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Impact of high pressure homogenization modification of a cellulose based fiber product on water binding properties

Marco Ulbrich*, Eckhard Flöter

Technische Universität Berlin, Department of Food Technology and Food Chemistry, Chair of Food Process Engineering, Office KL-H3, Koenigin-Luise-Strasse 22, D-14195 Berlin, Germany

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ABSTRACT

A commercial dietary fiber was mechanically modified by treating a suspension in a high pressure homogenizer. Different drying methods were applied prior to examining macroscopic and water binding properties. DSC experiments show that more water is bound non-freezable with increasing mechanical modification. Capillary suction method and centrifugation experiments confirm that the water binding capacity changes systematically on mechanical treatment. Rheological studies confirm that the modification has a profound effect on the physico-chemical properties of the fibers. Samples with higher water binding capacity result in gel-like structures relating to higher specific volumes or swelling and possibly increased mutual entanglement. The water binding is primarily linked to structural parameters, such as porosity and does not increase any further once additional mechanical treatment does not further lead to increased porosities. Electron microscope images support the experimental findings.

Effects of mechanical treatment supersede the impacts of the different drying methods. However, more gentle drying methods such as freeze drying and water-ethanol-extraction-drying yield in general higher water binding capacities than spray drying and oven drying.

The study documents that the profound effect of mechanical treatment on the water binding capacity of a cellulose based fibrous material is strongly related to the porosity of the dried samples.

1. Introduction

Starch is used for many technical and food applications (BeMiller & Huber, 2011). In the majority of the cases, the granular starch is brought into solution or molten applying heat. The occurrence of both states is strongly influenced by the presence of water. Especially at reduced water contents of about 30% (w/w) or lower, starch is able to melt (Jang & Pyun, 1996; Steeneken & Woortman, 2009). The melting characteristics of a starch, especially the transition temperature and the temperature range are, amongst others, a function of the water content and hence the water activity (a_W) (Burt & Russell, 1983).

Extrusion is a common industrial technique to process plasticizing materials like flour or starch by melting (Meuser, van Lengerich, & Reimers, 1984; Robin et al., 2011). If the starch is partly substituted by a cellulosic material, e. g. dietary fibers, a mixture with unknown water binding characteristics is processed. Simplified, at certain water content in a feed with more than one component (starch and fiber), the amount of water available to the melting starchy material depends on the water binding properties of the cellulosic fibers, too. Thus the water binding characteristics of dietary fibers are important for the processing of the other materials. Particularly during processing of a cellulosic fiber or a bran containing feed in an extruder, thermo-mechanical modification of the fiber product appears possible and consequently a continuous change of the water binding properties can be expected.

The water binding and the denotations of water located or bound differently into or onto a cellulosic material, generally polymeric polysaccharides, or polymers are not univocally described in literature (Agrawal, Manek, Kolling, & Neau, 2004; Blair, Buckton, Beezer, & Bloomfield, 1990; Froix & Nelson, 1975; Hatakeyama & Hatakeyama, 1998; Kunzek, Opel, & Senge, 1997; Li, Dickinson, & Chinachoti, 1998). Generally hydrated bio-based polymers have been extensively investigated. In the presence of a certain amount of water, hydrophilic polymers may become swollen, exhibiting changes in mechanical and processing properties, the latter especially due to impact on the interaction behavior with other chemicals or polymers.

The influence of a mechanical modification of a cellulosic material seems to be very important considering different water

* Corresponding author.
E-mail address: marco.ulbrich@tu-berlin.de (M. Ulbrich).

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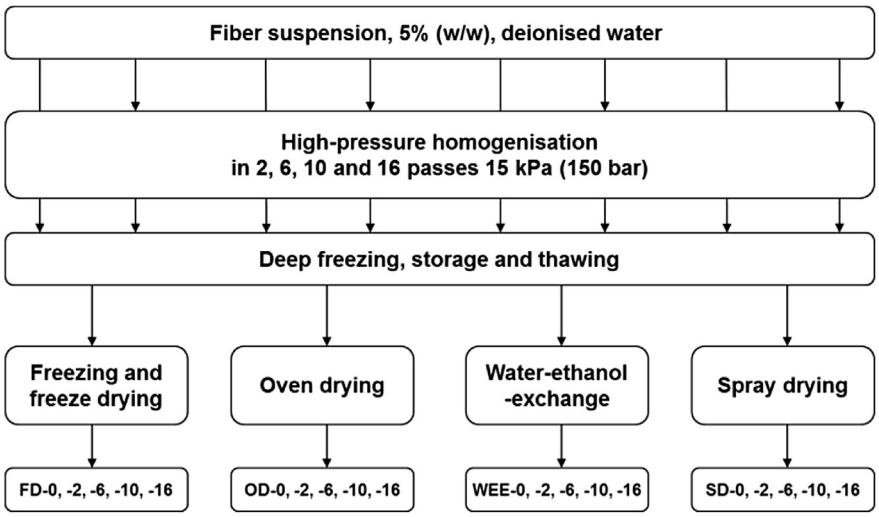

