall materials. The stability of the character impact constituents present in oleoresin decreased as the quantum of gum arabic decreased in its blend with maltodextrin and modified starch. GA/MD/MS (4/6:1/6:1/6) blend proved to be more efficient than the other blends, and even better than gum arabic itself. This ternary blend protected different constituents to varying degrees and was in the order of volatiles > *p*-cymene > cuminaldehyde > γ-terpinene.

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