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Effect of microencapsulation of cardamom's essential oil in gum Arabic and whey protein isolate using spray drying on its stability during storage

K.M. Al-Ismail[1*], G. Mehyar[1], H.S. Al-Khatib[2] and M. Al-Dabbas[1]

[1]The University of Jordan, Faculty of Agriculture, Department of Nutrition and Food Technology, Queen Rania Avenue, Amman, Jordan; [2]The University of Jordan, Faculty of Pharmacy, Department of Pharmaceutics and Pharmaceutical Technology, Amman, Jordan; kh.ismail@ju.edu.jo

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RESEARCH ARTICLE

Abstract

Cardamom essential oil (CEO) was microencapsulated in different materials; gum Arabic (GA), whey protein isolate (WPI) or their combinations (WPI+GA) at different mixing proportions; 1:1 and 3:1, respectively by using spray drying procedure. The stability of the encapsulated oil was evaluated by determining the concentration of encapsulated oil during storage at two temperatures (7 and 25 °C). Results showed that regardless of the storage temperature, GA microcapsules had the highest microencapsulation efficiency and retention of CEO throughout the storage period. However WPI microcapsules had the lowest microencapsulation efficiency and retention of CEO. Both WPI+GA combinations showed intermediate microencapsulation efficiency and CEO retention compared to the single component matrices. Micrographs revealed that both WPI and GA microcapsules were spherical in shape. However, WPI microcapsules had broken surfaces, whereas the GA microcapsules had dented and wrinkled surfaces. In conclusion, GA microcapsules showed the best overall encapsulation properties than other treatments.

Keywords: regression analysis, microcapsules, retention efficiency

1. Introduction

Flavours are complex mixtures of comparatively volatile and labile substances that their sensory perception can be deteriorated by oxidation, chemical interactions and volatilisation (Burt, 2004). Ground green cardamom loses its flavour quickly primarily by oxygen action on terpinic and lipid components (Burt, 2004). Cardamom essential oil (CEO), as one of the most commonly used flavours, is reported to develop some off-flavour in a few days of storage as a result of physical and chemical reactions (Krishnan *et. al.*, 2005; Ubbink and Kruger, 2006).

Microencapsulation technology has been used to protect sensitive products, such as CEO as well as other bioactive compounds, against the stress conditions of processing and storage (De Vos *et al.*, 2010; Given and Peter, 2009; Najafi *et al.*, 2011). Microencapsulation can be defined as the process of covering active compounds with protective layers against environmental factors and allowing controlled migration of these compounds in timely matter to the surrounding foods (Kuang *et al.*, 2010; Madene *et al.*, 2006). Several factors found to affect the protection action of microcapsules to flavours including microcapsules particle size, type of interaction between the wall material(s) and the encapsulated flavour, emulsion droplet size distribution and drying technique (Kaushik and Roos, 2007).

The spray drying technique has become one of the most important processes for microencapsulation. It is considered economical, flexible and capable of producing desired quality particles (Shahidi and Han, 1993). Spray drying was the recommended procedure for microencapsulating carotenoids, paprika oleoresins, and cardamom oleoresins (Krishnan *et al.*, 2005; Leach *et al.*, 1998). Retention of cardamom oil indicated by stabilising of its main components; 1,8 cineole and limonene, by spray-dried microcapsules made of skim milk or modified starch was better in comparison to freeze-dried microcapsules (Najafi *et al.*, 2011).

Gum Arabic (GA) is widely used as wall materials for microencapsulation due to its excellent solubility in water and its surface active properties and ability to form dried matrix around dispersed compounds upon dehydration, but it is an expensive ingredient and subjected to flocculation in cost and availability (Landy *et al.*, 1995; Qi and Xu, 1999). GA microcapsules were effective in preventing oxidation of linoleic acid by providing slow delivery and were effective in retaining intact encapsulated limonene (Fang *et al.*, 2005; Kaushik and Roos, 2007). Whey protein isolate (WPI) can stabilise emulsion droplets during homogenisation due to its ability to interact at the water-oil interface but it could be associated with allergy problems (Kuang *et al.*, 2010). The single matrix encapsulation could not possess all required properties; therefore efforts to combine wall materials to improve encapsulation have been done. Kishnana *et al.* (2005) found that combining GA with maltodextin and modified starch provided best protection for cardamom oleoresin than GA alone. Microencapsulation of cinnamon oleoresin by spray drying using GA: maltodextrin: modified starch blends offered protection better than GA (Kanakdande, *et al.*, 2007). Combination of GA and WPI as wall material for microencapsulating CEO has never been investigated. Therefore, the aim of the current study was to evaluate stability of cardamom's essential oil when microencapsulated in GA, whey protein isolate and their combinations by spray drying.

