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Effect of nano-encapsulation of flaxseed oil on the stability, characterization and incorporation on the quality of eggless cake

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ABSTRACT

The objective of this study was to investigate baking stability of nano-encapsulated spray-dried flax seed oil. Gum Arabic (GA), soy-protein isolate (SPI), sodium caseinate and sodium alginate were wall materials (20%), soy-lecithin as emulsifier and 6% flax seed oil. Powders were characterized for morphology, density, encapsulation efficiency (EE) and peroxide value. Four powders tested were below 100 nm in size with stable zeta potential values. Wall material containing GA (4%) and SPI (16%) showed highest ω−3 fatty acids (30%), encapsulation efficiency (72%) with lowest moisture (4%) and peroxide value (1.1 meq/kg).

The optimized nano-particles were incorporated into eggless cake with chia (T2) to investigate their effects on physical, rheological, textural and nutritional quality and compared with control (C) made with eggs and eggless cake with chia and flax-seed powder (T1). There was no significant difference between batter of C and T2 cakes in physical and textural attributes. T2 showed higher and stable ω-3 fatty acids (25.4) after baking. ω-6 FA levels were reduced with increased antioxidant capacities in T2. Healthy ω-3 rich cake of acceptable quality characteristics meeting ω-3 requirements of vegetarians was developed using nano-encapsulated flax seed oil.

1. Introduction

Flax seed is good vegetarian source of α-linolenic (ALA) fatty acids (El-Beltagi, Salama, & El-Hariri, 2007). Its bioavailability in flax seed oil is greater than milled seed, followed by whole seed (Alejandro et al., 2008). Flaxseeds have been incorporated into bread (Aguiar, Boroski, Monteiro, Souza, & Visentainer, 2011), biscuits (Heba, Agamy, & Ismail, 2018) and cakes (Bashir, Masud, & Latif, 2006). Flaxseed is naturally a rich source of essential omega-3 fatty acid, α-linolenic acid (ALA), which exhibits nearly 57% of its entire fatty acid profile. Oxidation of omega-3 fatty acids during processing and storage results in reduced shelf stability of food products, limited health potentials, decreased nutritional value and sensory quality (Gökmen, Magol, Lumaga, Fogliano-Vincezo, & Kaplun, 2011).

Encapsulation and micro-fluidization both involve the incorporation of food ingredients, enzymes, cells or other materials in small capsules. Applications for encapsulation however has increased in the food industry since the encapsulated materials can be protected from moisture, heat or other extreme conditions, thus enhancing stability and maintaining viability. Encapsulation of edible oils by powder particles, comprising carbohydrate or proteins is a scientific process followed to protect polyunsaturated oils from oxidation resultantly preserving flavors, aroma, and transport bioactive oils into easily managed dried solids for food supplementation (Mercier et al., 2014).

Encapsulation can be attained using nanotechnology, effectively protecting bioactive compounds against oxidation (Drozłowska et al., 2021) and thermal degradation (Wang, & Wan, 2009). Nano-encapsulation dramatically increases surface area to volume ratio (Kouassi et al., 2012). Gökmen et al. (2011) reported that nano-encapsulated flax seed oil reduces the risk of acrylamide formation which happens as the temperature of the crust approaches the oven temperature and the moisture content decreases to a critical level.

Common wall materials for encapsulation are Gum Arabic (GA), modified starches, proteins like caseinate, whey concentrate and isolate and soy-protein isolate (SPI), and maltodextrin (MD). SPI in combination with starch is the most effective encapsulation material during spray-drying (Patel, Khan, & Kar, 2016).

Two different drying methods viz freeze-drying and spray-drying are commonly applied in nano-emulsion systems to obtained powder encapsulates. Spray-drying is one of the most commonly used techniques and is a processing technique to shield omega-3 fatty acids from oxidative damage (Tatar, Tunç, Dervisoglu, Cekmecelioglu, & Kahyaoglu, 2014). The spray-drying process has shown to have better encapsulation efficiency and in-vitro bio-accessibility with a lower cost as compared to freeze drying (El-Messery, Altuntas, Altin & Özçeli, 2020). Spray drying

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Table 1a
Quality of nano-encapsulated powders.

Wall materials	Moisture %	pH	Bulk density (g/cm^3)	Tapped bulk density (g/cm^3)	Diameter of emulsion droplet (Dnm)	Zeta potential (mV)	Encapsulation Efficiency (%)	Dispersibility (s)	Wettability (s)	Peroxide value (meq/kg)
P1	5.3^a ± 0.40	5.70^b ± 0.22	0.35^a ± 0.01	0.51^a ± 0.02	70.9^c ± 18.2	- 40.48^b ± 0.05	57.4^b ± 5.87	38.4^b ± 0.83	32.16^c ± 0.94	2.16^b ± 0.31
P2	3.7^c ± 0.14	5.60^b ± 0.36	0.22^c ± 0.01	0.38^b ± 0.01	84.0^b ± 23.4	-32.06^c ± 0.03	71.6^a ± 3.09	38.2^b ± 0.86	34.73^b ± 2.59	1.14^c ± 0.15
P3	3.9^c ± 0.88	5.10^c ± 0.43	0.31^b ± 0.01	0.38^b ± 0.01	87.8^a ± 13.9	-40.36^b ± 0.07	70.6^a ± 14.13	39.3^a ± 0.71	35.30^b ± 2.98	2.57^b ± 0.17
P4	4.3^b ± 1.12	6.02^a ± 0.17	0.35^a ± 0.01	0.50^a ± 0.00	86.5^{ab} ± 23.1	- 47.57^a ± 0.06	59.9^b ± 5.95	40.4^a ± 2.90	39.13^a ± 2.57	4.62^a ± 0.23

