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Microencapsulation of flaxseed oil produced by sequential rotor stator homogenization and spray drying – Optimization of emulsion composition

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ABSTRACT

Microencapsulation of flaxseed oil (FO) has received lots of attention in the food and biopharmaceutical industries. To produce FO microcapsules, aqueous emulsions of FO with polymeric carbohydrates (maltodextrin (MD) with dextrose equivalent (DE) 19, gum Arabic (GA) and modified starch (MS)) were prepared by a rotor stator homogenization and subsequently, dehydration of emulsions were performed by spray drying (SD). The objective of this research was to study the effects of different combinations of polymeric carbohydrates with FO in emulsion to obtain maximum encapsulation efficiency (EE). A 3 factorials–3 levels Box–Behnken design was used for the optimization purpose. The maximum EE was achieved using 0.79 MD-GA ratio, 20.23% MS and 24.62% FO in emulsion. Microcapsules obtained by optimum condition had EE 77.68%, particle size (D_{32}) 120.0 ± 0.43 μm, moisture content1.6 ± 0.13%, wettability 192 ± 5.5 s, solubility 75.49 ± 1.3%, bulk density 0.31 ± 0.025 g mL^{-1}, tapped density 0.36 ± 0.01 g mL^{-1}, Carr's Index 13.88 ± 0.01% and Hausner Ratio 1.16 ± 0.01.

KEYWORDS

flaxseed oil, emulsion, microencapsulation, rotor stator homogenizer, spray drying

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INTRODUCTION

Wide ranges of nutritional benefits have catapulted flaxseed oil (FO) to the forefront of food and biopharmaceutical industries (Kaur et al., 2014). FO is a rich source of α-linoleic acid, an essential ω-3 polyunsaturated fatty acid (PUFA) which plays a crucial role in the wide ranges of physiological functions and maintaining overall health by the modulation of inflammatory reactions in hepatocytes and adipose tissues, metabolic pathway of fatty acids and synthesis of ketone body, blood pressure, mortal cardiac diseases, and many more. Furthermore, FO contains a variety of antioxidants, which are supposed to provide oxidative stability of fatty acids (Bali'c et al., 2020; Zou et al., 2017). The reductions of antioxidants in FO due to improper processing and storage may produce both primary and secondary oxidation products of fatty acids, which have negative impacts (Li et al., 2023; Zwyrzykowska-Wodzińska et al., 2023).

ω-3 PUFAs are highly susceptible to oxidation, which reduces the quality of FO by producing unpleasant off-flavors and aromas. Furthermore, oxidation of fatty acids reduces the potential health benefits of FO (Goyal et al., 2014; Gunstone, 2011). In order to preserve the shelf life of FO, addition of antioxidants to oil was considered (Omar et al., 2010; Lu et al., 2019, 2021). Furthermore, microencapsulation has emerged as a promising technique to preserve the shelf life of FO. In the microencapsulation process, droplets of FO are covered by a thin film coating, which is commonly known as a matrix or wall material. Therefore, wall material or matrix protects the fatty acids in FO against heat, light, moisture, oxidation and interaction with other molecules during processing and storage. Furthermore, it also influences the release of bioactive compounds from matrix during gastrointestinal digestion (Martins et al., 2022; Gouin, 2004). Microencapsulation of bioactive compounds can be performed in various ways. Conventional approach to microencapsulation is dehydration of emulsion containing bioactive compounds by freeze-drying (FD) and spray-drying (SD). Therefore, emulsion preparation and characteristics of emulsion are important issues because they play a pivotal role in the characteristics of microcapsules. Emulsion can be prepared by different technologies, mentioned below.

(A) Low energy consuming technologies (Friberg et al., 2011; Charcosset et al., 2004; Lapez-Montilla et al., 2002; Solans et al., 2016):
  (a) Phase inversion temperature
  (b) Membrane emulsification
  (c) Spontaneous emulsification of two immiscible liquids without any significant external thermal or mechanical energy

(B) High energy consuming technologies (Gaikwad and Pandit, 2008; Stang et al., 2001):
  (a) Ultrasound generator
  (b) High pressure homogenizer

Moreover, microencapsulation of bioactive compounds can be performed by spray granulation and liposome entrapment methods (Anwar et al., 2010).

Technologies to prepare microcapsules and characteristics of matrix influence the chemical and physical properties of microcapsules. These include moisture content, size and shape, bulk density, tapped density, flowability and cohesiveness (Onsaard and Onsaard, 2019).

Rotor-stator homogenization is a widely used method to prepare emulsion, where the intense shear forces produce fine droplets of oil and disperse them into the matrix in emulsion

(Gaikwad and Pandit, 2008). Subsequently, dehydration of emulsion could be performed by FD or SD, resulting in encapsulation of oil droplets within the matrix (Van der Schaaf and Karbstein, 2018). Several parameters of FD, such as temperature and system pressure, operational time of drying and the rate of freezing of emulsion influence the characteristics of the microcapsule (Rezvankhah et al., 2019; Haseley and Oetjen, 2017). Similarly, several parameters of the SD process, such as outlet and inlet air temperatures, and flow rate of emulsion can influence the size, morphology, moisture content and other properties of microcapsules (Anandharamakrishnan and Ishwarya, 2015; Rezvankhah et al., 2019).