2. Material and methods

Materials

CEO (Sigma-Aldrich, Saint Louis, MO, USA), GA (Laboratory Rasayan, Anand, India), WPI (Bi Pro; Davisco Foods International Inc., Le Sueur, MN, USA) were ordered from their producing company. Tween® 80 (Sigma-Aldrich) was purchased from the local suppliers. Tween 80 is nonionic surfactant and emulsifier derived from polyethoxylated sorbitan and oleic acid. All chemical reagents used were of analytical grade except the n-heptane which was GC grade.

Preparation of microcapsules by spray drying

The preparation of microcapsules was carried out following the method described by Najafi *et al.* (2011) with slight modification. Appropriate amounts (Table 1) of biopolymers, GA and WPI and their combinations (WPI+GA mixed at different proportions; 1:1 and 3:1, respectively) were dispersed in cold distilled water and mixed using a magnetic stirrer (Fisher Scientific, Pittsburgh, PA, USA) that was set at 1,200 rpm for 30 min. The dispersions were then kept in a refrigerator for about 18 h (8-10 °C) to allow full hydration of the polymers. About 18 g of CEO and 1.7 g of Tween 80 (1.7% of the wall material) were added to the mixtures and the total volume was made up to 500 ml. The mixtures were then emulsified using an Ultra Turrax homogeniser (IKAT 18 basic; Ika, Staufen, Germany) set at 11,000 rpm for 15 min. The resulted emulsions were spray dried using spray dryer (B290; BÜCHI Labortechnik, Flawil, Switzerland) by implementing of the following process variables: feed rate at 5 ml/min, aspirator at 100%, atomisation pressure at 0.3 bar, inlet air temperature at 150 °C, outlet air temperature at 50 °C, and the atomiser beak diameter was set at 1.4 mm. The resulted powders were collected and stored in screw capped bottles at -18 °C for further analysis.

Table 1. Formulation of emulsions used to prepare the microcapsules. Values are in grams per 500 ml dispersion.

Formulation	Biopolymer (g)		CEO (g)	Tween 80 (g)
	GA	WPI		
GA	100	–	18	1.7
WPI	–	100	18	1.7
WPI+GA (1:1)	50	50	18	1.7
WPI+GA (3:1)	75	25	18	1.7

CEO = cardamom essential oil; GA = gum Arabic; WPI = whey protein isolate.

Moisture determination

The moisture content of the microcapsules was determined using a moisture balance (PMP 202; Adam Equipment Inc., Milton Keynes, UK) and conventional oven set at 105±2 °C. 2 g of the microcapsules were dispersed on the surface of metal plate and dried until constant weight (Najafi *et al.*, 2011). The analysis was carried out in duplicate.

Determination of surface oil

The oil on the surface of microcapsules was determined by mixing one gram of the microcapsules with 20 ml hexane in a screw cap test tube. The mixture was agitated manually for 5 min and filtered through 0.45 µm filter (Soottitantawat *et al.*, 2005). 20 µl of the filtrates was injected into Knauer high-performance liquid chromatography (HPLC) system (Advanced Scientific Instruments, Berlin, Germany) consisting a pump (Smart Line 1000) and UV detector (Smart Line 2500). The mobile phase was composed of acetonitrile:water (70:30) passed through a C18 column (25 cm × 4 mm, 5 µm; Eurospher; Knauer, Berlin, Germany) at a rate of 1 ml/min. The detection wavelength was 270 nm. This wave length representing the maximum absorption of 1,8 cineole, one of the major components of the CEO that was used as indicator to monitor the CEO content in the microcapsules. The determination was according to the

method described by Marongiu *et al.* (2004). The calibration curve was prepared from CEO as external standard. The analysis was carried out in duplicate.