P1- 4 % Sodium Alginate +16 % Soy Protein Isolate; P2- 4 % Gum Arabic +16 % Soy Protein Isolate; P3- 4 % Gum Arabic + 16 % Sodium Caseinate; P4- 4 % Sodium Alginate +16 % Sodium Caseinate
Values are expressed as mean ± standard deviation (n =9). Means having different letters as superscripts with in the same column differ significantly (n=9) at $p<0.05$

below an inlet temperature of 160 ˚C and outlet temperature below 80 ˚C has been recommended for maximum ALA retention in encapsulated flax seed oil (Shahid et al., 2020)

The objective of this study was to evaluate wall material combinations for nano-encapsulation of flaxseed oil by spray-drying. The optimized nanoparticles were incorporated into eggless cake made with wheat flour and chia and both the batter and cake were tested for physical, color, rheological, textural, nutritional and organoleptic properties and compared with control cake (C) made using wheat flour and eggs and eggless cake made using wheat flour, chia and flax seed powder (T1) to investigate the effect of nano-encapsulated flax seed oil on quality characteristics of cake.

2. Materials and methods

2.1. Preparation of Nano encapsulated flaxseed oil

2.1.1. Materials

Organic cold-pressed flaxseed oil (Arya brand) was purchased from the local market of Bhopal. AG (density = 1.4 g/cm^3), soy lecithin (30%), ethanol, n-hexane, 2-propanol were purchased from Himedia Laboratories (Mumbai, India). SPI (Asitis brand) was purchased from Medigen labs Bangalore India. Deionized water was used for the preparation of solutions. All other chemicals used were analytical grade and obtained from Sigma Aldrich Co, India.

2.1.2. Preparation of nano-emulsion

The composition of cell wall materials for nano-encapsulation is given in Table 1. Pre-experimentation was carried out regarding different wall materials, their amounts in terms of encapsulation efficiency. The cell-wall material was stirred with a magnetic stirrer at 500 rpm and 60 ˚C for 1 h. 500 ml of de-ionized water was added to the mixture and kept for 24 h at 4 ˚C for maximum hydration. Soy lecithin (3% w/w) and flaxseed oil (6% w/w) were slowly added and the mixture was immediately stirred for 30 min at 500 rpm using a magnetic stirrer (Remi 1 MLH, Thane, India) and sonicated (Sonicator Chiller -SM100CHUS, Samarth Electronics, Thane, India) at 24 kHz for 120 s to produce nano-emulsions.

2.1.3. Preparation of Nanoparticles

The prepared nano-emulsions were converted to an encapsulated powder by spray-drying (Techno search Instrument SPD-111 Thane, India). Inlet air temperature was 150 ˚C for moisture content of powder below 5%. The outlet temperature was stabilized at 80- 85 ˚C by controlling the flow rate at 4 mL/min. The mixtures were sprayed into the drying chamber with an atomizer nozzle and the product dried almost instantaneously. Spray-dried powders were then transferred to sterile bottles with screw-on lids and stored at room temperature (RT).

Table 1b
Fatty acid profile of nano-encapsulated powders.

Wall materials	MUFA %	PUFA %	Omega-3 %	Omega 6 %
P1	23.73 ± 0.18	55.84^c ± 0.32	21.1^c ± 0.21	34.79^{ab} ± 0.11
P2	22.28 ± 0.95	61.44^a ± 1.95	29.7^a ± 1.70	31.74^c ± 0.25
P3	23.55 ± 0.76	56.00^{bc} ± 1.22	25.4^b ± 1.10	32.57^c ± 0.12
P4	23.22 ± 0.62	57.46^b ± 1.47	21.8^c ± 1.05	35.69^a ± 0.42

P1- 4 % Sodium Alginate +16 % Soy Protein Isolate; P2- 4 % Gum Arabic +16 % Soy Protein Isolate; P3- 4 % Gum Arabic + 16 % Sodium Caseinate; P4- 4 % Sodium Alginate +16 % Sodium Caseinate
Values are expressed as mean ± standard deviation (n =9). Means having different letters as superscripts with in the same column differ significantly (n=9) at $p<0.001$

2.1.4. Physical properties of nano-powders

The pH was measured using AOAC (2019) method with a pH meter (Metrohm 827, Switzerland). The moisture content was determined gravimetrically by drying in an oven at 70 ˚C for 24 h (AOAC, 2019). Bulk density (ρb) was determined by dividing the mass by the volume occupied in the cylinder. For tapped density (ρt), the cylinder was tapped vigorously by hand until no further change in volume occurred (Nakarin, Suphantharika, Jamnong, 2008). To calculate encapsulation efficiency (EE) the surface oil (Klinkesorn, Sophanodora, Chinachoti, Decker, & McClements, 2006) and total oil content (Jimenez, García, & Beristain, 2004) of the nano-particles were analyzed. The percent encapsulation efficiency (EE) was calculated by:

$$EE\% = \frac{[Total\ oil - Surface\ oil]\ x100}{Total\ oil} \quad (1)$$

The wettability of powder was determined by method of Yaakub, Zainil, & Xiang (2019) and dispersibility (D20 ˚C and D40 ˚C) was determined by method of Koç, Sakin-YIlmazer, Kaymak-Ertekin, & Balk (2014).

