

Microencapsulation of fig seed oil rich in polyunsaturated fatty acids by spray drying

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Abstract Fig oil is a nutritious vegetable based oil type which is rich in polyunsaturated fatty acids. In the present study, several attempts have been made to produce encapsulated oil with maximum efficiency. In the first step, the effect of feed concentration was observed at 140, 150 and 160 °C and 20 % dry matter was selected. In the second step, distribution of gum arabic (GA) and maltodextrin (MD) as a wall material was determined at different temperature levels (140, 150, 160, 170 and 180 °C), and optimum MD/GA ratio and temperature were found to be 8/1 and 150 °C, respectively. In the next step, impact of wall material/fig oil ratio on the encapsulation efficiency was investigated and it was determined to be 3.0. According to the results, optimum encapsulation ratio of MD/GA/oil and temperature were 8/1/3 and 150 °C, respectively. The physicochemical properties; particle size, peroxide value, dry matter content, and encapsulation efficiency of the oil encapsulated under this condition were determined. Water content and water solubility of the powder was determined as 2.875 and 97.85 %, respectively. At the optimum conditions particle of the fig oils showed smooth surface, non-homogeneous and spherical shape, and size of the most of the particles ranged between 8 and 40 µm.

Keywords Fig oil · Encapsulation · Wall material · Drying temperature · Polyunsaturated fatty acid

Introduction

Fig (*Ficus carica* L.) is one of the oldest cultivated fruits grown especially in warm and dry regions of the World [34]. Annual fig production is about one million tones that are mostly grown in Mediterranean region. Turkey is also known as one of the most important gene centers of fig [10].

Fig is commonly consumed fresh, either peeled or not. Drying is another method for consumption and preservation style of fig. Studies have shown that dried fig fruit is rich in nutritive components such as potassium, calcium, iron, riboflavin and thiamin. Another critical factor increasing nutritional quality of dried fig is its high crude fiber content [30, 35]. Fig fruit is also a source of phenolic acids such as rutin, (+)-catechin, chlorogenic acid, (–)-epicatechin and gallic acid, many of which possess antioxidant and phytochemical properties [30].

A fig fruit contains considerable numbers of small seeds with the varying numbers depending on the maturity and size of the fruit [21]. Seeds of dried figs have oil content up to 30 % that is characterized by the abundance of linolenic acid. Other fatty acids present in the seeds are linoleic, oleic, palmitic and stearic acids, respectively [16]. As understood, fatty acids of fig oil are composed of unsaturated ones, indicating that fig oil should take part in human diet due to its potential health benefits and its commercial production should be encouraged.

Direct use of fig seed oil as a food additive or a phytochemical product is possible. However, there are some problems concerning this. One of the major problems related to the oils rich in unsaturated fatty acids is their susceptibility to oxidative degradation and formation of undesirable/potential carcinogenic compounds [5]. This

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situation decreases shelf life and sensory acceptability of foods before/during consumption.

Microencapsulation with spray-drying is a well-defined method used for capsulation of unsaturated oils and other bioactive food components [4]. Microencapsulation has been successfully used for protection of different vegetable and animal oils from oxidation [1, 5, 20, 32, 36].

Isolation and use of fig seed oil in food industry is not common in the world. Generally, it is traditionally produced and used as lubricant [16]. However, according to our research in the literature, there has not been any study associated with isolation and determination of the bioactive properties and microencapsulation of fig seed oil. Therefore, the aims of this study were (i) to determine fatty acid composition of fig seed oil and (ii) to reveal the microencapsulation efficiency and stability of microencapsulated oil using maltodextrin (MD) and gum Arabic (GA) as coating material.

Materials and methods

Materials

Fig (*Ficus carica* L.) oil (FO) that was produced by cold pressing of fig seeds was provided from a local manufacturer (Oneva, Istanbul, Turkey). Physicochemical properties of the fig oil were presented in Table 1. Chemicals used in the current study were purchased from Merck (Darmstadt, Germany).