Determination of the encapsulated essential oil

1 gram of the hexane-washed microcapsules, obtained from surface oil determination procedure, was dispersed in 10 ml water in screw capped test tube, agitated for 3 min followed by addition of 5 ml hexane then agitated again manually for 3 min. The hexane layer was filtered through 0.45 µm filter. 20 µl of hexane layer was injected into HPLC system and analysed at the same conditions previously described for determination of the surface CEO. The analysis was carried out in duplicate.

Microencapsulation efficiency

The efficiency of microencapsulation was calculated as the ratio of the encapsulated oil in the obtained microcapsules to its theoretical loading in the emulsions (Jun-Xia *et al.*, 2011) as follows:

Microencapsulation efficiency = amount of CEO in dried microcapsules (g)/amount of CEO in the emulsion (g) × 100

Stability of the essential oil in the microcapsules

The stability of the essential oil in the microcapsules was evaluated during storage at 7 °C (representing refrigeration temperature) and 25 °C (representing room temperature) over 12 weeks. Samples were withdrawn at 0, 1, 2, 4, 8, and 12 weeks during storage and analysed for 1,8 cineole concentration, the main component of the CEO following a HPLC procedure as described by Marongiu *et al.* (2004). The half-time ($T_{1/2}$) was determined as the time required for reducing the amount encapsulated CEO to 50% of their original content in the produced microcapsules (Najafi *et al.*, 2011). Simple regression analysis for the obtained curves (time vs. 1.8 cineole concentration) for each treatment was performed. The analysis was carried out in duplicate.

Scanning electron microscopy

Scanning electron micrographs of the dried microcapsules was obtained using a scanning electron microscope (SEM) (Inspect F50; FEI, Eindhoven, the Netherlands) equipped with XT microscope server software (version 4.1.1.11935; FEI). A small amount of the microcapsules was scattered evenly over an aluminium stub covered with carbon tab. Excess material was removed using dry compressed air and the sample was sputter coated with platinum using a sputter coater (K550X, Quorum Emitech, East Grinslead, UK). The stub containing the coated sample was then placed in the specimen chamber under vacuum and at an accelerating voltage of 3.0 kV (Krishnan *et al.*, 2005).

Particle size distribution

The particle size analysis of the microcapsules was carried out using Mastersizer 2000 (Malvern Instruments, Malvern, UK), following the procedure described in the manual of the equipment. Microcapsules were suspended in ethanol and subjected to an ultrasound waves for 1.5 min. The analysis was performed at room temperature and at a constant agitation rate.

Statistical analysis

All experiments were carried out based on complete randomised design and the results represent the mean of at least two replicates. The data obtained was analysed by the analysis of variance (ANOVA) using SAS program (version 9.1; SAS Institute Inc., Cary, NC, USA). Significant differences between means were determined by Duncan's multiple range test at a probability levels of $P \leq 0.05$.

3. Results and discussion

Microencapsulation efficiency and surface oil content

The microencapsulation efficiency and percentage of surface oil of the prepared microcapsules from WPI, GA and CEO as affected by the type of wall materials are represented in Table 2. The diffusion of entrapped oil to the surface of capsules is not desirable as it can be easily oxidised there, producing unacceptable flavours. The data indicate that GA produced microcapsules with the highest microencapsulation efficiency and the lowest surface oil could be due to higher resistance of GA to the severe temperature during spray drying (Jafari *et al.*, 2008). WPI as wall material showed the lowest microencapsulation efficiency of CEO might be due to the denaturation and structural changes in the WPI during the heating period of spray drying (Najefi *et al.*, 2011). This could reduce the WPI ability to bind and encapsulate CEO thus lower entrapped oil and microencapsulation efficiency were detected (Table 2). Mixing GA with WPI at different ratios (1:3 and 1:1) produced intermediate ability to encapsulate CEO but higher microencapsulation efficiency and lower surface oil content were obtained compared to the WPI microcapsules (Table 2). WPI as emulsifier could form a robust structure by binding both the hydrophilic (GA) and hydrophobic (CEO) phases during the heating period whereas GA increased the viscosity of the emulsification solution, thus the combinations could produce better environment for entrapment of the CEO than WPI alone. Using the WPI at higher level in the wall material (3:1) adversely affected the microencapsulation efficiency and higher surface oil was detected for the produced microcapsules (Table 2). This could be due to the higher proportion of the ruptured WPI by heat denaturation during the spray drying process, produced excessive release of the CEO. It has been reported

Table 2. Effect of wall material type on the microencapsulation efficiency of cardamom essential oil, surface oil and moisture content of the microcapsules.