Oxidative stability of the nano-encapsulated flaxseed oil was evaluated based on the peroxide value (meqO_2/kg) and was obtained by the iodometric method based (BS EN ISO 3960:2010) (Domian, Brynda-Kopytowska, & Marzec, 2017).

Table 2
Composition of omega 3 enriched cakes.

Ingredients	C	T1	T2
Wheat flour (g)	75	70	74
Flaxseed meal (g)	-	5	0
Nano encapsulated flax seed oil (g)			1.5
Whole eggs (g)	30	-	-
Chia Seed (g)	-	10	10
Cocoa powder (g)	10	10	10
Castor sugar (g)	75	75	75
Vegetable oil (ml)	25	25	25
Baking powder (g)	5	5	5
Baking soda (g)	3	3	3
Water (ml)	35	60	60

C- Control cake, T1- Chia and Flaxseed Cake, T2-Nanoencapsulated flaxseed oil cake

2.1.5. Particle size of nano-particles

The particle size distribution was determined by dynamic light scattering (Beckman Coultar) to measure the average diameter of emulsion droplets with non-invasive backscatter (NIBS®) technology, which allowed detection of droplets in the range of 0.6-6000 nm. Particle-size distribution was determined using the DTS (nano) software (Version 5.0).

2.1.6. Zeta potential of nano-particles

The zeta potential was determined at 25 °C and 149 W in a Zeta sizer Nano ZS device (UK). The calculated electrophoretic mobility was converted into zeta potential values using the Smoluchowski model (Romică, & Şolea, 2017).

2.2. Preparation of cake

2.2.1. Materials for cake

Whole wheat flour (*Triticum aestivum*), chia seeds (*Salvia hispanica*), flax seeds (*Linum usitatissimum*), baking powder (Weikfield Foods Pvt. Ltd., India), baking soda, castor- sugar, salt, vegetable oil (Adani Wilmar Ltd., India), cocoa (Hersheys, India) and vanilla essence (Gujarat flavors Pvt. Ltd., India) were procured from local markets in Bhopal, India. Eggs were purchased fresh on the day of the experiment.

2.2.2. Preparation of cake batter

Formulations presented in Table 2 were used to produce cake with wheat flour and eggs as control (C). T1 was made using soaked chia seeds as egg-replacer and 5% of wheat flour replaced with flaxseed meal. Flaxseed meal was prepared by powdering flax seeds using an analytical mill at high speed for 1 min followed by sieving through mesh size 300 microns. T2 was made using soaked chia seeds as egg-replacer and nano-encapsulated flax seed oil powder. The method given by Agrahar-Murugkar, Zaidi, & Dwivedi, 2018 was followed to prepare the cakes. The batter was baked at 180 °C for 50 min cooled at RT for 1 h and used for further analysis. All batters and cakes were prepared thrice and measurements taken in triplicates for each cake and batter.

2.2.2.1. Physical and textural characteristics of batters. The specific gravity of cake batter was measured by the method of Turabi, Sumnu, & Sahin (2008). Batter density was determined using the method of Ronda, Oliete, Gómez, Caballero, & Pando (2011). Back extrusion was used to measure textural properties of cake batters using TA-XT plus Texture Analyser of Stable Micro Systems, UK equipped with a 50 kg-f load cell and operated through Texture Expert (Texture Technologies Corp., New York, USA). During the test, the disc penetrated to a depth of 30% (of sample height) at 1 mm/s test speed and returned to its original position. Data was recorded at a rate of 200 points per second. Texture quality parameters viz. firmness, consistency, cohesiveness and Index of viscosity were estimated using the procedure defined by Angioloni, & Collar (2009). All measurements were taken in triplicates.

2.2.2.2. Rheological properties of cake batters.

2.2.2.2.1. Batter flow behavior. Steady shear properties were obtained at 25 °C using a parallel-plate rheometer (Anton paar, Germany: Physica MCR 51). The plate gap was 1 mm. Shear rate increased linearly between 1 and $200s^{-1}$, and was applied to 2–3 g sample for 5 min. Through-out the tests, shear rate-shear stress and shear rate-apparent viscosity data were collected. Data were fitted to Casson Eq. (1), Herschel-Bulkley Model Eq. (2) and Power law rheological models Eq. (3):

$$\sigma^{0.5} = K_{oc} + K_c\ (y)^{0.5} \tag{1}$$

Casson yield stress (σ0c) and Casson plastic viscosity (ηca) was determined as square of intercept (K0c) and slope (Kc) respectively (Rao, 2014).

$$\tau = \tau 0 + K.\gamma n \tag{2}$$

Where σ is shear stress (Pa) and γ is shear rate ($\mathbf{s^{-1}}$). $\tau_{\ 0}$ is yield stress calculated from Casson Model (Eq. 1), KH is consistency index (Pa.sn) and n H is flow behaviour index of the model (Rao, 2014).