Selection of appropriate matrices is also a critical issue because the characteristics of wall material influence the moisture content, size and shape, bulk density, tapped density, flowability and cohesiveness of microcapsules. Furthermore, characteristics of matrices influence the release of bioactive compounds from matrices to environment in a controlled manner (Anandharamakrishnan and Ishwarya, 2015; Desai and Park, 2005). Commonly used wall materials for the microencapsulation of FO include maltodextrin (MD) (Can Karaca et al., 2013; Gallardo et al., 2013; Rubilar et al., 2012; Carneiro et al., 2013; Avramenko et al., 2016; Thirundas et al., 2012; Fioramonti et al., 2019; Fioramonti et al., 2017; Bajaj et al., 2017; Tontul and Topuz, 2014), gum Arabic (GA) (Gallardo et al., 2013; Rubilar et al., 2012; Carneiro et al., 2013; Thirundas et al., 2012; Tonon et al., 2011; Pedro et al., 2011), methyl cellulose (MC) (Gallardo et al., 2013), modified starch (MS) (Carneiro et al., 2013; Tonon et al., 2012; Barroso et al., 2014), sodium alginate (SA) (Fioramonti et al., 2017, 2019), whey protein isolate (WPI) (Gallardo et al., 2013; Fioramonti et al., 2017; Domian et al., 2017), whey protein concentrate (WPC) (Carneiro et al., 2013; Tonon et al., 2012; Goyal et al., 2015; Fioramonti et al., 2019; Tontul and Topuz, 2014), sodium caseinate (SC) (Goyal et al., 2015) and vegetable proteins (VPs) (Can Karaca et al., 2013; Avramenko et al., 2016; Kaushik et al., 2016; Bajaj et al., 2017; Domian et al., 2017). Each wall material offers distinct advantages and disadvantages, mentioned before (Yakdhane et al., 2021). Generally, polysaccharides are hydrophobic, have a high glass transition temperature (T_g), and have the ability to produce a fine and dense network, and glassy thin film (Anandharamakrishnan and Ishwarya, 2015). Proteins are amphiphilic in nature and have good film-forming properties and high water activity due to their low T_g. Therefore, combinations of different wall materials, mainly polysaccharides and proteins, have been often used for the microencapsulation of FO instead of single wall materials to get appreciable encapsulation efficiency (EE) (Can Karaca et al., 2013; Gallardo et al., 2013; Carneiro et al., 2013; Avramenko et al., 2016; Thirundas et al., 2012; Fioramonti et al., 2019; Fioramonti et al., 2017; Bajaj et al., 2017; Tontul and Topuz, 2014; Domian et al., 2017). Very interestingly, it can be mentioned that in some experiments, alone GA (Gallardo et al., 2013; Tonon et al., 2011, 2012; Pedro et al., 2011) and MS (Barroso et al., 2014; Tonon et al., 2012), and combinations of GA with MD (Gallardo et al., 2013; Rubilar et al., 2012; Carneiro et al., 2013) and MD with MS (Carneiro et al., 2013) were used instead of the combination of polysaccharide and protein. MDs are hydrolyzed starch and have different dextrose equivalent (DE) based on chain lengths of sugar molecules. The physicochemical and biochemical properties of MDs vary based on DE. For example, solubility, viscosity and water activity of MD are increased with an increase of DE value (Xiao et al., 2022). These characteristics of MD offer unique features of microcapsules. It was reported that microcapsules with MD having high DE, produced by SD can promote the stability of encapsulated bioactive compounds because matrices are more uniform after SD, which is not offered by MD having DE 10 (Ghani et al., 2017). MDs do not offer emulsifying property, which

encourages the use of MD along with other polysaccharide, such as GA having emulsifying property, high solubility, low viscosity and T_g. MS mainly consists of amylopectin, has low T_g and high solubility. Furthermore, it has high film-forming property and provides high retention of bioactives (Yousefi et al., 2011; Du et al., 2014).

In the present investigation, different proportions of polymeric carbohydrates, such as MD with dextrose equivalent (DE) 19, GA and high amylose containing MS from maize were used as a wall material for the microencapsulation of FO. Aqueous emulsions were prepared by the combinations of wall materials with FO by the laboratory rotor-stator homogenizer (RSH) prior to dehydration of emulsion by SD. A 3 factorials–3 levels Box–Behnken design was used to understand the optimum proportion of polymeric carbohydrates and FO to prepare emulsions and obtain maximum encapsulation efficiency (EE). Microcapsules having highest EE were characterized by particle size (D_{32} and D_{43}) and morphology, moisture content, wettability, solubility, bulk density (BD), tapped density (TD), and flowability and cohesiveness (Carr's Index and Hausner Ratio).