Wall material	Encapsulation efficiency (%)	Surface oil (%)	Moisture content (%)
GA	92.6[a]	0.49[a]	2.66[a]
WPI	69.2[d]	1.72[ab]	2.95[a]
WPI+GA (1:1)	83.3[b]	0.82[c]	2.67[ab]
WPI+GA (3:1)	74.3[c]	1.11[d]	2.94[a]

GA = gum Arabic; WPI = whey protein isolate.
Values with the same superscript letters within a column are not significantly different (P>0.05).

that freeze drying procedure provided better retention of CEO encapsulated in skim milk protein than spray drying and that was related to protein denaturation and structural changes of protein at the extreme conditions of spray drying (Najefi *et al.*, 2011). The surface oil contents of the microcapsules of this study were significantly lower than those reported by Krishnan *et al.* (2005). This could be related to differences in spray drying conditions and wall materials used in both studies.

Moisture content

The moisture contents of the resulted microcapsules are represented in Table 2. The data show that the moisture content of the four types of microcapsules was relatively low and not significantly (P>0.05) affected by the type of wall material. This may be produced from using suspensions that have the same total polymer concentration. Our preliminary data showed that increasing polymer concentration of the suspensions used with spray dryer produced microcapsules with higher moisture content than lower total solids suspensions. Other factors such as spray drying conditions and the type of wall material could also have an effect. Najefi *et al.* (2011) reported that spray-dried microcapsules made of skim milk and modified starch and their mixtures encapsulating CEO had lower moisture content (1.25-1.9%) than those reported in the current study (Table 2). They also reported that freeze-drying procedure produced higher moisture microcapsules than spray-drying for the same composition microcapsules.

Stability of microencapsulated cardamom essential oil

The ANOVA test shows that the main factors, type of coating material, storage temperature and time, and their interactions have a significant effect on the stability of the microencapsulated CEO (Table 3). The kinetics of the CEO loss from microcapsules during storage at 7 and 25 °C were evaluated by plotting the CEO concentration against storage time as shown in Figure 1, respectively. The figure showed a sharp linear decrease in encapsulated CEO content during the storage period. This decrease followed first order kinetics with coefficient of determination greater than 0.833 (Figure 1).

The half-life ($t_{1/2}$) was calculated from the slope of the curves (k) according to the formula $t_{1/2} = 0.693/k$ and the results are reported in Table 4 (Najafi *et al.*, 2011). The $t_{1/2}$ values indicate that the type of coating material significantly (P≤0.05) influenced the CEO retention in microcapsules. The GA microcapsules provided better protection for CEO than WPI microcapsules with $t_{1/2}$ of 173.3 weeks and 15.8 weeks, respectively at 7 °C and 173.3 weeks and 9.9 weeks, respectively at 25 °C. Furthermore, WPI+GA microcapsules at (1:1) and (3:1) afforded better protection for CEO than those consisted of WPI alone (Table 4). This may indicate

Table 3. Analysis of variance for the main experimental factors and their interactions.

Source[1]	Degrees of freedom	Sum of squares	F ratio	Prob. > F
Trt	3	9,841.0734	497.495	P<0.0001
Time	5	7,823.128	237.2892	P<0.0001
Trt×Time	15	3,375.84	34.1317	P<0.0001
Temp	1	964.7028	146.3056	P<0.0001
Trt×Temp	3	365.2284	18.4634	P<0.0001

[1] Trt = treatment; Temp = storage temperature; Time = time of storage.

