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Development and physico-chemical characterization of microencapsulated flaxseed oil powder: A functional ingredient for omega-3 fortification

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ABSTRACT

The objective of the study was to develop and characterize highly polyunsaturated flaxseed oil powder which could serve as a potential delivery system of omega-3 fatty acids in vegan diet. Three formulations of oil-in-water emulsions containing flaxseed oil (≥35% on dry basis), whey protein concentrate (WPC)/sodium caseinate (NaCas) and lactose were prepared, homogenized and spray dried for further physico-chemical analyses. Developed flaxseed oil powder was characterized for moisture content, water activity, particle size distribution, flowing characteristics, dissolution behavior, surface characteristics, oxidative stability and oil release behavior under simulated gastro-intestinal conditions. The results revealed that moisture content and water activity were in the range of 3–4% and 0.346–0.358, respectively, which is suitable for long term storage of powders. Particle size distribution profile showed poly-dispersed nature with mean droplet diameter ($d_{4,3}$) in the range of 5.82 to 10.01 μm. Scanning electron micrograph of microcapsules showed spherical shapes without any apparent fissures on surfaces. Peroxide value (PV) indicated high oxidative stability of microencapsulated oil at the end of six months storage at room temperature (35 ± 1 °C). Prepared flaxseed oil powder was fortified (at 1% level) in market milk, which showed sensory characteristics comparable to control ($p < 0.05$) for up to 5 days of storage. It can be concluded that flaxseed oil could be stabilized at higher concentration which can also be used as a fortifying agent in milk for meeting the nutritional requirements of ω − 3 fatty acids in vegan and non-fish eating meat eaters' diet.

1. Introduction

Omega-3 (ω − 3) fatty acids are essential fatty acids, which are primarily restricted to sea foods and some vegetable oils (such as flaxseed oil, canola oil). Several previous investigations have suggested that ω − 3 fatty acids are critical for appropriate growth and development in humans. As such, it is not surprising that ω − 3 fatty acids have also been implicated in the prevention of systemic diseases and syndromes including coronary heart diseases, hypertension, hypercholesterolemia, cancer (including colon, breast and prostate), inflammatory bowel diseases, diabetes and neurodegenerative disorders [1–3]. However, despite their essential roles in human growth, dietary ω − 3 fatty acids are mostly limited to sea-food consuming population only. Therefore, both vegetarians and non-fish eating populations are apparently at a disadvantage of not receiving appropriate levels of ω − 3 in their general diet. On the contrary, the consumption of ω − 6 rich refined vegetable oils (such as soybean, sunflower and groundnut oil) among general population has gradually increased in the last two decades resulting in disruption of ω − 6:ω − 3 metabolic homeostasis. Furthermore, it is also speculated that higher levels of ω − 6 in regular diet could be one of the major factors responsible for increased risk of cardiovascular and inflammatory disorders. According to a report by World Health Organization, cardiovascular diseases were responsible for over 17.3 million deaths in the year 2008, representing approximately 30% of global mortality in the world [4]. Therefore, it is strongly believed that adequate ω − 6:ω − 3 ratio could be beneficial to health because of increased antioxidant, anti-inflammatory and anti-arrhythmic functions. This scenario further gains importance as ω − 3 fatty acids have limited accessibility as well as suitability owing to diverse socio-geographical constraints of the consumers. Thus it is not surprising that nutritional strategies aiming at development of ω − 3 rich novel functional foods have gained prime importance among nutritionists worldwide.

Flaxseed (*Linum usitatissimum*) oil is the richest vegetarian source of ω − 3 fatty acid, comprising 50–60% α-linolenic acid (ALA, C18:3). Although available widely, flaxseed is not preferred as it is prone to oxidation due to high levels (~75%) of polyunsaturated fatty acids (PUFAs), resulting in production of off-flavors and toxic peroxides on

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heating [5,6]. Therefore, despite being the richest and prevalent source of ω − 3 fatty acids, flaxseed oil continues to remain potentially untapped for fulfilling the ω − 3 nutritional requirements in humans. Thus, strategies aimed at stabilization and fortification of flaxseed oil could have profound influence in developing novel ω − 3 rich functional ingredients and foods. Previous studies have attempted microencapsulation of flaxseed oil (9–20%) which however, could not yield satisfactory levels of ω − 3 in the final product [7–9]. Other researchers have also worked on the microencapsulation of omega-3 oils (flaxseed, chia and fish oil) but from a different point of view. Tontul and Topuz [10] studied the influence of emulsion composition and ultrasonication time on flaxseed oil powder properties and concluded that maltodextrin-WPC was the most successful combination for microencapsulation of flaxseed oil. Ixtaina et al. [11] worked on microencapsulation of chia oil using sodium caseinate and lactose and investigated the influence of operating conditions (homogenization pressure and spray-drying inlet/outlet temperatures) on physico-chemical characteristics of chia oil powder. Goyal et al. [12] studied the effect of microencapsulation on the oxidative stability and in vitro release behavior of flaxseed oil and concluded that milk proteins could successfully protected the highly polyunsaturated flaxseed oil. By keeping all these factors in consideration, the main objective of the study was to microencapsulate and stabilize high amount of flaxseed oil (≥35% on dry basis) using whey protein concentrate-80, sodium caseinate and lactose so as to develop a potentially efficient delivery system of ω − 3 fatty acids in vegetarian and non-fish eating meat eaters' diet. The other objective was to investigate in-vitro release behavior of flaxseed oil from microcapsules under simulated gastrointestinal conditions. The stabilized flaxseed oil powder was further utilized for fortification of milk followed by its organoleptic evaluation. A milk based delivery system was preferred owing to its widespread availability among general population.