MATERIALS AND METHODS

Materials

Cold-pressed FO was purchased from a grocery shop in Budapest, Hungary. Its composition per 100 mL: 100 g of fat, consisting of 10 g of saturated fatty acids, 20 g of monounsaturated fatty acids, and 70 g of polyunsaturated fatty acids, according to the information provided by the manufacturer. MD having DE 19, GA and high amylose MS from maize were purchased from the Buda Family Kft, Hungary, Bi-Bor Kft, Hungary and Ingredion, Hungary, respectively. Soy lecithin was purchased from Házi Ipari Szolgáltató és Kereskedelmi Kft., Hungary. All reagents and solvents were analytical grade, purchased from Ecolab-Hygiene Kft Hungary. De-ionized water (18.2 MΩ·cm) used in experiments were collected from Milli-Q Synergy/Elix water purification system (Merck-Millipore, Molsheim, France).

Microcapsule preparation

FO microcapsule was prepared by dehydration of emulsion by SD. The process is represented in Fig. 1.

Preparation of the emulsion by RSH. Prior to preparing emulsion, aqueous solutions of polymeric carbohydrates, such as MD having DE 19, GA and MS, and emulsifier soya lecithin were prepared by vigorous stirring at temperature 30 °C according to the experimental plan. Subsequently, the temperature of solutions was reduced to 25 °C. FO was added dropwise to the prepared aqueous solutions of polymeric carbohydrates under high shear. The homogenization process was carried out using a laboratory-scale RSH (DLAB D-160, `Scilogex, Rocky Hill, CT, USA). The homogenization speed was performed with 15,000 rpm for 5 min (Wang et al., 2022).

Dehydration of emulsion by spray drying. Emulsions were dehydrated by a laboratory-scale spray dryer (LabPlant SD-05, Keison, Chelmsford, UK) equipped with a peristaltic pump and a nozzle having diameter 0.5 mm. During the SD process, the emulsions were continuously stirred using a magnetic stirrer to ensure its homogeneity. In the spray dryer, inlet air temperature,

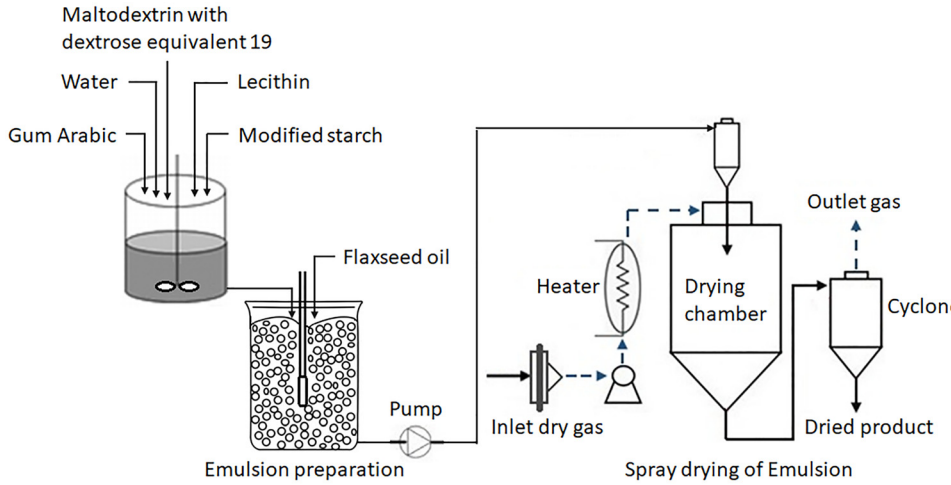

Fig. 1. Microcapsule of FO was prepared by the sequential steps: preparation of aqueous emulsion of polymeric carbohydrates (maltodextrin with dextrose equivalent 19, gum Arabic and modified starch), lecithin with flaxseed oil, followed by the dehydration of emulsion by spray drying

outlet air temperature, airflow rate and compressed air pressure were set to 185 ± 5°C, 105 ± 5°C, 3.6 bar and 74 m^3/h, respectively. Microcapsules were obtained from the glass chamber of spray dryer and they were kept in a glass container. They were stored under dark conditions at temperature 4 °C until analysis (Barroso et al., 2014).

Experimental design

Experimental plans were designed by the Design-Expert 13.0.1.0 software, (Stat-Ease, Minnesota, USA). Response surface methodology (RSM) was applied to investigate the effect of different combinations of wall materials on EE. A 3 factorials–3 levels Box–Behnken design was used for the optimization purpose because it has a good ability to understand main effects and interactions among three factors, without requiring a full factorial analysis. The box Behnken design ensures accurate assessments without confusing unworkable combinations. In addition, it can execute with fewer runs than full factorial designs, which saves cost of chemicals, materials, time and resources (Beg and Akhter, 2021). For the optimization of EE, three independent variables were used and were coded according to Table 1.

Table 1. Coding of independent variables for the optimization of emulsion

Variable	Coded X_i	Coded level		
		−1	0	1
Ratio of MD and GA (MD/GA)	X1	0	0,5	1
Amount of MS (%)	X2	0	20	40
Amount of FO (%)	X3	10	25	40

Emulsion characterization

Emulsion stability. Emulsion stability was evaluated through phase separation. After preparation of emulsion, 25 mL of emulsion was poured into a graduated cylinder and kept for 24 h at room temperature. The upper phase was measured and used for the calculation of separation's percentage according to the following formula (Tonon et al., 2012):

$$Separation\ (\%) = \left(\frac{Height\ of\ upper\ phase}{Initial\ height\ of\ emulsion}\right) \times 100 \quad (1)$$

It is known that zeta potential is crucial to understand the stability of emulsion and it represents the tendency of dispersed particles to aggregate in emulsion. The values of zeta potential of emulsions above ±30 mV represent the stability of emulsion (Rodriguez-Loya et al., 2023). Therefore, a zeta potential analyzer Zetasizer Nano ZS from the United Kingdom was also used to measure the zeta potential of emulsion.