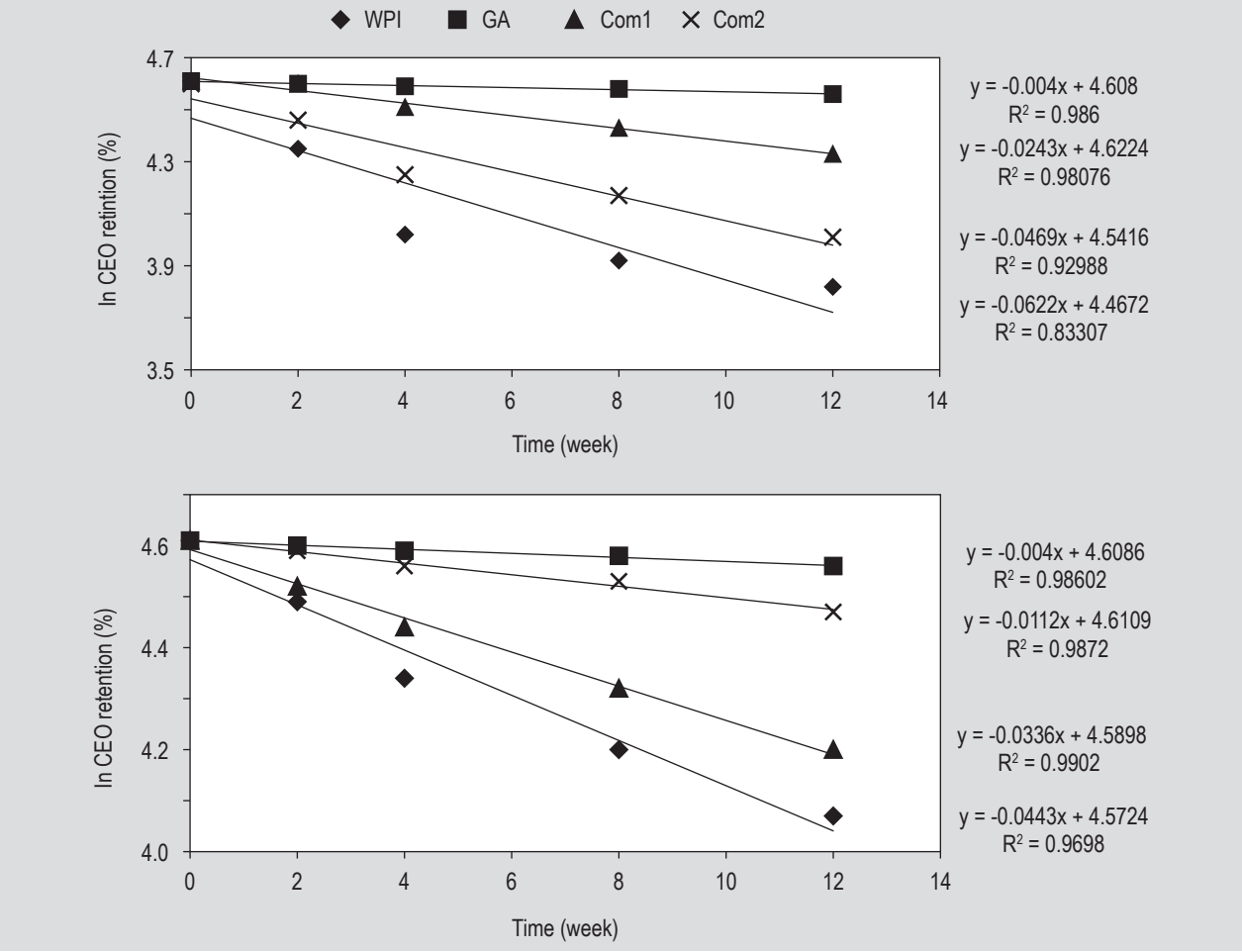

Figure 1. The stability of microencapsulated cardamom essential oil (CEO) during storage at (A) 25 °C and (B) 7 °C as affected by the type of wall material. WPI = whey protein isolate; GA = gum Arabic; Com1= WPI+GA (1:1); Com2 = WPI+GA (3:1).

Table 4. $T_{1/2}$ values of the microencapsulated cardamom essential oil stored at 7 and 25 °C for 12 weeks.

Wall material	$T_{1/2}$ at 7 °C (week)	$T_{1/2}$ at 25 °C (week)
GA	173.3[a]	173.3[a]
WPI	15.8[d]	9.9[d]
WPI+GA (1:1)	63[c]	13.9[c]
WPI+GA (3:1)	19.9[b]	28.9[b]

Values with the same superscript letters within a column are not significantly different (P>0.05).

that addition of GA to WPI improved the protective effect of WPI to CEO.