Preparation of the emulsions

In the preliminary studies, two emulsions with two different dry matter contents (20 and 30 %) were prepared. Gum

arabic (GA) and maltodextrin (MD) (2:1 w:w) were dissolved in water and mixed at 1000 rpm for 5 min using an ultra turrax (IKA, T25). During mixing, fig oil was incorporated into the solution dropwise. Emulsifying property of GA provided formation of a homogeneous emulsion. The resulting emulsion was homogenized at 10,000 rpm for 5 min with the ultra turrax to have a proper emulsion.

Experimental design

This study consists of three experimental stages. In the first stage, optimum drying conditions in terms of temperature and solid concentration were determined based on the product yield and maximum encapsulation efficiency. For this aim, drying temperature and solid concentration were adjusted as 140–160 °C and 20–30 %, respectively. Following the determination of optimum temperature and solid concentration, optimum wall material combination (MD/GA) was determined. In this stage, MD/GA was adjusted as 2/8 and temperature was 140–180 °C. In the final stage, wall to core material ratio (MD:GA combination to fig oil) was selected to be 2, 3, and 4. Particle characteristic analyses were conducted for the sample prepared at optimum spray drying conditions. All measurements were performed at triplicate.

Microencapsulation of fig oil by spray drying

The spray-drying process was performed using a laboratory-scale Mini Spray Dryer (Büchi B-290 Labortechnik AG, Switzerland). Nozzle 0.5 mm was used; the air atomizing pressure was kept constant at 5 bars for all the experiments. The microcapsules were obtained in triplicate under the following operating conditions: air inlet temperature, 140–180 °C; Flow meter spraying air (rotameter) 0.25 bar; pump setting, 30 % and aspirator setting, 100 %.

Determination of surface oil content and encapsulation efficiency

In order to determine surface oil content, firstly, ~3 g of the powder was washed with 20 mL of petroleum ether using rough filter paper for three times. The solvent was removed from the filtrate at 60 °C using rotary evaporator and the oil was obtained. Surface oil content was calculated by determination of the difference between the weights of initial clean flask and that containing the oil residue. Based on the amount of surface oil, the encapsulation efficiency (EE) was calculated as follows:

$$EE = \frac{(TO - SO)}{TO} \times 100 \quad (1)$$

Table 1 Physicochemical properties of the fig oil

Acidity (% FFA, major fatty acid)	0.95
Peroxide value (meq. g O ₂ /kg)	3.70
Tocopherol contents (mg/kg)	
Alpha tocopherol	46.00
Beta tocopherol	–
Gamma tocopherol	3918.80
Delta tocopherol	76.50
Total tocopherol	4041.30
Fatty acid profile (%)	
Palmitic C _{16:0}	7.16
Stearic C _{18:0}	2.90
Oleic (n-9) C _{18:1}	17.13
Linoleic (n-6) C _{18:2}	30.93
Linolenic (n-3) C _{18:3}	41.11
Trans fatty acid	0.00

where TO and SO are total and surface oil contents, respectively.

Characterization of the microcapsules

Particle size distribution

The particle size distribution of the encapsulated fig oil powder was determined by a nanoparticle size analyzer (Nano ZSP, Malvern Instruments Corp., Worcestershire, U.K.) at 25 °C. A small quantity of fig oil powder was dispersed in ethanol using ultrasonic bath. The size distribution was measured by five successive reading. The particle size was expressed as the volume weighted mean particle diameter (D_{4,3}), which is the mean diameter of a sphere with the same volume, and is generally used to characterize a particle.

Morphological characteristics

Microstructural/morphological properties of the capsule was analyzed by scanning electron microscopy (SEM) (JSM-5510, JEOL Ltd., Tokyo, Japan) under high vacuum with a distance of 8 mm. 5 kV accelerated voltage was applied to have secondary electron images. The images related with the capsules were acquired using Control User Interface (version 5.21; JEOL Ltd.).

Peroxide value

Three to 5 g of FO microcapsules were weighed into a flask and incorporated with 30 mL of acetic acid:chloroform solution (3:2 v:v). Following the manual homogenization of the mixture, 0.5 mL of saturated potassium iodide (KI) was added and the mixture was further homogenized for 1 min. After holding in dark for 10 min, 30 mL of distilled water was added and the titration was performed with 0.002 N of sodium thiosulfate (Na₂S₂O₃) and starch solution was used as an indicator. Peroxide value was calculated with the following equation:

$$PV = \frac{(A \times N \times 1000)}{E} \quad (2)$$

where *A* is the amount (mL) of sodium thiosulfate solution spent, *N* the normality of thiosulfate and *E* the sample amount (g) weighed (AOAC, [2]).