Droplet size and its distribution (span). Droplet size and their distribution in emulsions were assessed by a laser particle size analyzer (Bettersize ST, Bettersize Instruments Ltd., Dandong, China). Water suspension of emulsion was considered for measurement. The size distribution of droplets was monitored during each measurement until successive readings became constant. Diameters of droplets were expressed by the value of D_{32} (volume/surface mean) and D_{43} (mean diameter over volume), also known as the Sauter mean and the DeBroukere mean, respectively (Kowalczuk and Drzymala, 2015). The distributions of droplets size in emulsions were represented by the span value. Span value was calculated according to the following equation:

$$Span\ (-) = \frac{D90 - D10}{D50} \quad (2)$$

Where, D_{90} corresponds to the value of droplet diameter below 90% within the whole sample, D_{10} corresponds to the value of droplet diameter below 10% within the whole sample and D_{50} corresponds to the value of particle diameter below 50% within the whole sample.

Characterization of microcapsules

Encapsulation efficiency. For the evaluation of EE, 15 mL of Hexane were added to 2 g of microcapsules at room temperature, and it was shaken for 2 min in order to extract the surface oils from microcapsules. Subsequently, it was filtered by a Whatman filter paper 1. The retailed solids on the filter paper was rinsed three times with 20 mL of Hexane to recover encapsulated oils and filtered again. Filtrates were left at temperature 60 °C to evaporate solvents. EEs of microcapsules were calculated according to the following correlation (Tonon et al., 2012):

$$EE\% = \left(\frac{Total\ oil - Surface\ oil}{Total\ oil}\right) \times 100 \quad (3)$$

Particle size and its distribution (span). A Bettersize ST laser scattering particle size distribution analyzer (Bettersize Instruments Ltd. China) was used to determine the particle size distribution (PSD) of microcapsules. A small quantity of microcapsule was carefully suspended in

anhydrous ethanol with mild agitation and considered for scanning. The size of microcapsules (D_{32} and D_{43}) and the size distribution were measured until successive readings became constant (Barroso et al., 2014).

Moisture content. Moisture content of microcapsules was measured gravimetrically using a moisture analyzer (KERN MLS; KERN & SOHN GmbH, Balingen, Germany). 1 g of microcapsule was placed on the heating plate of moisture analyzer and a constant heating temperature 70 °C was maintained until a stable weight was reached (Santana et al., 2013).

Bulk density (BD), tapped density (TD), followability and cohesiveness. BDs of microcapsules were measured according to the protocol, mentioned by Getachew and Chun (2016). 2 g of microcapsules were placed in a 20 mL graduated cylinder and the height of powder within the cylinder was measured. BDs of microcapsules were calculated according to the following equation:

$$BD\left(\frac{g}{mL}\right) = \frac{Mass}{Volume} \tag{4}$$

TDs of microcapsules were measured according to the protocol, mentioned by Goula and Adamopoulos (2008) with some modification. 2 g of microcapsules were placed in a 20 mL of graduated cylinder and then manually tapped by lifting and dropping under its own weight from 5 cm of height. TDs of microcapsules were calculated according to the following equation:

$$TD\left(\frac{g}{mL}\right) = \frac{Mass}{Tapped\ volume} \tag{5}$$

Flowability of microcapsules was evaluated by Carr Index (CI) and was calculated from the value of the BD and TD of microcapsules (Shah et al., 2008):

$$CI(\%) = \frac{TD - BD}{TD} \times 100 \tag{6}$$

Cohesiveness of microcapsules was evaluated by Hausner ratio (HR) and it was assessed from the values of BDs and TDs of microcapsules (Shah et al., 2008). HRs of microcapsules were calculated according to the following correlation:

$$HR = \frac{TD}{BD} \tag{7}$$

Wettability. 2 g of microcapsules were added to 200 mL of deionized water at room temperature without agitation. The time duration when microcapsules were sediment at the bottom of water was considered for wettability of microcapsules (Fuchs et al., 2006).

Solubility. Solubility of microcapsules was determined by dissolving 2 g of microcapsule in 25 mL of deionized water. Solutions were filtered through Whatman paper 42. Subsequently, filter papers and residues were dried at temperature 105 °C in an oven for three hours. Weight of filter papers was measured when their temperature turned to room temperature. The solubility of microcapsules was calculated using the following equations (Cahyani et al., 2018):

$$\text{Solubility } \% = 100\% - \text{Residue}\% \tag{8}$$

where,

$$Residue\ (\%) = \frac{\text{Weight of filter paper and residue } - \text{Weight of filter paper}}{Weight\ of\ sample} \times 100 \tag{9}$$

Morphology. A Field Emission-Scanning Electron Micrographs (FE-SEM) JSM 5500 LV (Jeol Ltd., Japan) was used to understand the morphology of microcapsules. Microcapsules were fixed onto double-sided sticky black tape and mounted on the SEM sample platform. Mounted samples were covered by a combination of gold and platinum (60:40) for 10 min with 10 mA plasma current and it was placed in SEM. In FESEM, secondary electron ionization was used to understand the surface morphology of microcapsules (Naz et al., 2020).

