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Hydrophobic derivatives of guar gum hydrolyzate and gum Arabic as matrices for microencapsulation of mint oil

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ABSTRACT

Guar gum hydrolyzate (GGH) modified with *n*-octenyl succinic anhydride (OSA) and oleic acid having induced hydrophobicity was evaluated for encapsulation of mint oil and compared with gum Arabic (GA) and GA–OSA as wall material. Spray dried microcapsules prepared with these wall materials were evaluated for qualitative changes by principal component analysis and for percent retention of mint oil during 8-week storage. Results revealed that microcapsules with GGH–OSA and GGH-oleate showed slightly lower retention of mint oil as compared to GA. GA–OSA microcapsules showed better retention of mint oil than GA itself, as observed from the $t_{1/2}$, the time required for the mint oil to come down to 50% of its original content. The $t_{1/2}$ of mint oil in microcapsules of GA, GGH-oleate, GGH–OSA and GA–OSA was 26.12, 23.50, 24.11 and 29.67 weeks, respectively. The results suggested that GGH–OSA has the potential to replace gum Arabic for encapsulation of mint oil.

1. Introduction

Essential oils are sensitive to light, heat and oxygen, and have a short storage life if not stored properly. Microencapsulation improves the stability of essential oils and enables applications in food and flavor industries by providing resistance against degradative reactions and loss of volatiles (Carneiro, Tonon, Grosso, & Hubinger, 2012; Frascareli, Silva, Tonon, & Hubinger, 2012). Although there are various techniques for microencapsulation, spray drying is the most preferred and commonly used industrial practice. Selection of an appropriate wall material is of utmost importance for effective and efficient microencapsulation by spray drying. Wall materials for microencapsulation of hydrophobic compounds such as essential oils should have both hydrophilic and hydrophobic groups to enable its emulsification and stabilization in the matrix.

Gum Arabic (GA), the exudate from *Acacia senegal* is one of the most common wall materials used in microencapsulation for spray drying, and is considered as a benchmark commodity worldwide (Dickinson, 2003). This is attributed to its high solubility with low viscosity, good emulsifying, and film forming properties. The inconsistent supply, varying quality, as well as increasing prices of GA has propelled researchers to look for alternative and inexpensive natural polymers as wall materials that could replace it totally or partially (Charve & Reineccius, 2009). These efforts have resulted in development of carbohydrates such as maltodextrins and emulsifying starches (Krishnan, Bhosale, & Singhal, 2005; Reineccius, 1988, 1989), protein matrices such as zein (Quispe-Condori, Saldaña, & Temelli, 2011) and barley protien (Wang, Tian, & Chen, 2011), skim milk powder (Aghbashlo, Mobli, Rafiee, & Madadlou, 2012) and vegetable protein (Nesterenko, Alric, Silvestre, & Durrieu, 2013) to retain volatiles during drying processes.

Guar galactomannan is a water soluble, non-ionic, branched chain polymer consisting of straight chain of mannose units joined by β-D (1 → 4) linkages having α-D-galactopyranose units attached to this linear chain by (1 → 6) linkages and ratio of mannose to galactose units being 1.6:1. India accounts for almost 80% of the global production of guar gum (www.guargum.biz). Guar is used extensively in industry due to its excellent thickening properties and low cost. Development of guar gum based wall material for microencapsulation demands alteration of its chemical architecture or chain size. Besides, it lacks emulsifying activity that can be crucial in microencapsulation of some constituents. Prashanth et al. (2006) worked on development of low cost acetate, succinate and octenyl succinate derivatives (OSA) of galactomannans. Dokić, Krstonošić, and Nikolić (2012) reported hydrophobically modified starch with OSA to have strong surface activity resulting in stabilization of oil-in-water emulsions. Partial depolymerization of guar gum has been developed and is used as a natural, water soluble dietary fiber. The partially hydrolyzed guar gum or guar gum hydrolyzate (GGH) has a lower molecular weight and a markedly

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lower viscosity than the native guar gum (Slavin & Greenberg, 2003) with no emulsifying property. We have previously reported on the optimization of protocols of esterification of GGH with *n*-octenyl succinic anhydride (OSA) and oleic acid (OA) to develop GGH-oleate and GGH–OSA and their excellent emulsification properties using mint oil (Sarkar & Singhal, 2011).

