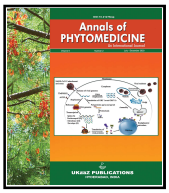
Annals of Phytomedicine 9(2): 277-282, 2020

DOI: http://dx.doi.org/10.21276/ap.2020.9.2.27

Annals of Phytomedicine: An International Journal

http://www.ukaazpublications.com/publications/index.php

Print ISSN : 2278-9839 Online ISSN : 2393-9885



Original article: Open access

Optimization of coating material for encapsulation of flax seed oil containing omega-3 fatty acids

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Article Info

Article history
Received 5 July 2020
Revised 22 August 2020
Accepted 25 August 2020
Published online 30 December 2020

Keywords
Encapsulation
Maltodextrin
Sodium alginate
Spray dryer

Abstract

This study is aimed to evaluate the potential of different wall materials for microencapsulation of flaxseed oil containing omega-3 fatty acids by spray drying. Maltodextrin (MD) was mixed with Sodium Caseinate (SC), Sodium Alginate (SA) and Gum Acacia (GA), at three different ratios including 5:2, 5:4 and 5:6. The feed emulsions used for particle production were characterized for stability, particle size, zeta potential analysis and encapsulated powder were characterized for encapsulation yield, encapsulation efficiency and scanning electron microscope analysis. Best encapsulation efficiency was obtained for MD: SC which was followed by the MD: SA, while the lowest encapsulation efficiency was obtained for MD: GA, which also showed poorest emulsion stability.

1. Introduction

Over the past decade, the demand for wholesome and nutritious food has been increased remarkably (Rajeshwari *et al.,* 2014). Flaxseed oil is a potent source of omega-3 fatty acid and gaining more attention towards nutritive food with several potential advantages to the food industry. Microencapsulation of omega-3 fatty acid rich flaxseed oil protects the core material by limiting its nutrient loss and converts it into a dispersed powder form (Desai and Park, 2005). The process of microencapsulation implies surface coating of active droplets of core material within coating material comprised of polysaccharides, lipids, and proteins to form microcapsules of micron to millimeter size range (Tyagi *et al*., 2011). Amongst the various microencapsulation techniques such as spray drying, freeze-drying, coacervation, and extrusion, generally spray drying technique is chosen because of its cheaper production cost (Desai and Park, 2005). The selection of coating material impersonates an influential part in the microencapsulation, as it shows a high impact on the stability of microcapsules, encapsulation efficiency, and the degree of protection to the core material. To achieve high encapsulation efficiency a combination of polysaccharides and proteins must be used for the production of microcapsules, as they both together shows synergistic effect, in which polysaccharides act as a matrix forming material and proteins act as emulsifying and film forming material (Gharsallaoui *et al*., 2010; Mishra *et al*., 2020; Nesterenko *et al*., 2013). Polysaccharides used are maltodextrins, starches, sodium alginate, pectin, and chitosan whereas both animal protein (*e.g.*, whey, gelatin, and casein) and plant protein (*e.g.*, soy and pea) are used as a coating material. Omega fatty acid (omega-3, -6, and -9) plays an essential role in prevention of several human diseases such as cardiovascular diseases, immune response disorders and maintenance of mental health (Shibasaki *et al*., 1999). Flaxseed oil comprising omega-3 fatty acid is unsaturated because of which it is chemically unstable and consequently becomes susceptible to oxidative deterioration leading to the production of free radicals, which thus affects the shelf-life, sensory attributes, and overall acceptability of food products (Velasco *et al*., 2000). Hence, microencapsulation of flaxseed oil provides an efficient way to minimize the susceptibility of flaxseed oil to various environmental parameters such as oxygen, light, temperature, and moisture, and thus enhances the product shelf-life and mask the undesirable taste and odor (Calvo *et al*., 2012).

The research was conducted with the aim to encapsulate the omega-3 fatty acid rich flaxseed oil by spray drying and to evaluate the potential of a different combination of wall materials (including maltodextrin (MD), sodium caseinate (SC), sodium alginate (SA) and gum acacia (GA) to produce stable microcapsules.