Krishnan *et al.* (2005) reported that the increasing the proportion of GA to maltodextrin or modified starch in wall material of freeze-dried microcapsules improved their preservation of encapsulated cardamom oileoresin. Emulsion droplet size for the suspensions that is used to prepare the microcapsules could also be a significant factor in CEO retention. Soottitantawat *et al.* (2005) found that GA produced smaller emulsion and its microcapsule had better d-limonene retention than the maltodextrin and modified starch microcapsules prepared by spray drying during storage at 51% RH and 50 °C. In another study, it was found that GA was effective in keeping limonene in freeze-dried microcapsules while mixing it with gelatin and sucrose at various concentrations reduced its effectiveness (Kaushik and Roos, 2007).

The results in Figure 1 indicate that decreasing the storage temperature from 25 to 7 °C decreased the rate of CEO loss (as indicated by the increased $t_{1/2}$) for the microcapsules made of WPI or WPI+GA. In the other hand, the $t_{1/2}$ for microcapsules made of WPI, WPI+GA (3:1) and WPI+GA (1:1) and stored at 7 °C were about 1.4 and 2 times longer than the corresponding microcapsules stored at 25 °C. Reduction of storage temperature did not influence the rate of CEO loss from GA microcapsules. This could suggest

superior protective effect of GA against temperature flocculation that could be related to structural compactness.

SEM of the microencapsulated cardamom essential oil

The SEM of the CEO microcapsules prepared from WPI, GA and their combinations are shown in Figure 2. The microcapsules of WPI were spherical with smooth surface but formed hollow and cracked particles. The cracks might be due to the high inlet temperature of the spray drier (Finotelli *et al.*, 2005). The broken surface and the presence of the holes may facilitate diffusion of entrapped oil to the surface as higher surface oil was detected in these microcapsules (Table 2). Excessive surface oil is undesirable as it can easily oxidise resulted in unacceptable off-flavour and decreased shelf life. The GA microcapsules were almost spherical but with dents and wrinkles. The higher resistance of GA to the severe drying conditions may affect the formation of spherical microcapsules and without cracks that could retain higher amount of entrapped CEO (Xie *et al.* 2010). The presence of surface dents on GA microcapsules might be attributed to the atomisation and drying conditions on GA. Similar results was reported by other researches for microcapsules made of GA (Krishnan *et al.*, 2005; Najafi *et al.*, 2011; Soottitantawat *et al.*, 2005). It has been reported that GA is among the high water absorbing polymers, upon hydration and swelling. Most of this moisture is lost during drying producing collapsed particles with wrinkles on the surface (Al-Assaf, 2008). Combining the GA with WPI (1:1 and 3:2) produced hybrid particles with mixed properties (Figures 2C and D, respectively).

