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Compressible extruded granules containing microencapsulated oil powders

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ABSTRACT

Gelatine-based dough formulations (47%, 60% or 70% *w/w* total solids) with embedded microencapsulated fish oil powders were extruded, dried and compressed to obtain tablets with 35% *w/w* oil. The effects of dough total solids and pre-conditioning temperature (20, 35 or 50 °C) prior to extrusion on the amount of oil leakage from the tablets were examined. Oil leakage from tablets was lowest when made from extrudates of dough with 47% *w/w* TS and pre-conditioned at 35 °C. Increasing dough total solids increased oil leakage. This suggests that adequate hydration of the matrix, necessary for functionalization of the protein, was essential for subsequent tableting performance. The ability to make directly compressible formulations with embedded microencapsulated oil powders broadens the application of spray-dried fish oil powders into the nutritional supplement markets.

1. Introduction

There is a growing demand in the supplement market and the functional food industry for stabilized bioactive formulations for fortification of foods or as over-the-counter formulations. Bioactives and healthy oils intended for food and supplement use are usually presented in the neat form, within gel capsules, or formulated with carrier matrices to form liquid emulsions or dried encapsulated ingredients. There is interest in developing systems for delivering oils in directly compressible formulations with other bioactives for tableting applications. For these applications it is desirable to have oil powders that can be carried by a matrix and compressed into tablets with minimum oil leakage.

The prior art in the related area of incorporation of bioactives into granules, tablets and extruded products can be found in the patent literature. Spray dried vitamin powder containing carbohydrates and gelatine may be directly compressed into tablets [1]. Bioactive and nutritional components may be embedded in a plasticizable matrix and extruded at low shear and low temperature [2]. Emulsions containing omega-3 fatty acids have been incorporated into a plasticizable dough and pelletized [3]. Encapsulated particles containing polyunsaturated fatty acids in a protein matrix have been used for production of extruded food products [4]. Bioactives, such as carotene that are prone to degradation, can also be encapsulated and incorporated into matrices that are extrusion cooked [5,6]. Even though examples of formulations and processes to produce extruded formulations with bioactive components have been described, a systematic study of the relationship between the interaction of components in the matrix during extrusion and its effect on the stability of the embedded bioactive during processing and storage has not been reported.

In this work, microencapsulated fish oil powders (50% *w/w* fish oil stabilized within a heated mixture of 1 sodium caseinate:1 glucose:1 dried glucose syrup) were embedded in a gelatine-based dough formulation (47%, 60% or 70% *w/w* total solids, TS), and pre-conditioned at various temperatures (20, 35 or 50 °C) prior to extrusion through a die. The extruded noodles were dried, crushed, sieved and the granules obtained, then compressed into tablets. The effects of the dough formulation and process conditions used to prepare the granules, on the total oil retained in the tablet after compression were examined. It was hypothesized that adequate hydration and functionalization of the protein component in the dough formulation prior to extrusion were required for optimizing oil retention and stability in the compressed tablet made from the granules.

2. Materials and methods

2.1. Materials

Gelatine (9.2% moisture) from beef skin (Type B, Sample ID: AUS-13027) with a bloom strength of 200, 20 mesh was provided by GELITA Australia Pty Ltd. According to the supplier specification, the gelatine gels at a temperature below 35 °C. Sodium caseinate (NaCas) was obtained from Fonterra, New Zealand and the carbohydrates (dried glucose syrup (Fieldose 30) and glucose) from Penford, Lane

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Cove, NSW, Australia. Refined and deodorized tuna oil containing 25% docosahexaenoic acid and 5% eicosapentaenoic acid (HiDHA® 25N Food, Nu-Mega Ingredients Pty Ltd., Altona North, VIC, Australia) was made into microencapsulated oil powder using a patented process [7] in the pilot plant of CSIRO and used in Trial 1. For Trial 2, a commercially available spray dried fish oil powder (Driphorm® HiDHA® 50) manufactured using a patented process [7], was kindly donated by Nu-Mega Ingredients Pty Ltd., Altona North, VIC, Australia. The fish oil powder (3.5% moisture) manufactured for Trial 1 contained 50% *w/w* tuna oil, 50% *w/w* heated protein–carbohydrate encapsulant matrix (dry basis). The heated protein–carbohydrate matrix comprised NaCas, glucose and dried glucose syrup at 1:1:1 ratio. The commercial fish oil powder (3.5% moisture) also contained 50% fish oil, according to specification provided.

2.2. Preparation and characterization of doughs for extrusion

2.2.1. Preparation of doughs

Gelatine solutions (20%, 30% and 40% *w/w*) were prepared by dispersing the gelatine powder in distilled water at 50 °C for 30 min. The pH of the gelatine solutions was 5.2. The microencapsulated fish oil powder was added to the gelatine solution at 50 °C and mixed until homogeneous to form a dough. Preliminary experiments were carried out to determine the range of total solids that could be used to form a workable dough. From these preliminary experiments it was determined that gelatine solutions with 20%, 30% and 40% *w/w* TS, can form a workable doughs that varied from a 47% *w/w* TS (very soft dough) to 70% *w/w* TS (hard dough) after the addition of fish oil powder. Therefore three dough formulations (47%, 60% and 70% *w/w* TS) (Table 1) were examined.