2. Materials and Methods

2.1 Materials

Flaxseed oil (Ceyon Healthcare India Private Limited, Lucknow, India) was used as active material. The edible grade wall materials used were: Maltodextrin, Sodium Caseinate, Gum Acacia and Sodium Alginate (Himedia, India).

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2.2 Emulsion preparation

Encapsulation of omega-3 fatty acid rich flaxseed oil was done by using three combinations of maltodextrin with three wall materials sodium caseinate, sodium alginate, and gum acacia with total nine treatments (Table 1). The wall material firstly added to distilled water at 25°C then the mixture was agitated until completely dissolved. The total solid concentration (wall material + oil) was set to 30%. Flaxseed oil was then added to the wall material solution at a concentration of 20% with respect to total solids (Ahn *et al*., 2008; Jafari *et al*., 2008; Charve and Reineccius, 2009). Emulsions were formed using an IKA T25 Digital Ultra-Turrax homogenizer (IKA, Germany) operating at 18,000 rpm for 5 min.

Table 1: Different combinations of wall material for encapsulation of flex seed oil

Treatments	Combination of wall material	Ratio of wall material	Flax seed oil (g)
T_1	Maltodextrin + Sodium caseinate	5:2	8
T_2	Maltodextrin + Sodium caseinate	5:4	8
T_3	Maltodextrin + Sodium caseinate	5:6	8
T_4	Maltodextrin + Sodium alginate	5:2	8
T_5	Maltodextrin + Sodium alginate	5:4	8
T_6	Maltodextrin + Sodium alginate	5:6	8
T_7	Maltodextrin + Gum acacia	5:2	8
T_8	Maltodextrin + Gum acacia	5:4	8
T_9	Maltodextrin + Gum acacia	5:6	8

2.3 Emulsion characterization

2.3.1 Emulsion zeta potential and droplet size

The zeta potential and droplet size of the prepared emulsions were measured using Zeta nanosizer (Malvern-Aimil Instruments Private Limited, New Delhi, India) with a dynamic light scattering system. The emulsions were initially dispersed in distilled water, and the droplet size distribution was then measured until three constant readings were obtained.

2.4 Microencapsulation by spray drying

After preparation of emulsion, the emulsion was subjected to spray drying using a laboratory scale spray dryer Lab Plant SD-48 (Huddersfield, England), with a nozzle atomization system with 0.5 mm diameter nozzle. The emulsion was fed through a peristaltic pump where the pump rotation speed regulates the flow rate of feed solution to 16 m/s. The pressure of the compressed air for concurrent flow of spray dryer was adjusted to 2 kg/cm^2. Inlet and outlet air temperature were 160 °C and 70 °C, respectively with aspirator speed of 1200 rpm.

2.5 Encapsulated powders analysis

2.5.1 Encapsulation efficiency

Encapsulation efficiency (EE) was determined according to the method reported by Bae and Lee (2008). Fifteen milliliters of hexane were added to 1.5 g of microcapsules in a glass jar with a lid, agitated for 2 min, for the extraction of free oil, at room temperature. The solvent mixture was then filtered through a Whatman filter paper number 1 and the microcapsules collected was rinsed three times with 20 ml of hexane. Then, the solvent was left to desiccate, at room temperature and 60 °C, until constant weight. The non-encapsulated oil (surface oil) was determined by the mass difference among the initial clean flask and that containing the extracted oil residue (Jafari *et al*., 2008). Total oil was supposed to be equivalent to the initial oil which was presumed as flaxseed oil. Encapsulation efficiency (EE) was calculated as given below:

$$EE = \frac{TO - SO}{TO} \times 100$$

Where TO and SO is the total and surface oil content, respectively.

2.5.2 Encapsulation yield

The encapsulation yield of microcapsules, estimated as the ratio of the weight of microcapsules and the weight of total solids (including wall and core material) expressed as a percentage of yield (Che Man *et al*., 1999).