Statistical analysis

All experiments were performed in triplicate and the mean values with standard deviations (S.D.) were calculated by SPSS (Statistical Package for the Social Sciences) software (v27, Armok, NY, USA: IBM Corp., 2020).

RESULTS AND DISCUSSIONS

EE influenced by different matrixes

To optimize the composition of emulsion for the microencapsulation of FO, an experimental 3 factorials–3 levels Box–Behnken design was applied with three-independent variables, such as ratio of MD and GA (X1), proportion of MS (X2) and concentration of FO (X3). The values of these independent variables, such as MD/GA, proportion of MS and concentration of FO were 0 (no MD) −1, 0–40% and 10–40%, respectively. The observed response EE was expressed as a function of these independent variables. This experimental design was based on the modelling of the results in the form of a polynomial function. The independent variables were optimized in the model in such a way that the response (EE) reaches the desired maximum value. In Table 2, experimental responses (EE) depending on independent variables, such as X1, X2 and X3 are presented.

Maximum EE (80.43%) was achieved according to the experimental scheme 8 out of all runs. The experimental run 8 was performed with an emulsion having MD/GA 1, concentration of MS 20% and FO 10%. On the other hand, lowest EE (49.87%) was obtained when there was no MD, MS 20% and FO 40% in emulsion (Run 2). It can be justified by the fact that EE can be reduced due to the high amount of FO in emulsion formulation and importance of MD to prepare microcapsules. MD can develop a glassy-type thin film more easily, which improves the adhesive strength and stability of microcapsules. It promotes the retention of oil within the matrix; however, MD has no emulsifying capacity. GA acts as an emulsifier and has a film-forming property because of its low T_g. Therefore, their combined effects offer higher EE. For the optimization of FO encapsulation with the mentioned polymeric carbohydrates and SD, a quadratic polynomial model is suggested with an adjusted R^2 value 0.9797, predicted

Table 2. Experimental responses of the designed combinations of independent variables

Run	X1	X2	X3	EE (%)
1	0	−1	1	50.08
2	−1	0	1	49.87
3	0	0	0	75.66
4	−1	0	−1	65.12
5	0	0	0	75.75
6	1	1	0	69.89
7	0	−1	−1	70.64
8	1	0	−1	80.43
9	−1	1	0	59.72
10	0	1	−1	72.39
11	−1	−1	0	56.77
12	1	−1	0	70.14
13	1	0	1	55.02
14	0	1	1	50.3
15	0	0	0	76.22

R^2 value 0.9583 and a nonsignificant lack of fit (P value >0.05), mentioned with underline in Table 3.

To describe the effects of independent variables on response, ANOVA results are shown in Table 4.

It is shown in Table 4, the F-value of the model is 458.16 and the P-value is less than 0.05. It implies that the model equation has a significant contribution to explain the optimization condition for the encapsulation of FO. The interaction between MS (X2) and amount of FO (X3) has an insignificant effect on EE ($P = 0.2728$); however, the contributions of other independent variables have a significant effect on EE ($P < 0.05$). As an example, the P value is 0.0496 when the contribution MD/GA (X1), and amount of MS (X2) are considered. In other cases, the P value is 0.0004 when the contribution of MD/GA (X1), and amount of FO (X3) are considered. The equation in terms of coded factors obtained from the model to describe the EE is mentioned below:

$$EE\% = 75.88 + 5.50X1 + 0.58X2 - 10.41X3 - 0.8X1X2 - 2.54X1X3 - 4.99X1^2 - 6.75\ X2^2 - 8.27X3^2 \quad (10)$$

The actual equation from the coded equation is mentioned below:

Table 3. Summary of the fitting of experimental results

Source	Sequential P-value	Lack of Fit P-value	Adjusted R^2	Predicted R^2	Comments
Linear	0.0033	0.0017	0.6164	0.5681	
2FI	0.9129	0.0012	0.5044	0.3895	
Quadratic	<0.0001	0.1373	0.9966	0.9822	Suggested
Cubic	0.1373		0.9992		Aliased

Table 4. Statistical results of of the Box-Behnken design

Source	Sum of Squares	Degree of freedom	Mean Square	F-value	*P*-value	Comments
Model	1590.24	9	176.69	458.16	<0.0001	Significant
X1	242.00	1	242.00	627.50	<0.0001	
X2	2.73	1	2.73	7.07	0.0450	
X3	867.57	1	867.57	2249.58	<0.0001	
X1X2	2.56	1	2.56	6.64	0.0496	
X1X3	25.81	1	25.81	66.92	0.0004	
X2X3	0.5852	1	0.5852	1.52	0.2728	
$X1^2$	92.11	1	92.11	238.83	<0.0001	
$X2^2$	168.33	1	168.33	436.49	<0.0001	
$X3^2$	252.65	1	252.65	655.13	<0.0001	
Residual	1.93	5	0.3857			
Lack of Fit	1.75	3	0.5825	6.44	0.1373	Not significant
Pure Error	0.1809	2	0.0904			

$$EE\% = 46.75 + 41.05\frac{MD}{GA} + 0.78MS + 1.34FO - 0.08\frac{MD}{GA}MS - 0.34\frac{MD}{GA}FO - 19.98\left(\frac{MD}{GA}\right)^2 - 0.02\ MS^2 - 0.04FO^2 \quad (11)$$

Normal % probability depending on externally studentized residuals based on theoretical and experimental results are mentioned in Fig. 2(A).