Mint oil is derived from *Mentha arvensis*, an aromatic annual herb plant and is classified as an industrial crop. India fulfills 80% of the total global demand with production of 16,000 tons of mint oil (Khanuja, 2007). Mint oil is used in food, perfumery, flavoring and pharmaceutical industry. The most abundant constituent of mint oil is menthol (Lawrence, 1997) which is a monoterpene alcohol present with volatile and labile monoterpenes components like β-pinene, limonene, isomenthone (Duriyaprapan & Britten, 1982). These components degrade during processing and storage which lead to changes in the sensory properties of mint oil. This can be overcome by microencapsulation of mint oil inside a matrix protecting it from environmental and processing adversities (Jackson & Lee, 1991; Zhong et al., 2009). Soottitantawat et al. (2005) studied microencapsulation of L-menthol by spray drying using gum Arabic and modified starch and showed the latter to have better retention of menthol than gum Arabic.

However, the ability of esterified GGH and GA as wall materials for encapsulation of essential oils has not been reported. Hence, in the present work, we report on the efficacy of esterified GGH and GA with *n*-octenyl succinic anhydride (OSA) and oleic acid as wall material to encapsulate mint oil and their storage stability.

2. Materials and methods

2.1. Materials

Guar gum hydrolyzate (GGH) was a commercial sample manufactured and gifted by Lucid Colloids Ltd., Mumbai, India. Gum Arabic (Encapcia) was gifted by Colloides Naturels International (CNI), Normandy, France. Octenyl succinic anhydride (OSA) 99.9% was obtained as a gift from Dixie Chemical, Texas, USA. Oleic acid was procured from S.D. Finechem, Mumbai, India. Mint oil was a gift sample from A. M. Todd, Mumbai, India. 2-octanol (Purity 97.8%) was procured from Sigma–Aldrich, Steinheim, Germany. Diethyl ether was procured from Merck India Ltd., Mumbai, India and distilled twice before use.

2.2. Methods

2.2.1. Preparation of oleate esters of guar gum hydrolyzate (GGH-oleate), GGH–OSA and GA–OSA

GGH-oleate, GGH–OSA and GA–OSA were synthesized under the optimized condition for maximum degree of substitution (DS) as described in our earlier work (Sarkar & Singhal, 2011). For GGH-oleate, 1.4 g oleic acid per 25 g starting material, a reaction time of 120 min at 80 °C gave a maximum DS of 0.061. For GGH–OSA, 1.6 g *n*-OSA per 25 g starting material, a reaction time of 160 min at 85 °C, a maximum DS of 0.072 was obtained. The corresponding values for GA–OSA were an *n*-OSA concentration of 1.5 g per 25 g starting material, a reaction time of 123 min at 77 °C which gave a maximum DS of 0.070.

2.2.2. Preparation of microcapsules by spray drying

The emulsions described earlier by Sarkar and Singhal (2011) briefly were prepared by mixing 20% w/v solution of individual esterified gum with 3 g (15% based on the esterified gum) of mint oil. Oil was dispersed in gum solution by shear homogenizer (Indofrench Industries Engineers, Mumbai, Model type- SPM-9) for 10 min at 3000 *g* until complete dispersion of oil. Emulsion was stored at 4 °C for 24 h for complete diffusion of esterified gum and stabilization of the oil–water interface. Emulsions were spray dried (JISL, LSD-48 mini spray drier, Mumbai, India, inside chamber dimension: 100 cm height and 60 cm diameter) equipped with 0.5 mm diameter nozzle. The pressure of compressed air for the flow of the spray was adjusted to 2 bar. The inlet temperature was maintained at 160 °C and outlet temperature was 95 ± 2 °C. A peristaltic pump was used to feed the spray drier at 400 ml/h. The microcapsules so prepared were collected from the collecting chamber and filled in airtight, self-sealable polyethylene pouches. Theses pouches were stored in a desiccator until further studies.