2.5.3 Morphology and size

Microcapsules were observed in a Scanning Electron Detector microscope (LEO Electron Microscopy, Oxford, England) working at 15 kV and electron beam current of 100 pA. The samples were fixed directly on door-metallic specimens (stubs) of 12 mm diameter and then subjected to metallization (sputtering) with a thin layer of gold/palladium in a Sputter Coater SC7620 polaron (VG Microtech, England) at a coverage rate of 0.51 Å/s for 180 s, with a current of 3.5 mA, 1 V and 2 102 Pa. After metallization, the samples were observed with magnifications of 4000, 5000 and 10,000. Image acquisition was performed by the LEO software, version 3.01.

2.6 Statistical analysis

The statistical difference between droplet size of emulsion and encapsulation efficiency and encapsulation yield of microcapsules for all the nine different combinations of wall material was determined using a two-way analysis of variance (ANOVA).

3. Results

3.1 Emulsion zeta potential and droplet size

The zeta potential analysis of emulsions (Table 2) shows that the values for maltodextrin and sodium caseinate, maltodextrin and sodium alginate, and maltodextrin and gum acacia combinations ranges from -10.7 mV to -29.9 mV, -18.3 mV to -42.7 mV, and -38.3 mV to -50.7 mV, respectively (Figure 1, Table 2). The particle charge for all the emulsions were kinetically stable, except for those produced with a combination of maltodextrin and gum acacia. The combination of maltodextrin and sodium caseinate was found to be more stable in comparison to other wall material combination. The combination of maltodextrin and sodium alginate shows that with increase or decrease in the concentration of sodium alginate results in the unstable emulsion.

Table 2: Zeta potential of (mV) various combinations of emulsions

Material	5:2	5:4	5:6	Stable/unstable
M + SC	-29.9	-11.3	-10.7	Stable
M + SA	-41.3	-18.3	-42.7	Unstable
M + GA	-507	-38.3	-60.4	Unstable

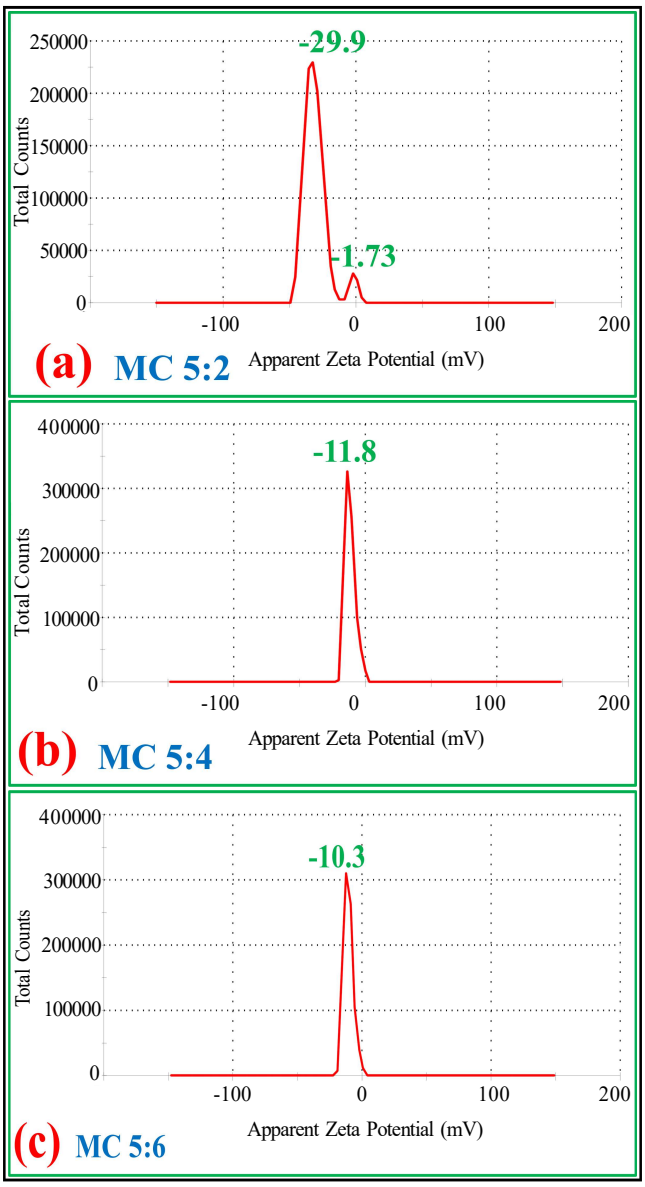

Figure 1: Zeta potential of the particles in the emulsion.