Moisture content

Moisture content of the microencapsulated fig oil was determined by gravimetrically. A 4–5 g of the sample was weighed in petri plates and dried under vacuum for 24 h at

70 °C. The results were expressed as percent moisture by [13].

Solubility in water

Solubility of the fig oil microcapsules in water was determined by the modified method described by [13]. A 0.4 g of the sample was mixed with 40 mL of distilled water and homogenized by vortex for 1 min. Following the centrifugation of the mixture at 5000 rpm for 10 min, 20 mL of the supernatant was dried at 70 °C for 24 h under vacuum conditions. The results were expressed as percentage solubility.

Statistical analysis

All measurements were conducted in triplicate and their mean values were presented. Statistical analysis of the data was carried out using the Statistica 8.0 (StatSoft Inc., Tulsa, UK) software package program. Analysis of variance (ANOVA) using Duncan's multiple range tests at 95 % of confidence (*P* < 0.05) was used for determination of the significant differences between the results.

Results and discussion

Effect of total solid concentration (TSC) and drying temperature on the encapsulation efficiency

The influence of total solid concentration of feed emulsion on the encapsulation efficiency of the process is tabulated in Table 2. Encapsulation efficiency changed between 40.69 and 77.10 %. Encapsulation efficiency was significantly affected by solid concentration and drying temperature. Encapsulation efficiency generally decreased with increasing temperature for the feed including 20 % while the yield increased. Considering the impact of solid concentration, it could be concluded that at all drying temperature levels, feed solid concentration of the 20 % had higher encapsulation efficiency and product yield. Encapsulation efficiency increased with lowering feed emulsion concentrations. Feed emulsion prepared with higher solid concentration led to adhesion of powders to column, resulting in decreasing powder yield. Therefore, 20 % concentration was determined as the optimum solid concentration for feed emulsion.

Our findings are contrary to those reported by several researchers who found that rising solid amount in the solution led to increasing encapsulation efficiency [9, 7, 12, 33]. They claimed that high solid content of the feed decreases the time desired for forming a semi-permeable layer at the surface of the particle to be

Table 2 Effect of total solid concentration of feed and drying temperature on the powder yield and encapsulation efficiency

Solid concentration (%)	Temperature (°C)	Encapsulation efficiency (%)
20	140	77.10 ± 2.04 ^{A,a}
	150	74.78 ± 0.81^{A,a}
	160	72.56 ± 0.53 ^{A,b}
30	140	40.69 ± 3.62 ^{B,b}
	150	63.72 ± 1.35 ^{B,a}
	160	61.98 ± 2.33 ^{B,a}

Different superscript uppercase letters show significant ($P < 0.05$) differences between concentrations within the same temperature. Different superscript lowercase letters show significant ($P < 0.05$) differences between temperatures within the same solid concentration. The bold highlights the selected condition for the second step

encapsulated or dried, which indicated that maximum amount of feed solid could be used to improve encapsulation process. Aggregation of core material on the surface of particle becomes difficult in the case of rapid formation of solid surface depending on the low levels of surface oil content [14]. Moreover, the direct correlation between encapsulation efficiency and solid content of feed could also be explained by the fact that circulation movement inside droplets could be prevented by higher emulsion viscosity directly proportional with solid content, which accelerates solid layer formation [15]. Encapsulation efficiency is also associated with the droplet size of the emulsion which decreases with increasing total solid content [28] and the increasing efficiency with reducing droplet size has been reported in many studies [23]. Despite of all suggestions mentioned above, there are also several contrary findings about the relation between solid content and encapsulation efficiency in the literature which reported that the increase in efficiency proceeds up to optimum level and at higher levels than optimum solid content it starts to decrease [3, 29]. Two different statements are possessive for explaining of the existence of optimum feed solid content. One of them is associated with the saturation of the feed solution. As the solution reaches to its saturation level, more wall material could not be solved and therefore, undissolved wall material cannot play any role in encapsulation [9]. The higher the feed solid concentration the larger exposure and the slower formation of separated droplets throughout atomization and the more difficulties being encountered during droplet occurrence [3, 12, 15]. Optimum level of the feed solid content could also be dependent to wall material type and their concentration distribution and the spray drying conditions. With reference to our findings in the light of the comments mentioned above, it could be deduced that optimum feed solid content could be between 20 and 30 %. After reaching to the optimum encapsulation efficiency decreased and feed solid content of 20 % was found as higher than that of 30 %. When comparing the encapsulation efficiency, 20 % solid