2.2.3. Analysis of microencapsulated mint oil

2.2.3.1. Determination of total and surface oil. The total oil content in the spray-dried microencapsules was estimated by steam distillation of 100 mg of encapsulated powder for 1 h in Nickerson apparatus with double distilled diethyl ether as extracting solvent (Nickerson & Likens, 1966). 2-Octanol was used as internal standard for quantification of the oil. Each extract was dried over anhydrous sodium sulphate then concentrated by Kuderna Danish apparatus at 40 ± 0.05 °C to less than 1 ml which was finally concentrated to 500 μl by gentle steam of nitrogen.

Surface oil is the oil that is not trapped in microcapsules during spray drying. It was estimated according to method described by Mortenson and Reineccius (2008) with minor modifications. In capped conical flask, the microencapsulated powder (500 mg) was extracted with 20 ml diethyl ether and the mixture was shaken at 150 rpm for 1 h at ambient temperature (27 ± 2 °C) in an orbital shaker. 2-Octanol was used as internal standard for quantification of the oil. The mixture was then filtered through a filter paper (Whatman No. 1) and the filtrate was concentrated by Kuderna Danish apparatus at (40 ± 0.05 °C) to less than 1 ml volume which was finally concentrated to 500 μl by gentle steam of nitrogen. The composition of the total and surface oils was analyzed by gas chromatography (Shimadzu Corporation, Kyoto, Japan) provided with a microthermal conductivity detector and equipped with a RTX5 (Restek corporation, USA) capillary column with 30 m length; 0.25 mm I.D. and film thickness of 0.25 μm. The operating conditions were an initial column temperature 40 °C with hold time of 5 min. Column temperature was then raised to 200 °C at the rate of 4 °C/min with hold time for 2 min, and further to 280 °C at the rate of 10 °C/min and holding for 10 min; injector and interface were kept at 210 and 280 °C, respectively. Helium was used as carrier gas with flow rate of 1.5 ml/min. Temperature of TCD was 290 °C with current value set at 75 mA. All the samples were analyzed in triplicates.

Mint oil obtained from steam distillation were analyzed using GC–MS (Shimadzu Corporation, Kyoto, Japan) having GC-17A gas chromatograph provided with DB-5 (J&W Scientific, California, USA) capillary column (30 m length; 0.25 mm I.D. and 0.25 μm film thickness). The operating conditions were column temperature programmed from 60 to 200 °C at the rate of 4 °C/min, held at initial temperature and at 200 °C for 5 min and further to 280 °C at the rate of 10 °C/min, held at final temperature for 20 min. Injector and interface temperatures were 210 and 280 °C, respectively. Helium (flow rate, 1.5 ml/min) was used as carrier gas. MS parameters were 70 eV ionization voltage and electron multiplier voltage of 1 kV. Peaks were tentatively identified by comparing its mass fragmentation pattern with standard spectra available in the spectral library (Wiley/NIST Libraries) of the instrument and with Kovats index.

2.2.3.2. Determination of encapsulation and entrapment efficiency. Encapsulation efficiency indicates the efficiency of the process to encapsulate and was determined as per Bule, Singhal, & Kennedy (2010). It was calculated as the ratio of the mass of mint oil present in the microcapsules to the mass of mint oil added at the time

Table 1
Encapsulation and entrapment efficiency of mint oil in different wall materials[*,**] and its half life in the microcapsules therein.

Encapsulation material	Encapsulation efficiency (%)	Entrapment efficiency (%)	Regression equation for storage stability	Half life $T_{1/2}$, 27 ± 2 °C (weeks)
Gum Arabic	80.66 ± 2.12^{a}	86.26 ± 1.33^{a}	ln (% retention of mint oil) = −0.027 time + 4.610 (R^2 = 0.991)	26.12
Gum Arabic–OSA	84.19 ± 1.17^{b}	89.20 ± 1.45^{b}	ln (% retention of mint oil) = −0.023 time + 4.660 (R^2 = 0.995)	29.67
GGH–OSA	77.56 ± 1.09^{c}	81.13 ± 1.75^{c}	ln (% retention of mint oil) = −0.028 time + 4.584(R^2 = 0.980)	24.11
GGH oleate	72.98 ± 1.99^{d}	77.29 ± 1.88^{d}	ln (% retention of mint oil) = −0.029 time + 4.450(R^2 = 0.983)	23.50
Free mint oil	–	–	ln (% retention of mint oil) = −0.056 time + 4.731(R^2 = 0.995)	12.36