This non-linear model, with appropriate values of τ_0, K and n, the model can be converted to power law model [Eq. (3)].

$$\tau = K.\gamma n \tag{3}$$

where τ (Pa) is shear stress, K (Pa s) is consistency coefficient, γ is shear rate ($\mathbf{s^{-1}}$) and n is the dimensionless flow behaviour index.

2.2.2.2.2. Batter viscoelastic behaviour. Small amplitude oscillatory shear flow measurements of storage modulus, G', loss modulus, G", and loss tangent, tan δ = G"/G' were obtained using 50 mm diameter parallel plates using the method of Agrahar-Murugkar et al. (2018). The temperature was controlled at 25 °C ± 0.1 °C using a Peltier controller and humidity covers were used to prevent drying of the sample (Singh, Liu, & Vaughn, 2012).

2.2.3. Physical and textural characteristics of cakes

Cake density was calculated using the method of de la Hera, Ruiz-París, Oliete, & Gómez, (2012) and cake volume was measured by the method of Sowmya, Jeyarani, Jyotsna, & Indrani (2009). The volume index of cake samples was measured according to AACC method 10-90.01 (AACC, 2001).

Weight loss (WL%) during baking was calculated by method of Rodríguez-García, Puig, Salvador, & Hernando (2013).

Textural characteristics were assessed through texture Analyzer (TA-XT2, Stable Micro Systems Ltd., Surrey, UK). Texture Profile Analysis (TPA) test was performed through a two-bite compression test at a pre-test speed of 5 mm s-1, test speed of 0.25 mm s-1, the time interval of 10 s and strain deformation of 25% through an aluminum cylindrical probe of 75 mm diameter. The hardness was obtained from a maximum force of the first bite of TPA test (F1); the ratio of areas (A2/A1) from force-distance curves indicated cohesiveness and springiness being determined by dividing the second peak time by the first peak time (t2/t1) (Agrahar-Murugkar et al., 2018).

2.2.4. Proximate and functional quality of cake

The fat, protein, calcium and iron contents of cakes estimated by standard methods (AOAC, 2019). Phenolics were quantified by the Folin–Ciocalteu method as described by Siwela, Taylor, de Milliano, & Duodu, (2010) with gallic acid (50-500 μg/ml) as standard and expressed as GAE mg/100g. Flavonoid content (QE mg/100g) was determined using Aluminium-chloride colorimetric method by Kiranmai, Kumar, & Mohammed (2011). Stable 1,1-diphenyl-2-picryl hydrazyl radical (DPPH) was used for determination of free radical-scavenging activity (RSA) by the method described by Ozcelik, Lee, & Min (2003).

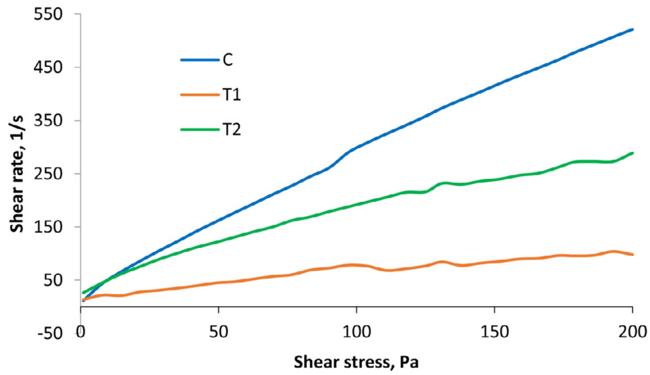

Fig. 1. a. Flow curve showing shear rate –shear stress relationship of cake batters where C- Control, T1- Chia and Flaxseed, T2-Nanoencapsulated flaxseed oil. b. Flow curve showing shear rate - apparent viscosity relationship of cake batters C- Control, T1- Chia and Flaxseed, T2-Nanoencapsulated flaxseed oil

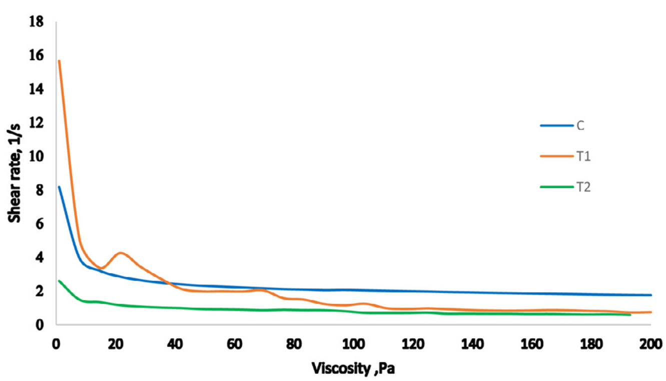

Fig. 1. Continued

For fatty acid analysis 0.5 to 1.0 g of dried cake powder was mixed with methanolic HCl using a vortexer (Spinix, Tarson, India) and then heated at 80 °C for 2 h. The mixture was cooled to room temperature (RT), n-hexane was added to the mixture and centrifuged (Thermo Fisher Scientific India Pvt Ltd.) at 5000 rpm. The supernatant of n-hexane layer was used for analysis. Quantification of fatty acid methyl esters was performed using gas chromatography with flame ionization detector (Agilent GC-7820A) (Zanwar, Hegde, Rojatkar, & Bodhankar, 2014). Fatty acids were identified relative to known external standards (FAME Mix C4-C24, Supelco, Bellefonte, PA. USA). All analyses were conducted in triplicate.