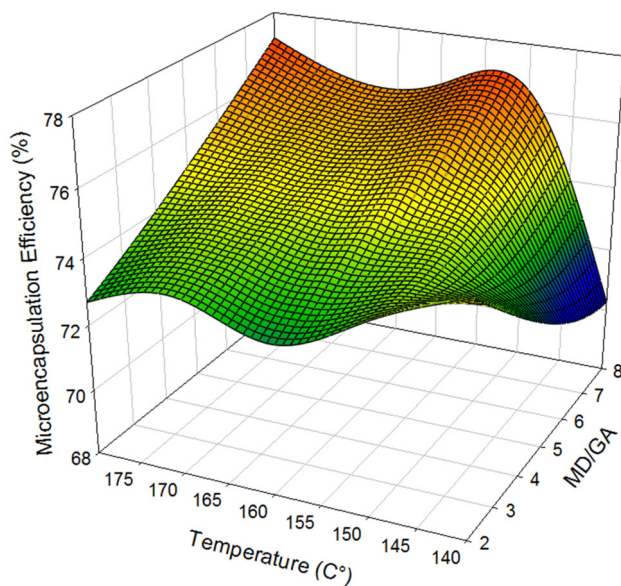
content was more suitable for the second step of the study. Although the highest efficiency was obtained as a result of the process performed at 140 °C, the temperature of 150 °C was found to be more preferable when the powder amount fabricated during encapsulation process was taken into consideration.

After determination of feed solid content, the effect of inlet temperature and MD/GA ratio of a wall material on the encapsulation efficiency was determined in the second part of the study. Five temperature (140, 150, 160, 170 and 180 °C) and two ratio (2 and 8) levels were selected for this aim and totally, 10 different encapsulated fig oils were fabricated under the conditions mentioned in Table 3 and shown Fig. 1. As seen, encapsulation efficiency changed between 70.27 and 77.29 %. Encapsulation efficiency was significantly affected by MD/GA ratio and MD/GA ratio of 8 was found as more effective than that of 2 in terms of the encapsulation efficiency with exception of encapsulation process carried out at 140 °C. Therefore, it could be concluded that optimum temperature could also be dependent on the wall material type or combination. Similar results were reported by [25] who encapsulated naringin with a resistant MD. They studied the effect of resistant MD concentration and inlet temperature on the encapsulation efficiency and they found that as efficiency was found as higher at 150 °C when MD was added to the feed solution in concentration of 20 %; however, at 180 °C, higher efficiency was observed in the sample prepared with 40 % MD. Therefore, wall material combination is very important for optimizing process condition. The polymer layer formed on the surface of the core material influenced the retention characteristics and film-forming capability [5], which depends on type and concentration of polymer used as a wall material. When the MD/GA ratio was selected as 8, it was possible to produce to obtain more encapsulation efficiency when the process temperature was adjusted to 150, 160, 170 and 180 °C. They were found as higher than process performed at 140 °C. In order to prevent degradation of oil due to the applied temperature, 150 °C was

Table 3 Effect of different maltodextrin/gum arabic ratio (MD/GA) on the solid yield and encapsulation efficiency

Ratio (w/w)	Temperature (°C)	Encapsulation efficiency (%)
2	140	74.92 ± 0.48 ^{A,a}
	150	73.78 ± 0.28 ^{B,a}
	160	72.56 ± 0.33 ^{B,a}
	170	73.37 ± 0.10 ^{B,a}
	180	72.76 ± 0.26 ^{B,a}
8	140	70.27 ± 0.17 ^{B,b}
	150	76.62 ± 0.14 ^{A,a}
	160	76.44 ± 0.53 ^{A,a}
	170	76.18 ± 0.15 ^{A,a}
	180	77.29 ± 0.12 ^{A,a}