[*] Values are mean ± SD of three or more determinations.
[**] Means in same columns with same superscripts do not differ significantly ($p < 0.05$).

of emulsion preparation prior to spray drying. The encapsulation efficiency was calculated using Eq. (1).

$$\text{Encapsulation efficiency}(\%) = \frac{\text{Total mint oil by experimental determination}(\text{g/g powder})}{\text{Theoritical loading of mint oil}(\text{g/g powder})} \times 100 \quad (1)$$

The entrapment efficiency is defined as percent of oil entrapped in capsules and was calculated by Eq. (2)

$$\text{Entrapment efficiency}(\%) = \frac{\text{Total mint oil}(\text{g/g powder}) - \text{surface min t oil}(\text{g/g powder})}{\text{Total mint oil}(\text{g/g powder})} \times 100 \quad (2)$$

2.2.4. Storage stability of mint oil microcapsules

Mint oil microcapsules obtained with GGH-oleate, GGH–OSA, GA and GA–OSA as wall material were analyzed for the content of total mint oil during 8-week storage at 27 ± 2 °C. The percentage retention of mint oil was calculated by the formula

$$\%\text{retention of mint oil} = \frac{\text{Mint oil at 'X' day of storage time}(A)}{\text{Mint oil at '0' day of storage}(A_0)} \times 100 \quad (3)$$

Stability of encapsulated mint oil was fitted in first order kinetic model during storage.

$$\frac{A}{A_0} = \exp(-kt) \quad (4)$$

where A_0 and A are the content of mint oil immediately after encapsulation and time t, respectively. A semi-log graph of percent retention of mint oil vs. time was plotted to obtain the rate constant (k) as the slope of the graph from which the half-life ($t_{1/2}$) i.e. the time required for 50% reduction in mint oil content was calculated as 0.693/k (Durge, Sarkar, Survase, & Singhal, 2011).

2.2.5. Morphological characteristics by scanning electron microscopy (SEM)

The external structure of the encapsulated powder was studied by SEM (JSM 5800, JEOL, Tokyo, Japan). The powders were placed on the SEM stubs using a two-sided adhesive tape (Nisshin EM, Tokyo, Japan) and then analyzed after Pt–Pd sputtering by MSP-1S magnetron sputter coater (Vacuum Device, Tokyo, Japan).

2.2.6. Statistical analysis

IBM® SPSS® statistic package was used for analysis of data. Analysis of variance (ANOVA) by Fisher's least significant difference was performed to examine effect of esterification on microencapsulation properties of gums. Principal component analysis was performed on relative areas of volatile compounds of mint oil as analyzed by gas chromatography using XLSTAT 2011 software.

3. Results and discussion

3.1. Analysis of mint oil microencapsulated in GA, GGH-oleate, GGH–OSA and GA–OSA

Effectiveness of microencapsulation is indicated by encapsulation efficiency and entrapment efficiency. The higher retention of mint oil in the spray-dried products is desirable and advantageous for industrial applications. The effect of wall material on retention of mint oil during spray drying is shown in Table 1. It can be seen from the values of encapsulation efficiency and entrapment efficiency of mint oil in GGH–OSA, GGH-oleate and GA–OSA that GA–OSA was better than GA itself as a wall material for encapsulation of mint oil.

The efficient entrapment of constituents in GA is due to good film forming capability and their plastic nature, rather than glassy property. Plasticity is known to prevent cracking of protection matrix (Bertolini, Siani, & Grosso, 2001). GA–OSA had good film forming property with improved emulsifying property so it had better retention of mint oil than GA due to presence of the hydrophobic octenyl group in GA as reported in our earlier work (Sarkar & Singhal, 2011). In GGH-oleate and GGH–OSA, the emulsifying property was introduced due to the hydrophobic molecule of oleate and succinic anhydride. GGH–OSA had better retention of mint oil than GGH-oleate microcapsules. This is in accordance with our earlier work where we reported OSA modified GGH to have better emulsification properties than GGH-oleate (Sarkar & Singhal, 2011).