2.2.2. Rheology of doughs

The doughs were mounted onto parallel plates (PP50/Q1 profile, diameter 50 mm) of a rheometer (Physica MCR 300, Anton Paar Germany GmbH). A Peltier system, consisted with a lower plate (P-PTD 200/56), was used to control the temperature. Measurements were carried out in oscillation mode (1% strain, 1 Hz) over a temperature of 20 to 80 °C at a heating rate of 2 °C/min after leaving the dough for 10 min at 20 °C. The dough was held at 80 °C for 20 min and then cooled to 20 °C at a cooling rate of 2 °C/min. Storage modulus (G′), representing elastic (solid like) nature of the materials, and loss modulus (G″), representing viscous (liquid like) nature of the materials, were recorded. Phase angle (δ°) was calculated using the equation: $\tan(\delta) = G'' / G'$. A higher phase angle means that a material is relatively more viscous and less elastic and vice versa [8]. Complex modulus (G*), representing the overall resistance to deformation of a materials, was calculated using the equation: $G^* = \sqrt{G'^2 + G''^2}$. A higher complex modulus means that a material has higher resistance to deformation regardless of whether that deformation is recoverable (elastic) or non-recoverable (viscous) [8].

Table 1
Composition of formulated doughs.

	Formulation 1§	Formulation 2§	Formulation 3§
Water (%)	53.0	40.0	30.0
Gelatine (%)	13.4	17.1	20.0
Oil powder† (%)	33.3	42.9	50.0
Oil % (from oil powder)	16.7	21.4	25.0
Total solids (gelatine + oil powder (% w/w))	47.0	60.0	70.0

§ For the preparation of formulations 1, 2 and 3, the oil powder was dispersed in 20%, 30%, and 40% gelatine solution respectively, and gelatine to oil powder ratio was 2:5 and the ratio of gelatine:heated protein–carbohydrate:oil was 2:2.5:2.5.

† The oil powder is a spray-dried oil-in-water emulsion stabilized by heated protein–carbohydrate mixture (50% *w/w* oil, 13.3% *w/w* protein and 33.7% *w/w* carbohydrate).

2.2.3. Assessment of dough extrudability at room temperature

The doughs (47%, 60% or 70% *w/w* TS) were pre-conditioned at 20, 35 or 50 °C, for 1 h prior to extrusion. These pre-conditioning temperatures were chosen as they were either below, at or above the gelling temperature of the gelatine used (~35 °C). The pre-conditioned doughs were loaded into a noodle press with internal diameter of 50 mm at room temperature (~23 °C) and pressed at an extrusion speed of 5 mm/min. A die with 36 holes (1 mm diameter) was used. The noodle press was connected to Instron 4465 and the force on extrusion of noodles was recorded, and taken to be an indication of the extrudability of the doughs.

2.3. Extrusion of doughs and production of dried granules and tablets

The procedure for making the granules and tablets is shown in Fig. 1.

2.3.1. Extrusion of doughs

The dough was extruded immediately after pre-conditioning at various temperatures (20, 35 or 50 °C). All doughs were extruded in a cool room (4 °C) to allow the noodles to gel/solidify as it exit the die and allow the collection of the formed extrudates.

2.3.2. Production of dried granules

In Trial 1, the wet extruded noodles were dried in a vacuum desiccator with silica gel at 4 °C for 5 days until it reached an a_w ~ 0.3, as measured using an Aqualab 4TE water activity meter (Decagon Devices Inc., Pullman, Washington, USA). The dried noodles were crushed and sieved (2 mm diameter) to obtain the granules. These granules were further equilibrated to ~34% relative humidity [9] over a saturated salt solution of $MgCl_2$ at 4 °C for 2 days, to make sure all the granule samples were at same water activity before compression into tablets. The drying procedure for Trial 2 was modified in order to shorten the drying time and

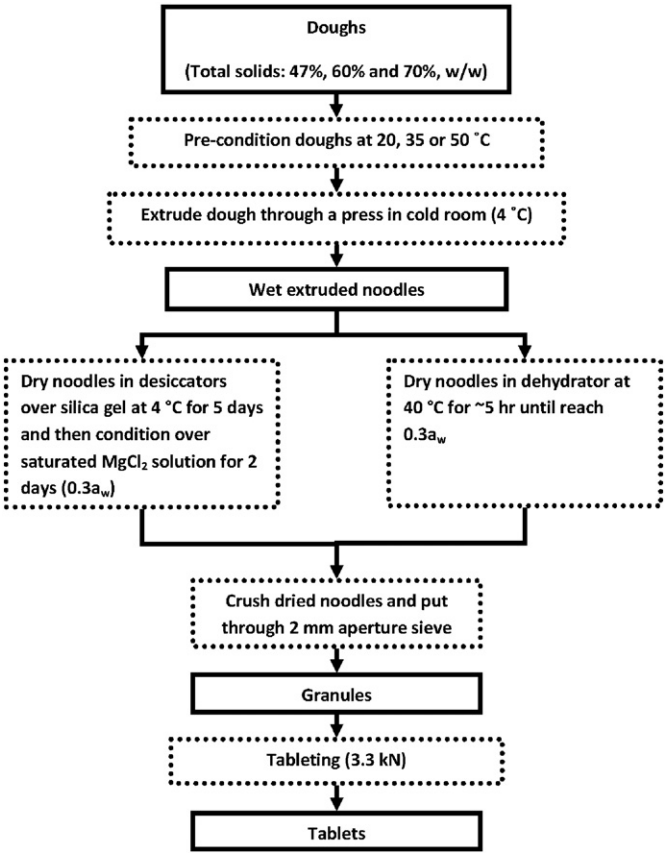

Fig. 1. Flow diagram for processing the granules and tablets.