Different superscript uppercase letters show significant ($P < 0.05$) differences between MD/GA ratios within the same temperature. Different superscript lowercase letters show significant ($P < 0.05$) differences between temperatures within the same MD/GA ratio. The bold highlights the selected condition for the second step. The bold highlights the selected condition for the third step. MD maltodextrin, GA gum arabic

**Fig. 1** Effect of temperature and Maltodextrin/Gum Arabic ratio (MD/GA) on the microencapsulation efficiency of fig oils

selected as suitable temperature level for encapsulation of the fig oil.

Effect of wall to core material ratio on the encapsulation and product yield

After selection of the optimum wall material combination and process parameters, the effect of total oil (O) content

of emulsion on microencapsulation and product yield was investigated. Total oil content of emulsion is another important factor affecting powder properties, microencapsulation, and product yield. In our study, ratio of the wall material to oil content (WM/O) was adjusted to 2, 3 and 4 (w/w). Powder yield and encapsulation efficiency were affected by oil content of the emulsion. Powder efficiency was found to be 3.71, 20.34 and 11.64 % for 2, 3, 4 wall material to oil ratio, respectively, showing that high oil content of the emulsion resulted in lower product yield. Encapsulation efficiency was obtained to be 72.57 and 75.61 % for 3 and 4 WM/O, respectively. Microencapsulation efficiency (ME) was not obtained for 2 WM/O emulsion due to the low powder efficiency. The results indicated that ME decreased with increasing oil content of emulsion. The low ME in high core material ratio can be explained by increasing of the droplet size and decreases emulsion stability with increasing oil ratio in the emulsion. Similar results were reported by polavarapu et al. [26]. They stated that ME was significantly increased with decreasing oil content in emulsion. In their study, ME was found to be 90 and 98 % at 25 and 50 % (w/w) emulsion composition, respectively. The decrease in ME at 50 % (w/w) was explained by the lower amount of continuous matrix in emulsion formulations compared to those of the 25 % w/w oil content. However, very low core to wall material ratio such as lower than 1:5 can be undesirable due to low oil content of the powder product in microcapsules [8]. These authors also recommended that wall material to core ratio between 2 and 4 was acceptable for many food applications and high level of the oil content in emulsion (higher than 50 %) caused to decrease in ME by increasing surface oil content. Wall to core material ratio was reported as between 3/1 and 4/1 (w/w) by several authors [19]. As a conclusion, our study showed that optimum temperature, solid concentration, wall material composition and wall to core material ratio were found as 150 °C and 20 %, 8/1 MD/GA (w/w), and 4/1(w/w), respectively.

Microcapsule characteristics

Water content and solubility

Water content of the fig oil powder was determined as 2.875 %. Water content of the fig oil encapsulated powder is agreement with powder of encapsulated other edible oils reported in the literature (0.41–4 %) [22, 19, 31]. Water content of the encapsulated oil powder is critical for promoting oxidative deterioration and off flavor [22]. Water content is influenced by several factors such as drying temperature, drying rate, emulsion viscosity, porosity of the wall material and feeding rate [6, 19]. Increasing of the drying temperature and decreasing of the feeding rate led to