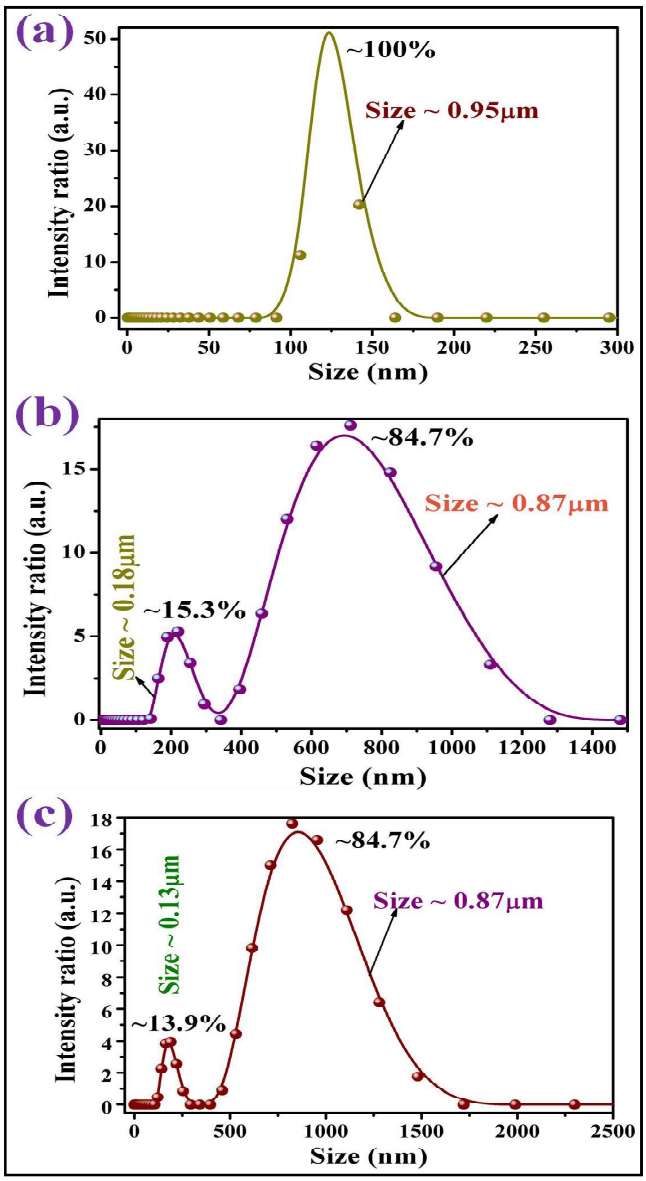

Figure 2: Particle size distribution of particles in different ratio (a) 5:2, (b) 5:4, (c) 5:6.

The droplet size distribution of the emulsions prepared with different wall material was shown in Table 3 and Figure 2. The emulsions prepared with a combination of maltodextrin and gum acacia (5.44 µm) had the largest droplet size diameter, and those prepared with a combination of maltodextrin and sodium caseinate (0.95 µm) had the smallest.

Table 3: Emulsion droplet size of various combinations of wall materials

S.N.	Experimental Trials	Droplet size
1.	T_1	0.95µm
2.	T_2	0.87 µm
3.	T_3	0.87 µm
4.	T_4	0.35 µm
5.	T_5	1.76 µm, 5.36 µm
6.	T_6	0.59 µm, 5.20 µm
7.	T_7	0.33 µm, 2.11 µm
8.	T_8	2.88 µm
9.	T_9	1.03 µm

3.2 Encapsulation Efficiency

Encapsulation efficiency (EE) of all nine treatments was revealed in the Figure 3 and Table 4. The encapsulation efficiency of experimental trial T_2 (89.97%) was found to be higher accompanied by that of trial T_5 (86.03%). The combination of maltodextrin and gum acacia shows reduced encapsulation efficiency amongst all the other combination of the wall material. The encapsulation efficiency of microcapsules depicts that, in the maltodextrin and sodium alginate combination, the encapsulation efficiency increases when the amount of maltodextrin and sodium alginate are approximately equal.