2.3. Statistical analysis

The experiments were carried out in triplicate and results are presented as mean values with standard deviations. Different mean values were analyzed by one-way analysis of variance (ANOVA) and Duncan test using SPSS software version 18.0. A probability level of $p<0.05$ was considered to be significant for all statistical procedures.

3. Results and Discussion

3.1. Characteristics of nano-encapsulated powders

Table 1a gives the quality of nano-encapsulated powders and shows that wall composition significantly influenced moisture content. Moisture content of powders containing GA showed a lower moisture content which is beneficial to keeping quality. All powders had moisture lower than 6% which is a good indicator of low susceptibility to microorganism proliferation (Barra et al., 2019). The pH of all wall materials were

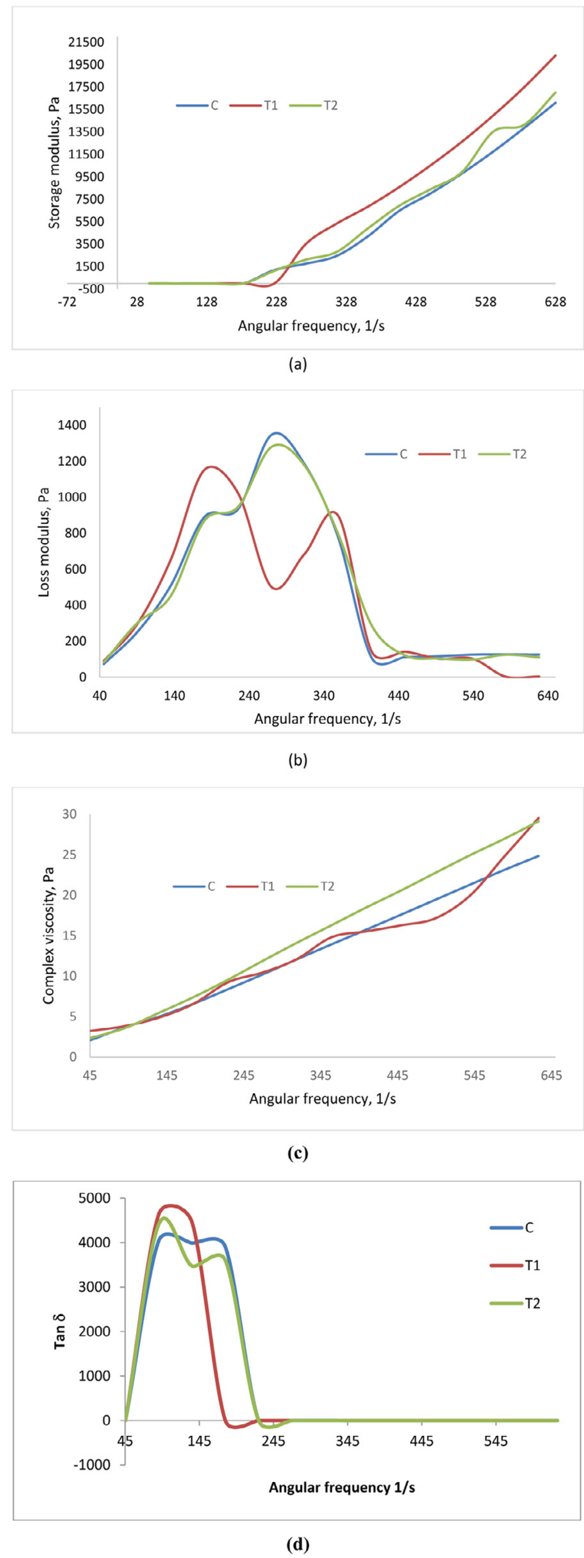

Fig. 2. (a) Storage modulus (b) Loss modulus (c) Complex viscosity (d) Tan (δ) parameters of cake batters where C- Control batter, T1- Chia and Flaxseed batter, T2-Nanoencapsulated flaxseed oil batter

Table 3
Physical properties and texture of cake batters.