In plot 2(A) it is noted that the residual values (difference between model-derived and experimental values) are within the acceptance limit. Externally studentized residuals for each run are mentioned in Fig. 2(B). It is shown that the residual values are randomly scattered around the zero line; however, they are situated within the limits ±6.25. Predicted values in comparison with the actual values are mentioned in Fig. 2(C). It demonstrates that the proposed model can adequately support experimental results.

Response surfaces of outcome (EE) based on different independent variables, such as X1, X2 and X3 are shown in the subsequent sections. Based on Figs 3–5, a distinct quadratic influence of all factors, such as X1, X2 and X3 on EE is visible. Surface plots represent that EE is influenced by the composition of the matrix and amount of FO in emulsion.

Response surface of outcome (EE) based on the MD/GA (X1) and MS (X2) is represented in Fig. 3.

In Fig. 3, a vault-like shape with a noticeable peak around the midpoint of the studied ranges of independent variables is shown. With a constant oil content, the EE is increased with the increase of X1 and subsequently, it becomes constant. Furthermore, it is noted that the EE increases with the increase of X2 until 20%; eventually, it is decreased. It can be justified by the fact that the combination of MD and GA can offer higher stability of emulsion, improve the stability of the matrix due to high T_g and emulsifying property. However, MS has a high film-forming property, higher amount of MS to prepare matrix is not suitable because it has low T_g, which can reduce the stability of microcapsule and leads to the leakage of encapsulated oil.

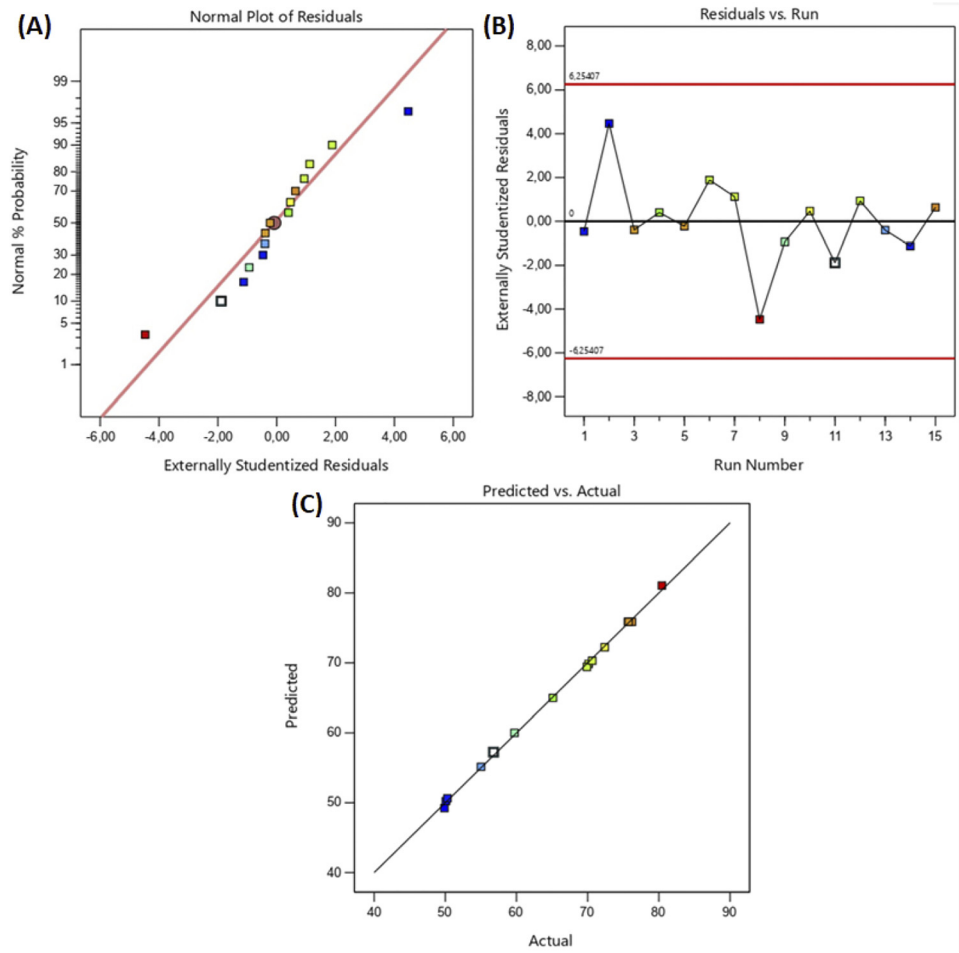

Fig. 2. Plots of experimental and model-derived values. (A) Normal % probability vs. Externally studentized residuals, (B) Externally studentized residuals vs. Run number, and (C) Predicted responses vs. actual values

Response surface of outcome (EE) based on the MD/GA (X1) and FO (X3) is represented in Fig. 4.