3.3 Encapsulation Yield

The encapsulation yield of all the nine flaxseed microcapsules explains that the combination of maltodextrin and sodium caseinate of ratio 5:2 and 5:4 proffers higher encapsulation yield of 28% and

24.21 %, respectively (Figure 3; Table 4). In maltodextrin and sodium alginate combination of ratio 5:4 confers the highest encapsulation yield of 23.20% in comparison to other ratios of the same combination. The encapsulation yield of maltodextrin and gum acacia combination was seen to be non-significant to other combination of wall material due to its larger particle size (Figure 3).

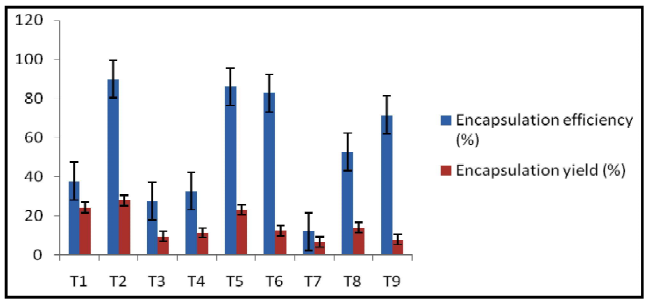

Figure 3: Encapsulation efficiency and encapsulation yield of flaxseed oil microcapsules Values are mean ± SD (n=3).

Table 4: Encapsulation efficiency in different treatments

S.No.	Treatments	Encapsulation efficiency (%)	Encapsulation yield (%)
1	T_1	37.68	24.21
2	T_2	89.97	28.00
3	T_3	27.32	9.48
4	T_4	32.71	11.31
5	T_5	86.03	23.20
6	T_6	82.76	12.48
7	T_7	12.09	6.552
8	T_8	52.82	13.85
9	T_9	71.62	7.80

3.4 Morphology and size

The external morphology and crystalline nature of the flaxseed microcapsules were revealed using a scanning electron microscope (SEM). Figure 4 explains the morphology and size of flaxseed microcapsules. The morphology of flaxseed microcapsules revealed that the microcapsules were spherical with no surface dents and a size range of 3.57-11.56 µm. The microcapsules prepared from combination ratio of 5:6 of maltodextrin and sodium caseinate and maltodextrin and sodium alginate show that the capsules are hollow these were similar to microcapsules prepared from all the ratio of maltodextrin and gum acacia combination which indicates that the flaxseed oil is not encapsulated. The microcapsule of combination maltodextrin and sodium caseinate of ratio 5:4 shows uniform covering resulting in no crack on the surface of the microcapsules which thus limits the air incursion (Aghbashlo *et al*., 2012). The present study agrees with the study reported by Kolanowski *et al*. (2006) resulting in spherical microcapsules of fish oil. Other combinations show depression in the surface of microcapsules which is due to the breakdown of droplets by particle shrinkage during the process of drying.