Batter	Physical properties			Texture			
	Moisture (%, wet basis)	Specific gravity	Batter Density (g/ml)	Firmness (N)	Consistency (N.s)	Cohesiveness (N)	Index of Viscosity (N.s)
C	30.8 ± 0.06	2.26^{a} ± 0.03	0.95^{b} ± 0.47	2.03^{b} ± 0.21	55.4^{b} ± 5.96	-4.84^{b} ± 0.23	-8.43^{b} ± 0.63
T1	31.6 ± 0.45	1.20^{b} ± 0.03	1.04^{a} ± 0.02	2.23^{a} ± 0.14	62.1^{a} ± 4.11	-5.62^{a} ± 0.37	-11.14^{a} ± 0.76
T2	31.4 ± 0.21	2.12 ^{a}± 0.01	0.98^{b} ± 0.01	2.04^{b} ± 0.32	57.8^{b} ± 3.96	-4.82^{b} ± 0.55	-8.73^{b} ± 0.21

Where C- Control cake, T1- Chia and flaxseed cake, T2- Nano encapsulated flaxseed cake
Mean ±SD, results are the average of six replicates; for each analysis, data followed by different letters in the same column are significantly different ($p \leq 0.05$).

Table 4
Casson, Power and Herschel Bulkley model constants for cake batters.

Models	Casson				Herschel Bukley					Power Law			
Cakes	Casson's yield stress (Pa. s)	Casson's plastic viscosity	R^2	MSE	Consistency index (Pa s^n)	Yield stress (Pa. s)	Flow behavior index (n)	R^2	MSE	Consistency index (Pa s^n)	Flow behavior index (n)	R^2	MSE
C	5.68	2.14	0.94	16.43	1.23	20.69	1.18	0.99	498.53	9.40	0.75	0.99	223.08
T1	13.92	0.27	0.75	69.09	4.65	11.31	0.61	0.84	587.85	7.77	0.52	0.97	203.33
T2	13.10	0.23	0.93	88.65	2.22	20.41	0.79	0.97	54.02	7.67	0.57	0.95	

Where C- Control cake, T1- Chia and flaxseed cake, T2- Nano encapsulated flaxseed cake

mildly acidic due to protein nature and may decrease the rate of lipid oxidation (Kim et al., 2016).

Sodium caseinate powders showed higher bulk density and lower interstitial air compared to powders containing SPI. Similar values for bulk density were reported by Koç,et al. (2014) of microencapsulated flaxseed oil. Particle size is important for functionality of the inclusion complexes as a delivery system. All powders had particle sizes below 100 nm in the nano-range (Kouassi et al., 2012). Nanoparticles with zeta Potential values greater than +25 mV or less than -25 mV have high degrees of colloidal stability and the powders lay within the stable range. Particles encapsulated with AG and SPI had highest EE and lowest surface oil because of the smaller diameter. The difference of molecular weight of wall materials and viscosity in the continuous phase reduced mobility of the droplets and created enough time for adsorption of the emulsifier onto the surface of the water and oil droplets. This may have reinforced droplets against sedimentation and stabilized the emulsion, creating capsules with higher EE values after drying. These results are comparable with reports for squalene encapsulated in chitosan (Kumar et al., 2017). Higher wettability and dispersibility in powders containing SPI showed increased solubility and dissolution rate (Palzer, 2010), which may lead to greater availability of the nano-encapsulated active substance. The stability of microencapsulated oils after powder storage was evaluated based on peroxide value which ranged from 1.14 in P2 to 4.64 meq O_2/kg in P4.

Table 1b gives the fatty acid composition of the nano-powders. It was observed that P2 showed the highest ω-3 content and lowest peroxide value (Table 1a), indicating the better oxidative stability.

With all these above factors in mind a combination of AG (4%) and SPI (16%) was optimized as the wall material for the flax seed encapsulated nano-powder. All further studies on cakes were carried out using this combination.

3.2. Effect of nano-encapsulated flaxseed oil on cake batter properties

Physical characteristics (Table 3) showed that C and T2 had higher specific gravity and lower density which is related to high degree of air incorporation, improving cake volume (Campbell, & Mougeot, 1999). Batter texture studies, revealed that T2 batters showed no significant difference with C. Higher values of hardness indicate that the cakes are firmer. Consistency is the positive area under the extrusion graph; the larger the area, the denser the sample. The negative region of the graph is drawn when the extrusion probe returns and is defined as viscosity index and depends on the weight of the sample lifted by the surface of the probe. The maximum negative force is taken as a measurement of the cohesiveness of the batter. The significant enhancement of textural properties of T2 was due to nano-encapsulated particles vis a vis flaxseed meal in T1. T1 therefore showed higher moisture and high water-holding and water-absorption capacity enhancing swelling ability in chia gum resulting in a higher viscosity of batter (Segura-Campos, Ciau-Solís, Rosado-Rubio, Chel-Guerrero, & Betancur-Ancona, 2014).

3.3. Rheological properties of batters

3.3.1. Flow behavior

Fig. 1 (a-b) presents the relationship of shear stress and apparent viscosity with the shear rate of the batters. From the flow curves it was observed that shear stress (Pa) increased with shear rate while apparent viscosity decreased. This decrease in apparent viscosity indicates that cake batters exhibited shear thinning or psuedoplastic behaviour in the presence of yield stress which is in line with the findings of Agrahar-Murugkar et al. (2018). Fig. 1 (a) showed that at all shear rates the shear stress values of C were higher than test batters. This could be due to the presence of chia gel which increased moisture and elasticity resulting in lower resistance of batter in a flow field resulting in decreased shear stress. The shear stress values of C and T2 batters were lower than T1, especially at higher stress rates, indicating a lower degree of shear thickening and lower elasticity. The curves of the T1 were not as smooth probably due to the presence of flax seed powder.