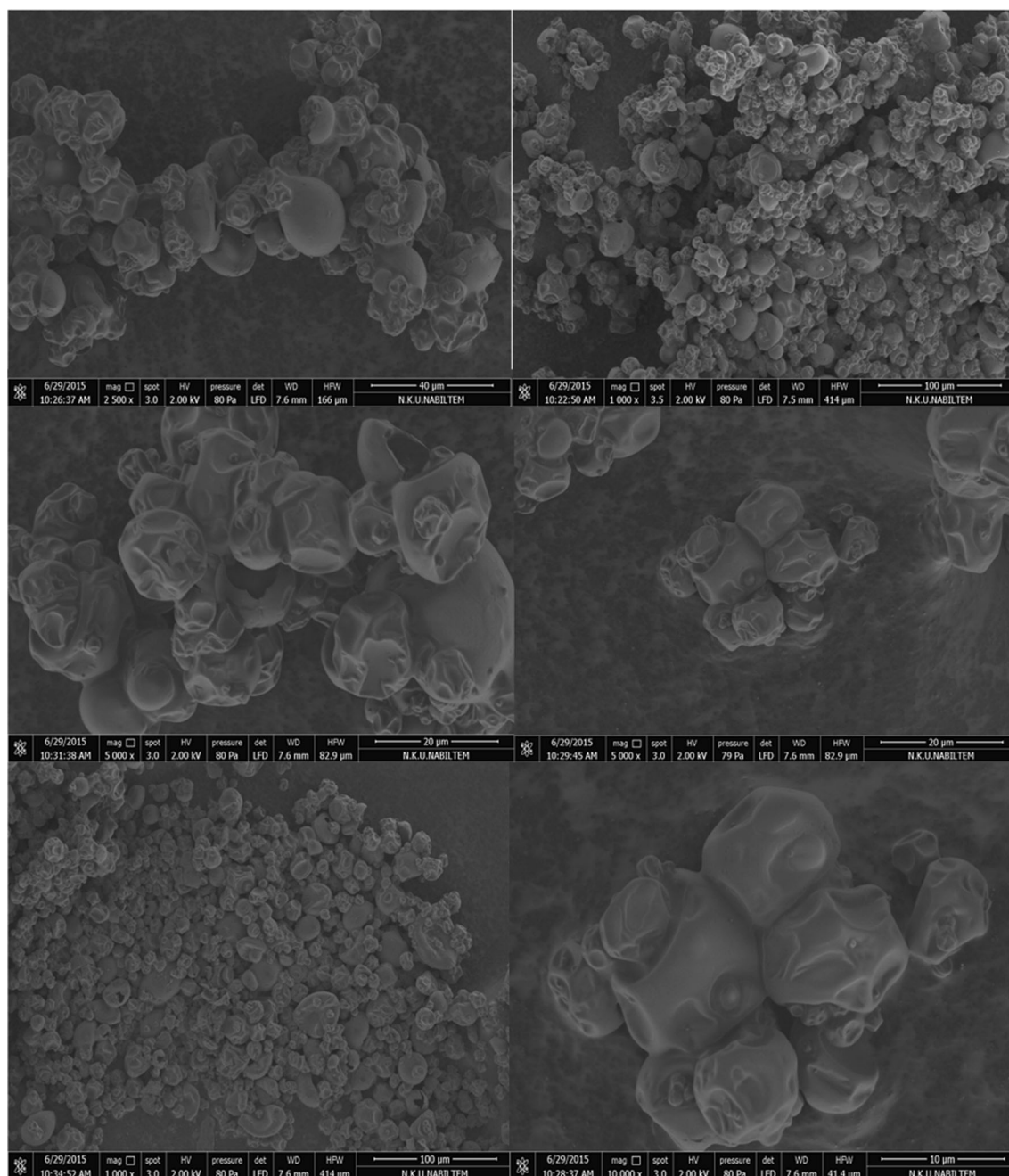


Fig. 2 Scanning electron microscopy images of microencapsulated fig oil powders obtained at optimum conditions

lower moisture contents of the powders. Water content of the encapsulated fig oil was lower than 3–4 %, which is the maximum limit of the water content in dried powders for food industry [18]. Spray drying temperature should be increased in order to reduce water content in encapsulated fig powder because water content is close to maximum limits indicated above. Water solubility of the encapsulated fig powder was found as 97.85 %. High solubility and relatively high moisture content can be explained by use of MD, which is a highly hydrophilic sugar, as a wall

material. These results were also supported by Koç et al. [19] who reported that water content of the powder produced with MD was higher than those of the others.