2. Materials and methods

2.1. Materials

Refined flaxseed oil was procured from Kamani Oil Industries Pvt. Ltd., Khopoli, Maharashtra, India. Sodium caseinate and whey protein concentrate (WPC)-80 (Davisco, USA) were purchased from Ace International LLP, New Delhi, India. Whey protein concentrate (WPC) was claimed to contain 82.5% protein (on dry basis), 6.4% fat, 0.2% moisture, 7.5% lactose and 2.4% ash content. Lactose was purchased from Fischer Scientific, Mumbai, India; and antioxidants were purchased from Sigma-Aldrich, Germany. Other chemicals were of analytical grade and were purchased from Sigma-Aldrich, Germany and Himedia, Mumbai, India.

2.1.1. Qualitative determination for the presence of antioxidant in flaxseed oil by thin layer chromatographic (TLC) method

Before starting the experiments, flaxseed oil was qualitatively analyzed for the presence/absence of any synthetic antioxidants (BHA, BHT and TBHQ) through thin-layer chromatographic method. To check the presence of antioxidants, first each of the synthetic antioxidants was added in flaxseed oil at 100 ppm concentration. Then the antioxidants were extracted from the flaxseed oil (antioxidants added and sample) by AOAC [13] method. The developing glass chamber was saturated with petroleum ether:benzene:glacial acetic acid::2:2:1 solvent system. Six microliter extract solution was applied along with standards on TLC plate coated with silica gel G (Merck, Germany). The plate was developed to a distance of 15 cm, then left for air drying and finally sprayed with Gibb's reagent. The plate was re-dried at 103 ± 2 °C for 5 min. Color and position of spots (bands) were compared with the standards. Chromatogram indicated no bands in flaxseed oil used corresponding to the bands of BHA, BHT and TBHQ (chromatogram is not shown here). This TLC profile of the flaxseed oil clearly suggested that there was no synthetic antioxidant present in the oil used in the study.

2.1.2. Preparation of emulsions followed by spray drying

Three formulations of emulsions were used to encapsulate flaxseed oil (Table 1). These formulations were made to obtain a high solid content in O/W emulsions (32.5% on wet basis) and to accommodate high content of flaxseed oil (~35% w/w, on dry basis) in the final spray dried powder. In the first formulation (FO/WPC), lactose and WPC were mixed in distilled water followed by addition of flaxseed oil. In the second formulation (FO-AO/WPC), ascorbyl palmitate was mixed in flaxseed oil. This mixture was further added to the pre-mixed solution of the dry-ingredients. In the third formulation (FO/NaCas), sodium caseinate was dissolved in warm water (55 ± 5 °C) followed by addition of lactose and flaxseed oil.

All the formulations were mixed well and homogenized at low pressure [69 bar @ 20 l per hour (LPH)] to obtain coarse emulsions, followed by high pressure homogenization (241.31 bar @ 20 LPH; Goma Engineering Pvt. Ltd., Thane, India). The emulsions were spray dried by single stage spray drier with 10 kg/h capacity (SSP Pvt. Ltd, Faridabad, India) equipped with rotary atomizer. The rotational speed and diameter of the wheel were ~15,000 revolutions per minute (rpm) and 12 cm, respectively. Spray dryer was operated in co-current manner with air flow rate of 450 m^3/h. The tower height and inner diameter of the spray dryer were 4 and 2 m, respectively. Emulsions were pumped to the spray drier at a flow rate of 40 mL/min at room temperature (30–35 °C). The inlet and outlet temperatures of spray dryer were maintained at 170 ± 1 °C and 75 ± 1 °C, respectively. The developed spray dried preparations [microencapsulated flaxseed oil powder (MFOP)] were packed separately in an aluminum foil pack (thickness: 80–100 μm) and stored at room temperature (30–35 °C) for further analysis.

2.1.3. Physico-chemical characterization of flaxseed oil microcapsules

2.1.3.1. Moisture content and water activity (a_w). The moisture content of flaxseed oil powder was determined by a Halogen moisture analyzer (WENSAR, HMB 100, Bengaluru, India) standardized at 108 °C for 5 min. Each analysis was performed in triplicates. Water activity (a_w) of MFOP was measured using water activity analyzer (AQUA Lab Pre, Decagon Devices, WA, USA) at a temperature of 35 °C. The instrument was calibrated first with charcoal powder at 35 °C.

2.1.3.2. Bulk (ρ_B) and tapped (ρ_T) density. For bulk (ρ_B) density, each powder sample (2 g) was filled in 25 mL measuring glass cylinder (diameter 2.5 cm) and the cylinder was slightly tapped to remove the powder sticking to the walls. The volume (Vo) was read directly from the cylinder and bulk density was calculated by using following formula (Eq. (1)). For tapped density (ρ_T), the cylinder was tapped manually approximately 50 times on marble solid surface from a height of 10 cm to measure final volume (Vn) of the powder (Eq. (2)):

$$\text{Bulk density}(\rho_B) = m/Vo \tag{1}$$

$$\text{Tapped density}(\rho_T) = m/Vn. \tag{2}$$

2.1.3.3. Flowing properties. The flowing characteristics of MFOP were evaluated by using Carr's index (Compressibility Index: C) and Hausner ratio (HR) by the method given by Turchiuli et al. [15]. Carr's index indicates the compressibility or free-flowing property; while HR indicates the cohesiveness of powder. The Carr's index (C) and Hausner ratio (HR) were calculated using bulk density and tapped density by the following equations (Eqs. (3) and (4)):

$$\text{Carr's index}(C) = \frac{\text{Tapped density}(\rho_T) - \text{Bulk density}(\rho_B)}{\text{Tapped density}(\rho_T)} \times 100 \tag{3}$$