Shear stress (σ) versus shear rate (γ) data obtained for different cake batters were fitted to Casson (Eq. 1) and Herschel Bulkley model (Eq. 2) and Power model (Eq. 3) (Table 4). Casson and Herschel Bulkley model fitted poorly but Power Model fitted well for T1. However for C and T2 all three models fit well, but the best fit with highest r^2 values were observed for the Herschel Bulkley model. Under the Herschel Bulkey model, the flow behavior index (n_H) of batters ranged from 0.61 to 1.18 which indicated shear thinning behavior due to alignment of microstructure with flow direction and breaking up of agglomerated particles into the smaller ones resulting in increased fluidity (Song, Kim, & Chang,

Table 5
Physical properties and texture profile of cakes.

Cake	Physical properties					Texture					
	Moisture (%)	Volume of cake (cm^3)	Volume index (mm)	Cake density (g/cm^3)	Weight loss (%)	Hardness (N)	Springiness	Resilience	Cohesiveness	Chewiness (N)	Gumminess
C1	27.4[c] ± 2.31	494.16[a] ± 3.53	129.3[a] ± 2.68	0.45[b] ± 0.01	24.3[a] ± 1.12	4.8[a] ± 0.96	0.58[a] ± 0.05	0.52[a] ± 0.03	0.91 ± 0.02	2.57 [a] ± 0.05	4.4[a] ± 0.89
T1	36.6[a] ± 1.77	399.2[c] ± 2.30	71.6[c] ± 2.68	0.60[a] ± 0.06	21.1[c] ± 3.61	2.5[c] ± 0.23	0.43[b] ± 0.04	0.47[b] ± 0.03	0.88 ± 0.02	1.11[b] ± 0.08	2.2[b] ± 0.03
T2	33.9[b] ± 4.97	439.66[b] ± 1.33	107.6[b] ± 2.22	0.47[b] ± 0.02	22.0[b] ± 1.33	3.6[b] ± 0.27	0.54[a] ± 0.13	0.50[a] ± 0.08	0.89 ± 0.10	2.53[a] ± 0.13	4.1[a] ± 0.20

Where C- Control cake, T1- Chia and flaxseed cake, T2- Nano encapsulated flaxseed cake
Mean ±SD, results are the average of six replicates; for each analysis, data followed by different letters in the same column are significantly different ($p \leq 0.05$).

Table 6
Proximate, mineral, fatty acid and anti-oxidant composition of cakes on dry matter basis.

Cakes	Protein (g/100g)	Fat	Calcium (mg/100g)	Iron	MUFA (% fatty acid composition)	PUFA	Omega-3 FA	Omega-6 FA	Phenolics (GAE mg /100g)	Flavonoids (QE mg /100g	Antioxidants (%RSA)
Control	10.3[a] ± 0.88	21.0[a] ± 0.25	82[c] ± 6.8	9.81[a] ± 0.37	30.3[b] ± 0.69	46.8[a] ± 0.51	0.00[c] ± 0.00	46.8[b] ± 0.51	69.1[c] ± 1.62	3.7[c] ± 0.27	46.0[b] ± 2.54
T1	8.0[b] ± 0.39	16.9[b] ± 0.19	120[a] ± 1.8	8.14[b] ±0.64	38.4[a] ± 0.03	29.0[c] ± 1.25	17.8[b] ± 0.01	47.94[a] ± 0.52	75.4[a] ± 6.81	14.3[a] ± 1.42	43.6[c] ± 1.66
T2	8.2[b] ± 0.08	17.7[b] ± 0.40	100[b] ± 3.7	8.23[b] ± 0.99	21.67[c] ±5.68	44.8[b] ±10.66	25.4[a] ± 0.17	19.4[c] ± 12.83	74.4[b] ± 5.29	13.7[b] ± 2.01	65.4[a] ± 0.34

Where C- Control cake, T1-Chia and flaxseed cake, T2- Nano encapsulated flaxseed oil and chia cake
Mean ± SD, results are the average of six replicates; for each analysis, data followed by different letters in the same column are significantly different ($p \leq 0.05$)

2006). Consistency index (K_H) gives an indication of the capacity of batter for retaining air (Tan et al., 2014). T1 had a significantly higher value for consistency index followed by T2 and C batters. T1 also contained flax seed flour which may have resulted in the increase in viscosity as compared to T2 which comprised of nano-encapsulated powder with lower water holding capacity. No significant difference was found among C and T2 batters for consistency index as well as flow behavior index.