Fig. 1. Fiber modification and sample preparation.

binding mechanisms. Müller and Peters (1998), for example, stated a significant size reduction of different model drugs (no cellulosic materials) increasing high pressure homogenizer (HPH) cycle numbers. Other authors investigated the modification in terms of crystallinity. Spence, Venditti, Habibi, Rojas, and Pawlak (2010) reported a slight decrease in crystallinity of different wood pulps due to HPH treatment. Alila, Besbesa, Rei Vilar, Mutjéc, and Boufia (2013) reported drastically reduced degrees of crystallinity for microfibrillated cellulose after repeated HPH processing. Blair et al. (1990) related significantly increased moisture sorption of MCC at higher relative humidity to reduced or eliminated crystallinity. Also Luukkonen, Maloney, Rantanen, Paulapuro, and Yliruusi (2001) found an impact of mechanical modification on the water binding properties. They concluded, that granulation of MCC has the same effect as beating has on cellulose fibers during the papermaking process by opening of larger pores in the cell wall, increasing the swelling of the pulp. The bonds between the microfibrils are loosened and the external bulk water can adsorb into the internal structure of the cellulose particle causing swelling. For silicified MCC it was documented that at low moisture content only non-freezing water is present. With increasing moisture content micropores seem to fill and next to an increasing amount of non-freezing bound water also freezing bound water can be identified.

In summary the process of water binding in cellulosic samples is complex. For a single component the chemical composition of the sample and the water content of a product determine the water binding mechanisms and their interplay. In mixed systems such as starch and cellulose mixtures the emerging picture becomes more complex. In practise, as for example extruder processing, one rarely deals with a single polymeric material that has the capacity to bind water. In such a mixed system the water binding characteristics can hence not be predicted straightforwardly. Additionally, the above described shear effects during processing in an extruder can interfere with the competitive water binding.

The present work systematically investigates changes in water binding characteristics of a cellulosic material due to mechanical treatment. For a commercial dietary fiber a range of modified fiber samples with different microscopic and macroscopic characteristics was obtained after HPH treatment and subsequent drying. Both, the number of homogenization steps and the drying methods were varied during the sample preparation. Several methods were applied to assess the effect of structure changes on the water binding characteristics.

2. Materials and methods

2.1. Fiber

The fiber used is a commercial dietary fiber (oat fiber, VITACEL®, J. Rettenmaier & Söhne GmbH + Co). Its fiber content given on dry substance is approximately 96% w/w, the protein content is 0.26% w/w and fat content is about 0.1% w/w (supplier information). The moisture content was determined to about 10%. The product was stored at 20 ± 2 °C in a closed container.

2.1.1. Fiber modification through mechanical treatment

The high-pressure homogenization of the fiber material is sketched in Fig. 1. Approximately 3 L of a 5% w/w fiber suspension in deionized water were stored for 60 min at 20 °C with occasional stirring. After the first two runs at 15 MPa pressure through the homogenizer (APV Gaulin, LAB 100-4 PSX, 3 valves, Germany) a sample of the suspension was taken and stored frozen. The remaining suspension was homogenized again. In total samples were taken and stored under freezing conditions after 0, 2, 6, 10, and 16 passes through the homogenizer.

2.1.2. Drying

For freeze drying, the frozen suspension samples were thawed, portioned in stainless steel dishes and deep frozen again (−18 °C) before freeze drying (Christ alpha 1-4, Germany) for about 24–36 h. The set temperature of the ice condenser was −55 °C, the set pressure was adjusted to 0.05 mbar.

Alternatively water-ethanol-exchange was carried out as follows: Approximately 1 L of the thawed suspension was centrifuged at 3000 min^{-1} (1960 *g*) for 10 min and the clear supernatant was decanted and discarded. 250 mL of 70% ethanol (diluted with deionized water, v/v) was added to the remained pulp and stirred for 10 min and centrifuged again at the same conditions. The procedure was repeated with 90% (v/v) and 95% (v/v) ethanol and in the last step with acetone (99.9%). After the centrifugation step the samples were air dried overnight.

For oven drying the samples were thawed and centrifuged at 3000 min^{-1} for 10 min. The supernatant was decanted and the fiber was transferred to Petri dishes and dried in an oven for about 48 h at 80–90 °C. The dry product was gently crushed in a mill (IKA M 20, IKA-Werke GmbH & CO.KG, Staufen, Germany). Spray drying was carried out using a OTW 50 Spray dryer (Bühler AG, Utzwil,