Surface oil is an important factor influencing the storage stability of the microcapsules as the surface oil can easily oxidize resulting in unacceptable off-flavors, and entrapment efficiency is indicator of surface oil. Gum Arabic and its OSA derivative had lower surface oil due to uniform film formation of the microcapsules than GGH–OSA and GGH-oleate. Microcapsules were subjected to storage stability study for a period of eight weeks at ambient temperature (27 ± 2 °C) and analyzed for retention of total mint oil within the capsules. A linear nature of semi-log plot of percent retention of mint oil vs. storage time showed that the loss of mint oil from the microcapsules followed first order kinetics (Fig. 1).

The regression equation and $t_{1/2}$ values for GA, GGH-oleate, GGH–OSA and GA–OSA microcapsules are also tabulated in Table 1. Shaikh, Bhosale, and Singhal 2006 reported first order kinetics for encapsulated black pepper oleoresin where they showed GA to have better encapsulation ability than modified starch. Bule

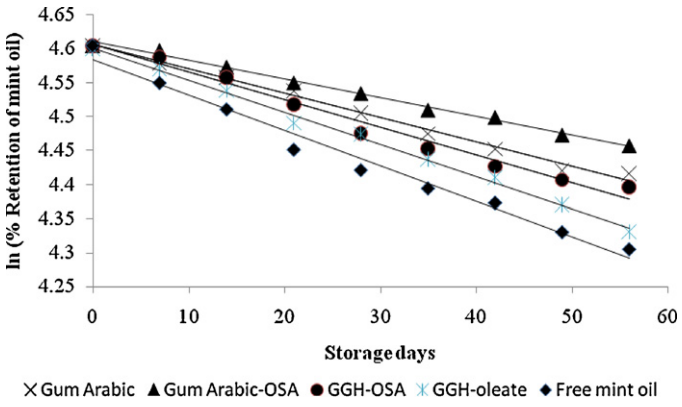

Fig. 1. Stability of encapsulated mint oil measured as ln retention of mint oil vs. storage time in days (a) gum Arabic; (b) gum Arabic–OSA; (c) GGH–OSA; (d) GGH-oleate; (e) free mint oil; stored at ambient temperature (27 ± 2 °C).

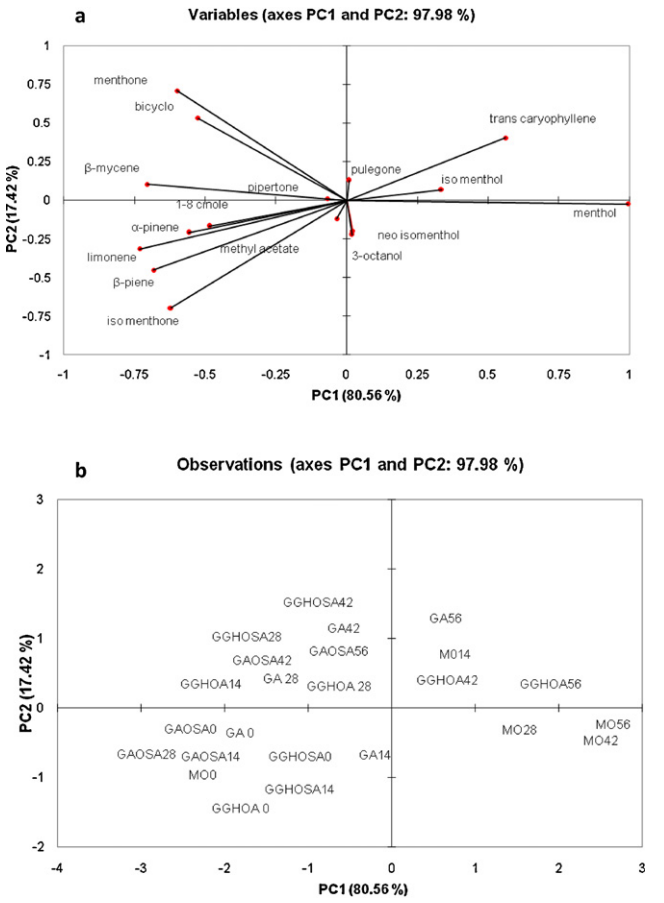

Fig. 2. Principal component analysis of volatiles of mint oil encapsulated with different wall materials after various storage periods (plots of first two principal components) (a) loading plot depicting distribution of various mint oil compounds with F1 and F2; (b) score plot depicting distribution of various samples with F1 and F2.

