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Pelletisation of canola meal by extrusion–spheronisation for ethanol dehydration

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ABSTRACT

Canola meal has been previously demonstrated to be an attractive biomaterial for dehydrating wet ethanol vapours in bioethanol manufacture. Extrusion–spheronisation was employed to prepare reasonably spherical pellets of canola meal for use in dehydration units. Canola meal pastes were prepared at water volume fractions of 57–70% and extruded through single and multi-holed dies with diameters 2, 3.5 and 4.5 mm. The pressure required to extrude the pastes, size and shape distribution of pellets and strength of dried pellets were measured. Formulations with a water volume fraction of 70% gave low extrusion pressures and highest pellet strength. Die land diameters of 2 mm gave the best combination of specific surface area, size and shape distribution, packing density and ethanol adsorption. Dehydration testing confirmed that the canola meal pellets could dehydrate water/ethanol vapour from an ethanol mass fraction of 92.5% (below the azeotrope at 1 bara) to 99%. The equilibrium water loading of 47.3 mg water per g adsorbent is larger than other biomass-based adsorbents reported for this application.

1. Introduction

Current concerns about energy supply have prompted the development of various renewable fuel sources. Biofuels have received substantial attention because of the widespread availability of biomass. Biofuels such as ethanol, methanol, isopropanol and butanol generated by fermentation routes require separation and purification in order to achieve fuel grade specifications, and the current processing techniques have limitations.

For example, conversion of carbohydrates to ethanol via fermentation usually yields aqueous solutions with ethanol mass fractions of 5–12% ethanol in water with other organics. Recovery of ethanol from the fermentation broth to yield fuel grade ethanol is mainly performed at the industrial scale by distillation, giving ethanol–water mixtures with ethanol mass fractions of 75–92%, below the azeotrope (95.6% at 1 atmospheric pressure), followed by adsorption using adsorbents [1].

Biomaterials represent a potential source of bioadsorbents. Corn grits have been reported to be used in industry [2] but use of corn places pressure on food supply. Alternative

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bioadsorbents for bioethanol dehydration have included cellulosic materials such as canola meal [3], kenaf core [4], bleached wood pulp [4], and starchy materials such as cassava pearls [5] and corn meal [6]. Studies [3–6] have shown that surface area, density, porosity, particle size and mechanical strength of adsorbent particles have significant effects on industrial ethanol dehydration. Most of these biosorbents are prepared by grinding the raw biomaterial and the resulting particle size and shape distributions are not controlled. No results have been reported to date for biosorbents for ethanol dehydration with controlled shape and particle size. Developing methods for pelletising biosorbents with controlled size, shape and mechanical strength (friability) are important as these properties determine whether the materials can be readily used in packed bed devices.

Extrusion–spheronisation is widely used in the pharmaceutical industry to manufacture pellets with high sphericity from powder feedstocks [7,8]. In this process, powders such as microcrystalline cellulose (MCC) are combined with a liquid binder to produce a viscoplastic paste (a highly-filled suspension) which is extruded to give cylindrical extrudates which are subsequently broken up and rounded on a rotating friction plate. The pellets are then dried to remove the free (not strongly absorbed) liquid. This technology has potential for making bioadsorbent pellets from cellulosic or starchy materials. Previous work in paste processing has demonstrated that physical properties including particle size and shape, density, and porosity, liquid content and die geometry all influence the performance of the extrusion and spheronisation steps [9–11].

This paper investigates the use of extrusion–spheronisation to manufacture bioadsorbent pellets from canola meal. Canola meal is obtained by grinding the canola seed cake after oil extraction. It is an abundant by-product from canola oil extraction and biodiesel production. Its composition, by mass, is 36–40% crude protein, 12% moisture, 20% neutral detergent fibre consisting of celluloses, hemicelluloses and lignins, 5% starch, 10% free sugar and non-starch polysaccharides, 4% crude fat, and 6% ash [12]. Water adsorption by biomaterials is reported to involve the polar attraction between water and the cellulosic hydroxyl components and the protein carboxyl and amine groups in the adsorbent [13,14].

In a previous study, Baylak et al. [3] demonstrated that adsorbents prepared from raw canola meal particles were able to dehydrate ethanol from solutions with mass fraction of 65–95% ethanol to yield fuel ethanol at mass fraction higher than 99%. The meal adsorbs water from the vapour phase, so that the ethanol-rich vapour can be condensed directly to give the product. Regeneration of the meal yields water, which renders the regeneration step relatively safe. This paper presents an investigation of pelletisation of canola meal by extrusion–-spheronisation, characterization of the pellets thus generated, and a short trial of their ethanol dehydration performance.

2. Materials and methods

2.1. Canola meal

The canola meal used in this work was purchased from Federated Co-Operatives Limited (Saskatoon, Canada). The mass fractions of the major meal components (supplier assay) were: crude protein 36.0%, crude fat 2.0–5.0%, crude fibre (cellulose, hemicellulose, and lignin) 12.0%, moisture 12.0%, and non-starch polysaccharides, starch, ash, etc. constituting the remainder. The meal was sieved and particles passing through a 500 μm mesh were used for making pellets. In this paper the term 'particle' refers to individual elements of the canola meal feedstock and 'pellet' is used for the granulated assemblies of particles.

The moisture content of particles and pellets was determined gravimetrically. The weight of the sample was measured before and after oven drying at 105 °C for 24 h, or at reaching constant weight if this was reached earlier. Surface area of particles and pellets was determined by a Micromeritics ASAP 2020 surface area analyser. The true density of canola meal particles, ρ_{CM}, was measured by a pycnometer (Micromeritics AccuPyc 1330). The as-poured aerated and tapped bulk densities of the meal particles were measured on an automated tap density analyser (Quantachrome Instruments, Reading, UK) using a measuring cylinder of internal diameter 26.4 mm. The cylinder was filled to the 100 ml mark and the mass of charge measured to give the as-poured density. The accuracy of volume readings was ±0.5 ml. The tapped bulk density was obtained at 800 taps when the change of bulk volume was invisible by eye observation.

2.2. Paste preparation

Canola meal pastes were prepared by mixing canola meal particles with known amounts of reverse osmosis water in a Kenwood Chef domestic planetary mixer (Kenwood Ltd, UK). The powder was first mixed at the lowest speed for 5 min and water added slowly by pouring on to the powder bed. Paste was then mixed at the highest speed for 10 min, pausing every 2 min to remove the paste adhered on the wall and bottom of the mixing bowl with a plastic spatula. The freshly mixed paste was stored in a sealed plastic bag in a refrigerator for 2 h to allow the water to equilibrate throughout the mass. Control of laboratory conditions was important to limit evaporation. The room temperature was 22.7 ± 1.3 °C and relative humidity 0.55 ± 0.03.

2.3. Extrusion and spheronisation

The water–canola meal pastes were extruded through a computer-controlled ram extruder based on a Zwick/Roell 50 kN strain frame (Zwick Testing Machines Ltd., Leominster, UK). The extruder comprised a cylindrical ram, cylindrical 316 stainless steel barrel of internal diameter (D_0) 25 mm and various concentric square entry dies. A detailed description of the unit is given in Mascia et al. [15]. The average diameter of the pellets obtained by spheronisation is determined by the extrudate diameter, D, so different pellet sizes were obtained by using different die sizes. The dies used in this work all featured cylindrical die lands, with length (L) to diameter ratios (L/D) of 4–4.5. Single, centrally holed dies were used for initial tests, with die land dimensions ($L \times D$) 8 mm × 2 mm; 16 mm × 3.5 mm; 18 mm × 4.5 mm. A multi-holed die was also used to

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