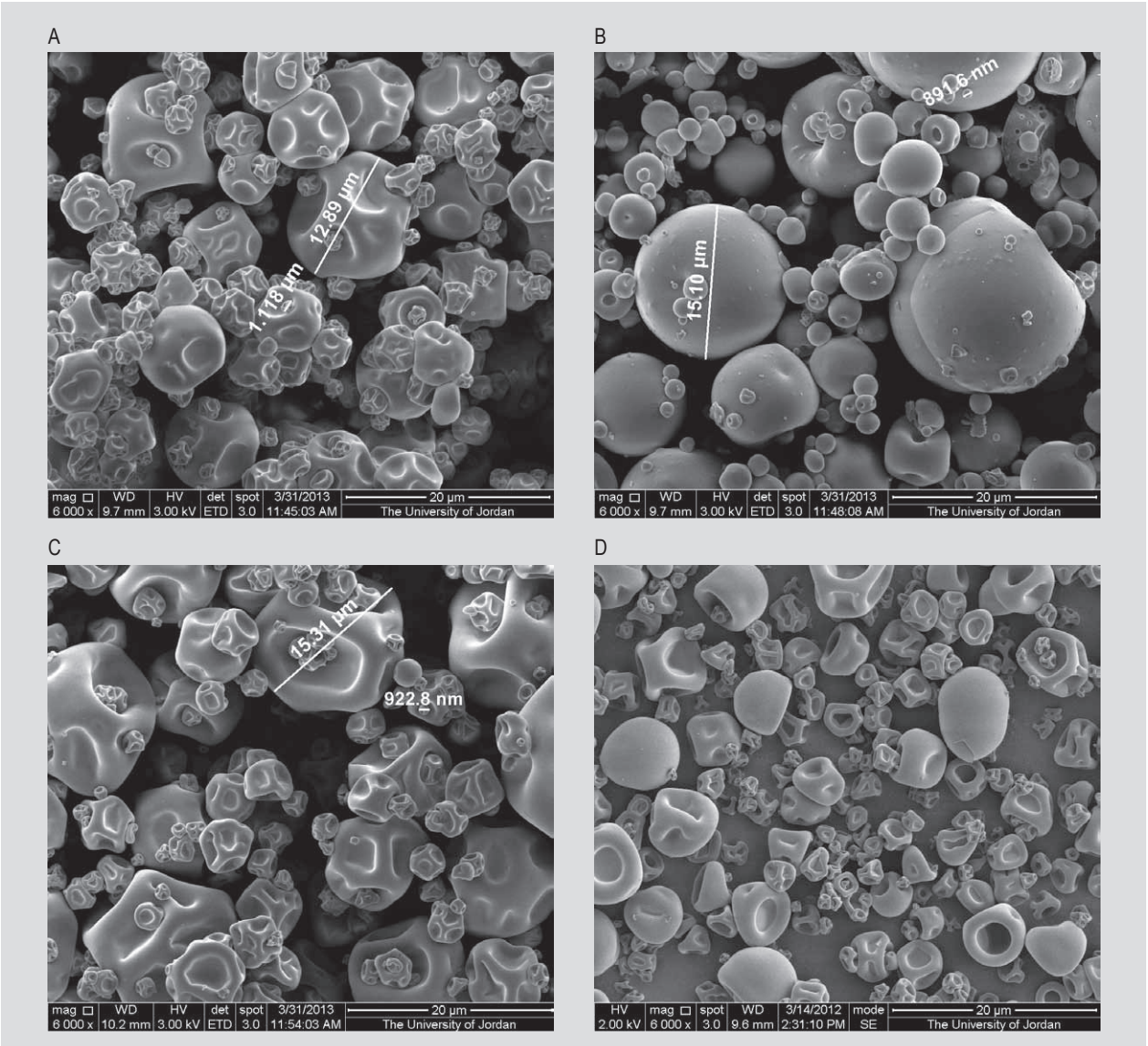

Figure 2. Micrographs of microcapsules prepared from (A) gum Arabic (GA); (B) whey protein isolate (WPI); (C) WPI+GA (1:1); and (D) WPI+GA (3:1). White arrows refer to the cracked microcapsules.

Particle size distribution

Table 5 shows a multimodal particle size distribution for all samples with mean diameters were between 4.98 and 5.74 µm. There were no significant differences (P>0.05) between mean diameters (ranged between 2.22 and 16.08 µm) of the particles size of microcapsules made of different wall materials, although the size of GA microcapsules are numerically higher than those of WPI or WPI+GA combinations.

4. Conclusions

The results indicate that GA is a better wall material for microencapsulation of CEO than WPI as determined in its ability to retain CEO with higher encapsulation efficiency and lower surface oil and to produce intact microcapsules. Combined wall materials of GA and WPI (1:1 or 1:3) produced intermediate properties. However, these properties decreased with increasing WPI concentration in the WPI+GA mixture. Lower storage temperature improved the stability of microencapsulated CEO in WPI and WPI+GA combinations. However, the storage temperature did not affect the stability of CEO in the GA microcapsules. Although GA microcapsules had numerically higher particles' size, no significant differences in particle size between treatments were detected.

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Table 5. Particle size distribution of cardamom essential oil microcapsules prepared from gum Arabic (GA), whey protein isolate (WPI) and blends of them.

Treatment	Diameter (µm)		
	d(0.1)[a]	d(0.5)[b]	d(0.9)[c]
WPI	2.22	4.98	12.77
GA	2.23	5.50	16.08
WPI+GA (1:1)	2.48	5.74	13.37
WPI+GA (1:3)	2.31	5.34	12.51

[a] The sieve diameter (µm), which divides the population into 2 halves: 90% above this value and 10% below this value.
[b] The sieve diameter (µm), which divides the population exactly into 2 equal halves that means there is 50% of the distribution above this value and 50% below.
[c] The sieve diameter (µm), which divides the population into 2 halves: 10% above this value and 90% below this value.

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