et al. (2010) reported CoQ10 to be best retained in a wall material combination of gum Arabic, maltodextrin and modified starch (67:17:17) which also followed first order kinetics during storage. We recently reported the retention of mint oil in microcpasules developed from gamma depolymerized guar gum and GA as wall material to follow first order kinetics during storage (Sarkar, Gupta, Variyar, Sharma, & Singhal, 2012). Microcapsules prepared using GA and GA–OSA as sole wall material also had a higher percent retention of 60.36 ± 1.21 and 64.33 ± 1.56%, respectively, after 8-weeks as opposed to GGH-oleate (54.89 ± 1.79) and GGH–OSA (56.12 ± 1.98).

GA and GA–OSA microcapsules demonstrated significant ($p < 0.05$) higher $t_{1/2}$ values and higher retention of mint oil after 8 weeks of storage as compared to GGH-oleate and GGH–OSA. OSA modification improved the emulsifying property of GA and resulted in better film forming ability and consequently better retention of mint oil within the microcapsules. However, GGH which initially did not show emulsifying property could also demonstrate emulsifying activity after esterification with OSA and oleic acid which in turn improved the encapsulating ability of GGH (Sarkar & Singhal, 2011).

Qualitative changes in mint oil entrapped in GA, GGH-oleate, GGH–OSA and GA–OSA microcapsules during storage was assessed by principal component analysis (PCA). First two principal components explained 80.56% and 17.42% of data variance (PC1 and PC2, respectively). Loading and score plots are depicted in Fig. 2. PC1 was highly influenced by menthol, α-pinene, β-pinene, limonene, methyl acetate, 3-octanol while compounds contributing more toward PC2 were neo isomenthone, pulegone, isomenthol, menthone, and 1,8-cinole (Fig. 2a). The score distribution from first two PCs (Fig. 2b) demonstrated three separate groups in samples analyzed. First group had samples (GA0, GA14, GAOSA0, GAOSA14, GAOSA28, GGHOleic acid0, GGHOSA0 and MO0) in initial storage period and was located on negative side of both PC1 and PC2. GA28, GA42, GAOSA42, GAOSA56, GGHOSA28, GGHOSA42, GGHOleic acid14 and GGHOleic acid28 constituted second group located on negative side of PC1 but positive side on PC2 and third group (GA56, GGHOleic acid42, GGHOleic acid 56, GGHOSA56, MO14, MO28, MO42 and MO56) was located on negative side of both PC1 and PC2. From loading plot (Fig. 2a), it can be concluded that monoterpene hydrocarbons like α-pinene, β-pinene, limonene, 1,8-cineole, and isomenthone are correlated with the first group while oxygenated monoterpenes like menthol and neo isomenthol are positively correlated to third group. It can be concluded that there was a decrease in content of monoterpene hydrocarbons with corresponding increase in oxygenated monoterpenes like menthol on storage of the microcapsules. Qualitative change in microencapsulated mint oil in GGH-oleate and GGH–OSA as wall material showed comparable retention of mint oil components with GA. However GA–OSA microcapsules demonstrated better qualitative retention of mint oil components as compared to GA alone as wall material.

3.2. Morphological characterization by scanning electron microscopy (SEM)

SEM was used to investigate the morphological structure of microcapsules prepared from GA, GGH-oleate, GGH–OSA and GA–OSA microcapsules (Fig. 3). The external morphology of the encapsulated particles showed spherical shape with both grooves and smooth surfaces. The particle size ranged from 2.29 to 15.45 μm for GA, 2.11–16.44 μm for GGH-oleate, 2.56-15.85 μm for GGH–OSA and 2.22–15.82 μm for GA–OSA microcapsules. No visible cracks or fissures were observed in the outer surfaces of the particles, which is a good indicator of the microencapsulation efficiency of microcapsules preparation process by spray drying (Trindade & Grosso, 2000). It implied the capsules to have lower permeability to gases which translates in to increasing protection and retention of the active material.