Figure 4 shows that at a constant value of MS, EE increases significantly with increasing X1. The justification of the result is mentioned before. On the other hand, EE increases with the increase of X3 upto 24% and subsequently, it is decreased. This can be explained by the fact that higher conctration of oil (more than 24%) is not suitable for encapsulation.

Response surface of outcome (EE) based on MS (X2) and FO (X3) is represented in Fig. 5.

In Fig. 5, it is shown that EE increases with the increase of MS up to 25% and it reduces when the amount of MS is more than 25%. Similarly, it is noted that EE increases with the increase of

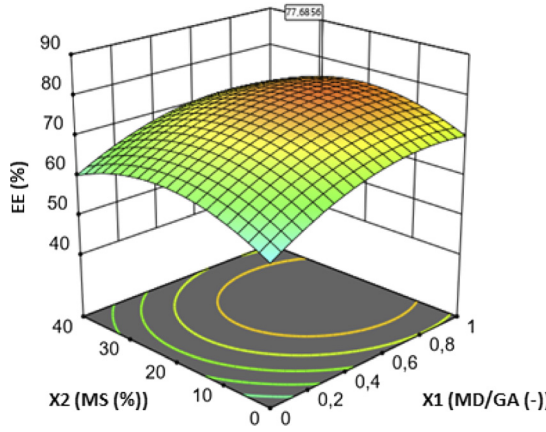

Fig. 3. Response surface plot of EE based on MD/GA (X1) and MS (X2). MD: Maltodextrin, GA: Gum Arabic, MS: Modified starch

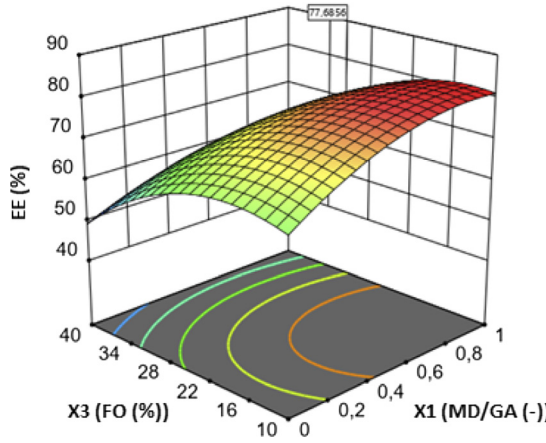

Fig. 4. Response surface plot of EE based on MD/GA (X1) and FO (X3). MD: Maltodextrin, GA: Gum Arabic, FO: Flaxseed oil

oil concentration; however, it reduces when the concentration of oil is more than 25%. The reasons of these results are mentioned earlier.

The optimum composition of emulsion suggested by the software according to the constraints set in Table 5 is MD/GA 0.79, MS 20.23% and FO 24.62%. With this composition, optimum predicted value of EE is 77.68 % with 0.76 desirability.

The optimum composition of emulsion obtained from RSM was further confirmed by the experimental results. Optimum EE is achieved 77.34% after performing experiment. Compared with the predicted value of 77.68%, the verification result is in the range of 95% PI low (76.49) and 95% PI high (78.94). Subsequently, verification was strengthened by the sample *t*-test. Results

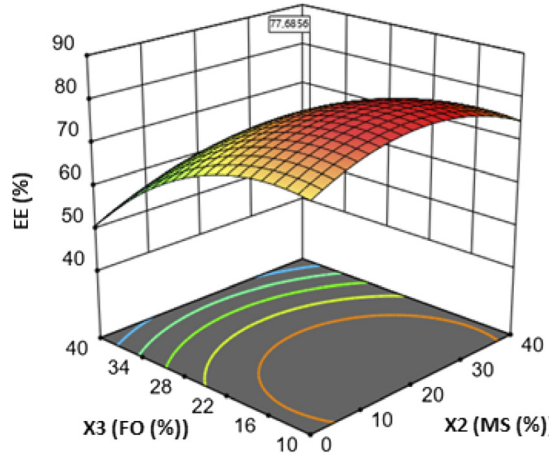

Fig. 5. Response surface plot of EE based on MS (X2) and FO (X3). MS: Modified starch, FO: Flaxseed oil

Table 5. Constraints of independent variables for the optimization of emulsion composition

Name	Goal	Lower limit	Upper limit	Lower weight	Upper weight	Importance
X1	is in range	0	1	1	1	2
X2	is in range	0	40	1	1	2
X3	maximize	10	40	1	1	2
EE (%)	maximize	49.87	80.43	1	1	5

showed that the values were not significantly different ($P > 0.05$). Experimental result was closer to the predicted result. It can be concluded that the RSM by the Box-Behnken model could be used to optimize the composition of emulsion, prepare with MD having DE 19, GA, MS and FO.