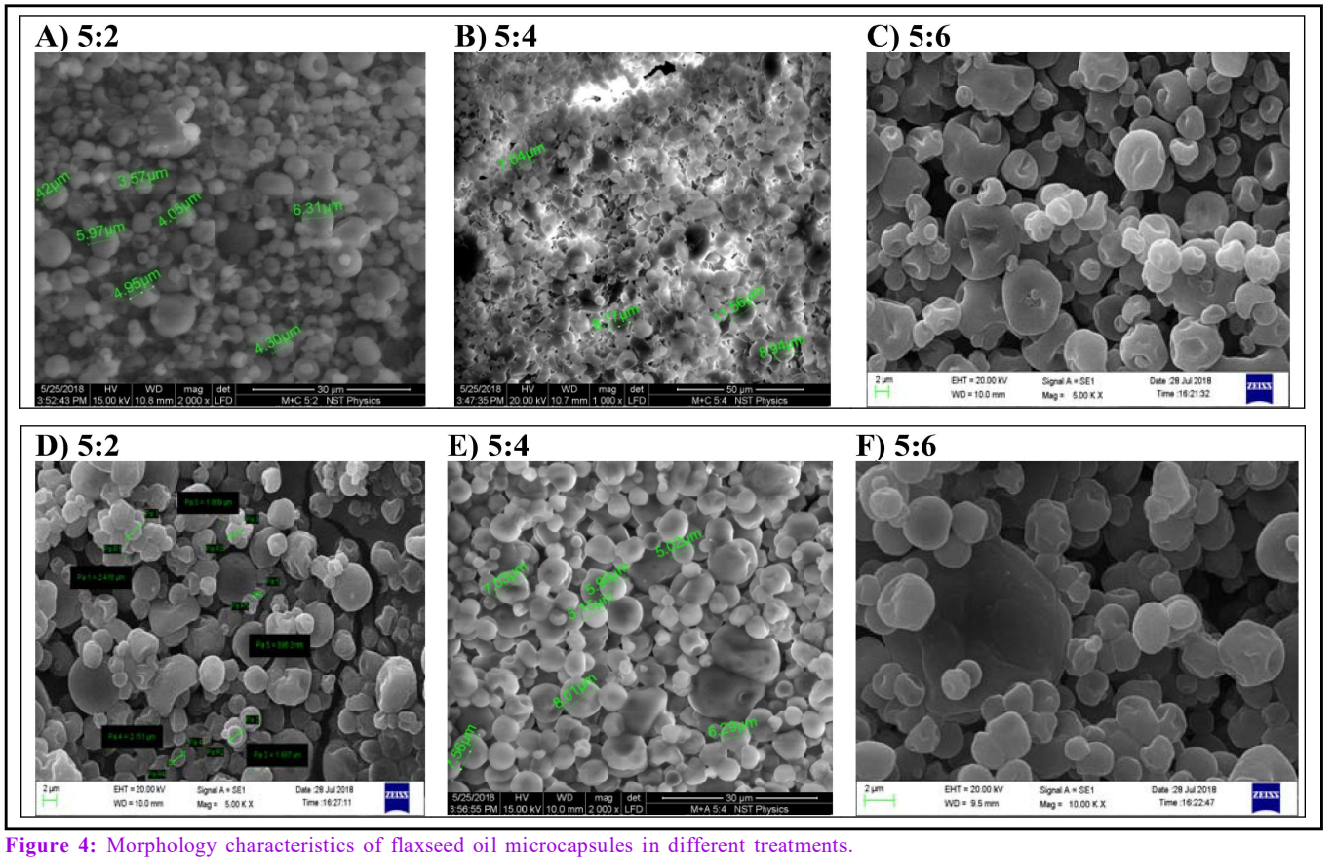

Figure 4: Morphology characteristics of flaxseed oil microcapsules in different treatments.
A-C: Maltodextrin + Sodium Casinate
D-F: Maltodextrin + Sodium Alginate