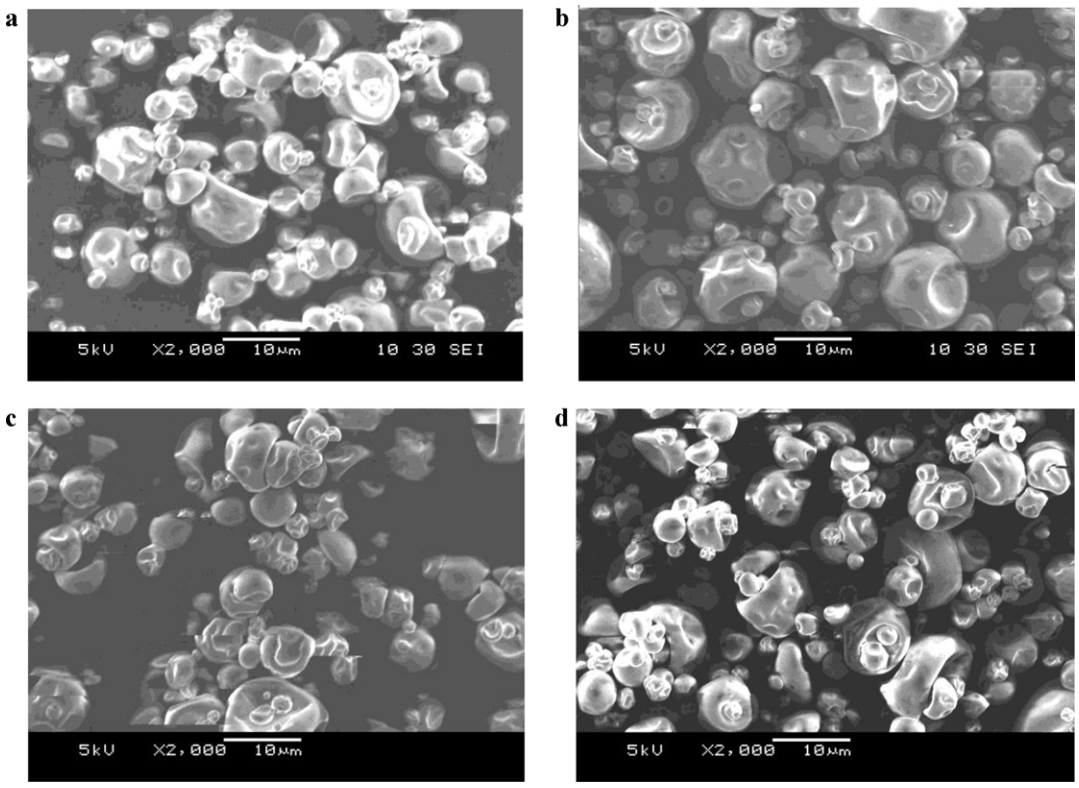

Fig. 3. Scanning electron microscopy of the microcapsules of mint oil obtained after spray drying with (a) gum Arabic; (b) gum Arabic–OSA; (c) GGH–OSA; (d) GGH-oleate magnification 2000×.

4. Conclusion

Mint based oil microcapsules were successfully produced by spray drying using GA, GGH-oleate, GGH–OSA and GA–OSA. GA–OSA proved to be a superior wall material than GA for encapsulation of mint oil, a fact which is being reported for the first time. Retention of mint oil in GGH-oleate and GGH–OSA were slightly lower than GA, although qualitative changes in mint oil volatiles in microcapsules during storage was similar to GA. GGH modified with OSA and oleic acid introduced emulsifying activity and good flavor encapsulation ability and qualified them as important alternative encapsulating wall materials. The results of this study reveal the potential of GGH-oleate and GGH–OSA as a substitute for gum Arabic in encapsulated essential oil and further development of a gum Arabic derivative that is superior to gum Arabic itself for the intended purpose.

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