Characterization of microcapsule

Microcapsules produced from the optimum emulsion composition by a RSH and SD were characterized. Size of microcapsules and size distribution of microcapsules are represented in Table 6. The values of D_{32} and D_{43} were 18.15 ± 0.08 µm and 120.0 ± 0.43 µm, respectively. Furthermore, it is noted that microcapsules are not monodispersed because the span value is higher than 0.4. It can be possible due to the presence of high concentration of polymeric

Table 6. Emulsion droplet size, microcapsule size and their distributions. Microcapsules were prepared by the optimum emulsion composition and SD

Diameter	Emulsion	Microcapsule
D_{32} (µm)	7.71 ± 0.01	18.15 ± 0.08
D_{43} (µm)	13.62 ± 0.09	120.0 ± 0.43
Span (–)	1.3 ± 0.02	2.89 ± 0.95

carbohydrates in the emulsion. The value of D_{32} and D_{43} of emulsion droplets were 7.71 ± 0.01 μm and 13.62 ± 0.09 μm, respectively. Similar to the size distribution of microcapsules, emulsion was not monodispersed. Stability of emulsion also influences the EE and characteristics of microcapsules. Percentage separation of oil in emulsion after 24 h was 17. The value of zeta potential was −29.5 ± 0.5 mV which signifies a weaker electrostatic repulsion force between the droplets. Weaker electrostatic repulsion force increases coalescence between droplets and consequently makes agglomeration.

The morphology of microcapsules is presented in Fig. 6. It is shown that microcapsules produced from the emulsion obtained at optimum composition and SD are sphere-shaped and have a smooth surface without visible dent, pores and cracks; however, they are agglomerated.

The spherical-shaped microcapsules are developed by the melting of polymeric carbohydrates in the emulsion because of a high inlet temperature and the nozzle of the spray dryer. Agglomeration of microcapsules may be influenced by the characteristics of polymeric carbohydrates in the emulsion. In the emulsion MD having DE 19 was hydrophilic due to presence of lower molecular weight of carbohydrates. Furthermore, GA and MS in emulsion were also hydrophilic. Therefore, microcapsules produced from the emulsion, having MD, GA and MS have high water activity.

Moisture content, wettability, solubility, BD, TD, *CI* and HR are crucial factors to understand the characteristics of microcapsules. Higher moisture content generally indicates higher water activity and better solubility. Moisture content of microcapsules is also related with wettability and solubility. Wettability of microcapsules represents their shrinking property within water. Solubility of microcapsules represents their dissolving potentiality in aqueous medium. The BD microcapsules refers to the ratio of the mass of untapped microcapsules and its volume including the contribution of the void. On the other hand, TD refers to the ratio of the mass of tapped microcapsules and its volume. *CI* and HR are directly correlated with BD and TD. They are important to understand the flowability and cohesiveness of microcapsules. Microcapsules produced by optimum composition of emulsion and SD had good flow properties. The results of the characteristics of microcapsules are mentioned in Table 7.

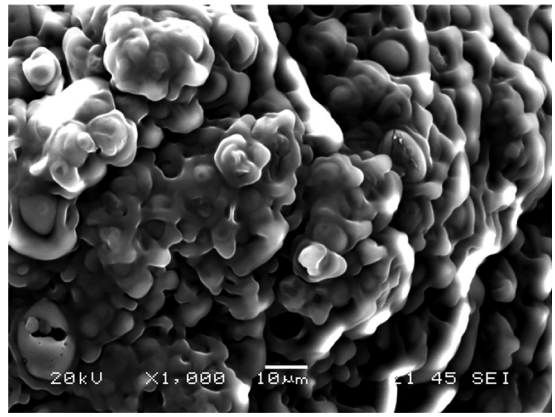

Fig. 6. Scanning electron microscopy images of FO microcapsule prepared by the dehydration of emulsion having MD, GA, MS and FO

Table 7. Moisture content, wettability, solubility, BD, TD, *CI* and HR of microcapsules were prepared by the optimum emulsion composition and SD

Moisture content (%)	1.6 ± 0.13
Wettability (s)	192 ± 5.5
Solubility (%)	75.49 ± 1.3
BD (g mL^{-1})	0.31 ± 0.025
TD (g mL^{-1})	0.36 ± 0.01
CI (%)	13.88 ± 0.01
HR	1.16 ± 0.01

CONCLUSION

FO is enriched with PUFA (α-linolenic acid) and wide ranges of antioxidants (tocopherols, β-carotene, phytosterols, flavonoids and polyphenols). In the present investigation, microencapsulation of FO has taken into consideration. Microencapsulation of FO was prepared by the dehydration of aqueous emulsions of FO with polymeric carbohydrates, such as MD with DE 19, GA and MS. Emulsion was prepared by a RSH and subsequently drying of emulsion by SD. Composition of emulsion was successfully optimized by a 3 factorials–3 levels Box–Behnken experimental design to obtain maximum EE of microcapsules. Microcapsules having high EE were spherical with smooth surface, wide ranges of size distribution and agglomerated due to the presence of GA, MS and MD with 19 in matrix.

In this investigation, only polymeric carbohydrates were considered for the preparation of emulsion and the microencapsulation of FO. In future, researches might be performed with other wall materials, such as MD with different DE, animal- and plant-based proteins. Future researches will be considered to realize the drying methods and associated drying parameters on EE. Furthermore, researches might be performed to understand the quality of encapsulated FO during storage, and the release of PUFA and phytochemicals during digestion.

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