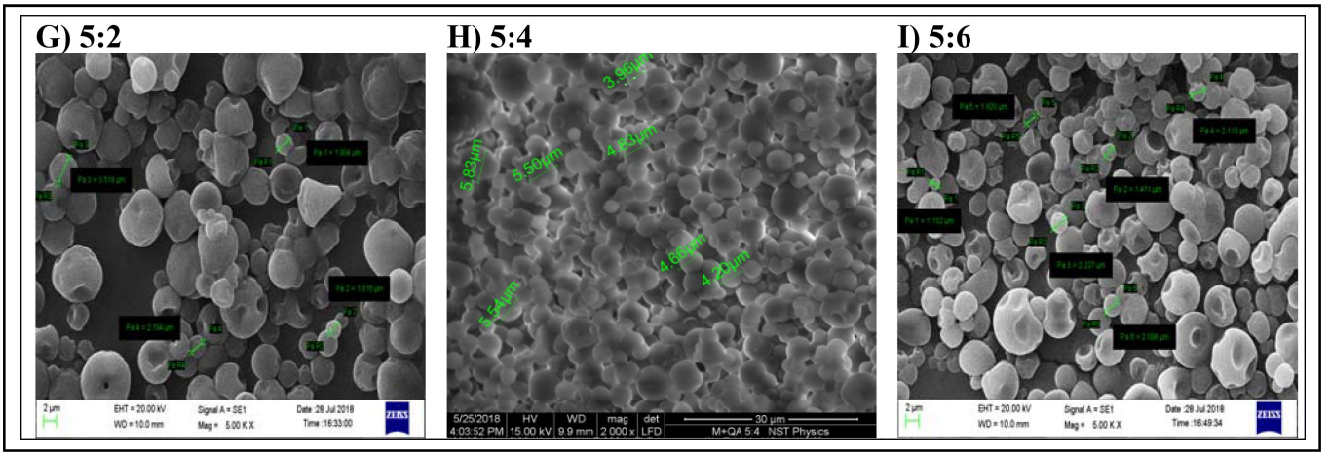

Figure 4: Morphology characteristics of flaxseed oil microcapsules in different treatments.
G-I: Maltodextrin + Gum Acacia

4. Discussion

Oils are easily available and have been used from ancient time for medicinal purpose (Fehim *et al*., 2017). Zeta potential of emulsion confers data regarding the emulsion stability by establishing the efficient electric charge on the surface of the emulsion. The zeta potential of emulsion provides particle charge (-10 to +10 mV and -30 to +30 mV) which act as stability determining factor. With the increase in the electrostatic repulsion between the particles, the stability of emulsion increased. The instability of emulsion produced with gum acacia was due to the unfolding of protein molecules at the droplet surface, which heightens the protein-protein interaction, leading to flocculation during emulsification. The unfolding of protein units of the oil-water interface may lead to alters the secondary and tertiary structures and, consequently, exposure of their residues, which would be associated (-S-S linkages or disulfide linkages) within the primary globular structure, resulting in the formation of intermolecular interaction at the oil-water interface and flocculation (Tonon *et al*., 2012).

The encapsulation efficiency depicts the amount of oil that has been retained within the wall material, comparative to the surface oil present on microcapsules (Klinkesorn *et al*., 2006). Encapsulation efficiency is linked with emulsion stability as an increase in encapsulation efficiency results in increased stability (Ortega-Rivas *et al*., 2006). This statement agrees with the present research, both the trial T_2 and T_5 had high emulsion stability resulting in increased encapsulation efficiency. The encapsulation efficiency of the present research was comparable as described by Chan *et al*. (2011). Statistical data shows that the selection of wall material and oil concentration was a highly significant factor, for the encapsulation efficiency, though, the interaction of combination prepared from gum acacia (T_7, T_8, T_9) was not significant. The emulsion stability correlates with the encapsulation efficiency as the poor stability for combination maltodextrin and gum acacia resulted in lower encapsulation efficiency. The large droplet size indicates high surface oil content, which thus results in reduced encapsulation efficiency (Soottitantawat *et al*., 2003). The smaller emulsion droplet size results in increased stability of emulsion with adequate retention of active material (Carneiro *et al*., 2013; Jafari *et al*., 2008).

5. Conclusion

Flaxseed oil is known to be the rich vegetarian source of omega 3 fatty acid and have nutraceuticals used in prevention of cardiovascular diseases. The parameters like encapsulation efficiency, encapsulation yield and the emulsion stability was analyzed in the screening of wall material for the microencapsulation of flaxseed oil. The morphological characteristics reveal that the treatment T_2 covers the active material completely. The microencapsulation of flaxseed oil enhances its solubility resulting easy release of flaxseed oil. Moreover, microencapsulation influences the oxidative stability of the oil. Further research needs to carry out on fortification of various food products with flaxseed oil microcapsules, as this will have a positive impact on the overall health of the consumer.

Acknowledgements

Authors acknowledge Department of Biotechnology, Government of India for their financial support.

Conflict of interest

The authors declare that there are no conflicts of interest in the course of conducting the research. All the authors had final decision regarding the manuscript and decision to submit the findings for publication.

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Citation **Dinesh Chandra Rai, Arvind and Vinod Kumar Paswan (2020). Optimization of coating material for encapsulation of flax seed oil containing omega-3 fatty acids. Ann. Phytomed., 9(2):277-282. http://dx.doi.org/10.21276/ap.2020.9.2.27**