Peroxide value

Peroxide value is an indicator of the oxidative stability and initial oxidation of the edible oils. Peroxide value of the encapsulated oil can be influenced by several factors such as temperature, homogenization rate and wall material

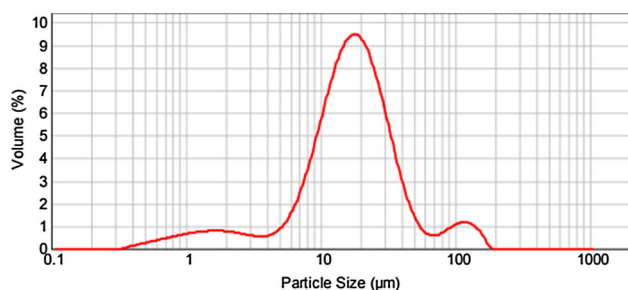


Fig. 3 Particle size distribution of the encapsulated fig oil

composition [11]. Peroxide value of the cold pressed liquid and encapsulated figs oil were found as 7.17, and 7.36 (meq. O_2 /kg oil), respectively. A slight increase was observed in peroxide value of the figs oil after encapsulation, suggesting that microencapsulation process of the figs oil at optimum conditions should be used in encapsulation of figs oil to lower peroxidation. Similar results were reported by Carneiro et al. [5] who found that oxidative stability of edible oil was significantly affected from wall material composition while MD was a good protective agent against oxidation.

Particle morphology and size

Figure 2 exhibits the SEM images of the microencapsulated fig oil fabricated under optimum drying conditions. As can be seen, particle of the microencapsulated fig oil showed smooth surface, non-homogeneous and spherical shape. Smooth surface of the encapsulated fig oil can be explained by low molecular weight sugar content of MD. Loksuwan [24] reported that low molecular weight sugar of the MD could show plasticizing effect on the particle surface. The figure also shows that visible hole and cracks was observed on some particles. Some cracks and holes observed on the particle surface was explained by the mechanical stress resulted from drying phase [17]. Positive correlation between high microencapsulation efficiency (higher than 83 %) and low shrinkage and cracks was reported in the literature [27]. Our microencapsulation efficiency was about 76 %. Therefore, cracks and holes seen on the particle surface can be eliminated by optimization of other parameters for obtaining higher microencapsulation efficiency levels.

Most of the particles of the microencapsulated fig oil is between 8 and 40 μm in size (Fig. 3), and $D_{4,3}$ (volume mean diameter) and $D_{3,2}$ (surface weighted mean) was found as 23.422 and 7.696 μm , respectively. $D_{4,3}$ value was reported to be 15.32–23.03 μm for encapsulated flaxseed oil [5], 10.14–54.36 μm for encapsulated red pitaya seed oil and 10.83–55.98 μm for encapsulated virgin olive oil [19]. As can be seen, $D_{4,3}$ value of the encapsulated fig

oil is comparable with the literature. Uniformity of the particle distribution can be considered as high (79.8 %). Our results is agreement with other studies related with the encapsulation of other edible oils [22]. The low particle diameter and high uniformity can be attributed to high microencapsulation efficiency. In our study, low particle diameter and high uniformity in spite of the moderate level of microencapsulation efficiency can be explained by the presence of MD and GA in the wall material composition, and low drying temperature. Carneiro et al. [5] reported that particle diameter of the oil capsules capsulated with MD was lower than those of the capsulated with GA and MD combination. These results were associated with the high viscosity of the GA and MD emulsions. It can be concluded that encapsulated figs oil at optimum drying conditions showed good particle properties in terms of particle size and morphology.

Conclusion

This study is the first attempt for encapsulation of fig oil under different processing conditions. This study suggested that encapsulation efficiency of the fig oil was significantly affected by temperature, solid concentration, wall to core material ratio, and wall material composition (GA/MD). Optimum temperature, solid concentration, wall material composition and wall to core material ratio were found as 150 $^{\circ}C$ and 20 %, 8/1 MD/GA (w/w), and 4/1(w/w), respectively. Encapsulated fig oil produced under optimum conditions showed good particle characteristic such high water solubility, low water content, and homogeneous particle size distribution and good morphology. This study also suggested that the quality of the fig oil could be preserved successfully by microencapsulation carried out at optimum conditions since peroxide value of the fig oil did not change significantly after microencapsulation process. In this study, moderate level of the encapsulation efficiency (74 %) was obtained. From this point of the view, process parameters and other conditions should be adjusted for the improving microencapsulation efficiency and product quality.

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