3.3.2. Viscoelastic behaviour

Frequency sweep test at 15 different frequencies for storage modulus (G'), loss modulus (G'') and complex viscosity (η^*) and have been presented in Fig. 2(a-d). G' value is a measure of elastic behaviour, while G'' value represents viscous behaviour and at lower angular frequencies, cake batters showed lower value indicating less viscous and less elastic behaviour. However at higher angular frequencies, G' value was found to be higher than G'' value for all batters indicating more elastic behaviour. For all the batters, values of G' were higher than G'' at all frequencies indicating that the cake batter behaved like gel (Giuseppe et al., 2009). Investigation of tan (δ) values (Fig. 2d) for all batters revealed that although G' and G'' of all samples increased at higher frequency, tan (δ) decreased. Tan et al. (2014) suggested that solid-like property of batters increased at higher frequencies. The phenomenon was more prominent in C1 and T2. Batters made from egg are more solid and elastic-like with higher G' values than G'' resulting in lower value of tan (δ) (Tan et al., 2014) as is seen in our study as well. It is noteworthy that the trends in case of T2 are similar to C as compared to T1.

3.4. Physical and textural properties of cakes

Significant differences ($p<0.05$) between cakes was observed in all parameters except cohesiveness (Table 5). Highest moisture and cake density; lowest volume, volume index, weight loss, hardness, springiness, resilience, chewiness and gumminess was observed in T1.The higher moisture content may be due to the high absorption capacity of chia gel compared with eggs. Control cakes had significantly ($p< 0.05$) higher volume lower density and good textural profile than the T cakes, due to foaming, emulsifying and heat coagulation properties of egg proteins. Cake volume depends on air incorporated into batter, air produced, retained and formation of stable structure during baking. In T1, chia gel and flax seed powder increased soluble fiber, increasing batter viscosity. Loaf volume of cakes also decreased as flaxseed powder inhibited gluten development due to high fat content decreasing cake height. Our results of a decrease in texture attribute value coincide with the findings of Felisberto, Wahanik, Gomes-Ruffi, & Sil (2015). Cohesiveness quantifies internal resistance of food structure wherein the larger the amount of entrapped air in cakes, the greater the number of air channels resulting in a less cohesive structure (Matsakidou, Blekas, & Paraskevopoulou, 2010) as seen in T1 cakes probably due to chia and flax seed powder used in the formulation. Among T cakes, T2 had all textural attributes values closer to C indicating the high potential of using nano-encapsulated powders in eggless cakes.

3.5. Nutritional and functional quality

Nutritional quality results of the control and treatment cakes are shown in Table 6. The protein content of the control cake as compared with the treatment cakes was significantly higher. This may be due to the presence of egg albumin protein in C cake. The control cakes were also found to be significantly higher in fat content due to high-fat content of egg-yolk in the formulation followed by T2 which was due to the nano-encapsulated flaxseed oil. Lower fat content in T cakes can be justified by the difference in the fat content of chia gel and eggs. These findings were in agreement with the results of Zettel & Hitzmann (2016) where effects of gel from chia were studied as a fat replacer. T2 had the highest phenolic content followed by T1 and C which might be due to the addition of flaxseed meal which is high in phenolics and antioxidant activity (Kaur, Kaur, & Gill, 2017). Flavonoid content and antioxidant activity was highest in T1 followed by T2 and control. There was a significant increase in ω-3 FA content in T2 as compared to C and T1. More interestingly, ω-6 FA levels were reduced with increased antioxidant capacities in T2 as compared to T1 and control. This clearly establishes, T2 is better option over T1 for incorporation of ω-3 FA in cake in stabilized form. Total ω-3 content was found highest for T2 followed by T1 and control. The ω-6 content was highest for T1 followed by control cakes and T2.

4. Conclusion

Spray dried nano-particles containing flax seed oil were prepared using gum arabic (GA), sodium alginate, soy-protein isolate (SPI) and sodium casienate as wall materials with lecithin as the emulsifier for the formation of nano-sized complexes with flaxseed oil. On the basis of lower bulk density, particle size, stable zeta potential and high encapsulation efficiency, wettability and dispersibility a combination of GA and SPI was optimized as the wall material. This nano-powder was added to eggless cake (T2) and compared with an eggless flax seed powder cake (T1) and control cake (C). T2 showed no significant difference for batter textural properties compared to C. For rheological studies, Casson, Power and Herschel Bulkley model were fitted to all batters which showed shear thinning behavior of batters. There were no significant difference observed for flow behavior index and consistency index among T2 and C batter. However, consistency index and flow behavior index for T1 was significantly different from C and T2 cake. T cakes had significantly lower cake volume and volume index in comparison to C whereas among T cakes, T2 had all textural attributes values closer to C indicating high potential of using nano-encapsulated powders in eggless cakes. Though protein, fat and mineral content of C cakes was higher than T cakes due to the presence of egg, the latter showed very high amounts of anti-oxidants and essential ω-3 fatty acids especially in T2 cake. Overall results showed that healthy and tasty ω-3 rich cake of acceptable quality characteristics can be obtained meeting requirements of vegetarians using nano-encapsulated flax seed oil powder.

Declaration of Competing Interest

Kindly find the enclosed original research paper titled “Effect of Nano-encapsulation of flaxseed oil on the stability and characterization and its incorporation on the quality of eggless cake”. This paper is based on the original work carried out at Central Institute of Agricultural Engineering Bhopal. All the co-authors are aware of the submission of this research work and have approved of the manuscript and there is no conflict of interest. The manuscript has been prepared strictly according to the Journal format as provided. This is also to certify that the paper has not been submitted for publication/ consideration to any other journal/ magazine etc.

Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.afres.2021.100025.

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