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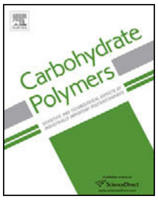

Morphological, physicochemical, and viscoelastic properties of sonicated corn starch

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ABSTRACT

In the present work, different parameters of ultrasound treatment were studied for physical modification of corn starch. The results revealed that the influence of sonication strongly depends on temperature (25–65 °C) and exposure time (5–15 min), while concentration (10–20% w/w) and ultrasound amplitude (50 and 100%) have little influence on functional and rheological properties. SEM micrographs demonstrated the damage induced by ultrasound on starch granules' surface. The solubility, swelling power, and gel clarity were increased. Ultrasonication decreased the gelatinisation enthalpy and temperature range while the X-ray pattern and crystallinity remained almost unchanged, except for samples treated at onset temperature as measured by DSC. The pseudoplasticity and consistency coefficient decreased; also, apparent viscosity diminished prominently. The pasting behaviour of samples was altered without any clear change in gel strength characterised by loss factor. The results of the present work provide further insight into the mode of action of ultrasound on modifying corn starch granules.

1. Introduction

Starch is a valuable ingredient widely used as a thickening, gelling, bulking, and water retention agent in the food industry. Some technological applications of starch require specific functional properties, which could not be satisfied by starch in its native form; consequently, different methods of starch modifications including chemical, enzymatic, and physical processes have been proposed and adapted to fulfil these requirements.

Ultrasound treatment, as a physical method of starch modification, has shown many advantages in terms of higher selectivity and quality, reduced use of chemicals and processing time, and finally serving as an environment-friendly processing (Chemat, Huma, & Khan, 2011; Zuo, Knoerzer, Mawson, Kentish, & Ashokkumar, 2009). Applications of ultrasound technology in different areas of food science have been well documented elsewhere (Chandrapala, Oliver, Kentish, & Ashokkumar, 2012; Chemat et al., 2011; Ebringerova & Hromadkova, 2010; Knorr, Zenker, Heinz, & Lee, 2004; Mason, Paniwnyk, & Lorimer, 1996; McClements, 1995; Soria & Villamiel, 2010). The term ultrasound refers to the sound waves exceeding the audible threshold of human hearing (18 kHz), which originates from elastic deformation of ferro- and/or piezo-electric materials (called transducer) within high frequency electrical fields that create high-energy vibrations (Jambrak et al., 2010). Based on the solvent's capacity for absorbing and transmitting the energy of ultrasound, these vibrations cause cavitation, and subsequently, the collapse of cavitation bubbles results in hot spots (high temperatures, ca. 5000 K), high pressures (1000–2000 atm), and microjet streaming with high velocities of about hundreds of m/s through the medium and adjacent to interfaces (Czechowska-Biskup, Rokita, Lotfy, Ulanski, & Rosiak, 2005; Ebringerova & Hromadkova, 2010; Soria & Villamiel, 2010).

Several works have been published regarding ultrasound treatment of different types of starch with different botanical origins, either in the granular or gelatinised form. From an extensive investigation of the available literature, it was revealed that sonication affects the physico-chemical properties of starch including: solubility, swelling power, gelatinisation temperature and enthalpy, depolymerisation, viscosity, and surface properties (Azhar & Hamdy, 1979; Chan, Bhat, & Karim, 2010; Chung, Moon, Kim, & Chun, 2002; Czechowska-Biskup et al., 2005; Degrois, Gallant, Baldo, & Guilbot, 1974; Gallant, Degrois, Sterling, & Guilbot, 1972; Huang, Li, & Fu, 2007; Iida, Tuziuti, Yasui, Towata, & Kozuka, 2008; Isono, Kumagai, & Watanabe, 1994; Izidoro, Sierakowski, Windson Isidoro Haminiuk, de Souza, & de Paula Scheer, 2011; Jambrak et al., 2010; Lima & Andrade, 2010; Luo et al., 2008; Montalbo-Lomboy et al., 2010; Ono, 1940; Seguchi, Higasa, & Mori, 1994; Wu, Du, Ge, & Lv, 2011; Zhu, Li, Chen, & Li, 2012; Zuo et al., 2009; Zuo, Hebraud, Hemar, & Ashokkumar, 2012).

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The effects of ultrasound on starch depend strongly on processing parameters (such as frequency, power, amplitude, time, and temperature of sonication), the properties of the starch (such as composition and physical state), and suspension concentration (Czechowska-Biskup et al., 2005; Sujka & Jamroz, 2013; Zuo et al., 2009).

From a critical review of the published works, it was observed that most of the research has focused on one or a few physical and/or functional properties of starch in relation to sonication treatment and that there was not comprehensive information in terms of morphological, functional, and viscoelastic properties altogether. In addition, most of the research investigated the sonication of either granular or gelatinised starch, and the influence of physical state of starch in relation to sonication temperature have not been studied. Therefore, the aim of the present work was to perform a complete investigation to assess the effect of sonication amplitude (0–100%), time of exposure (5–15 min), temperature (25–65 °C), and suspension concentration (10–20% w/w) on functional, morphological, and viscoelastic properties of corn starch.

2. Materials and methods

2.1. Materials

Corn starch (S4126) was supplied by Sigma (St. Louis, MO, USA). All chemicals were of reagent grade (Merck, Darmstadt, Germany) and were used as received. The deionised water was used in all experiments, unless otherwise specified.

2.2. Ultrasound treatment

Dispersions of corn starch with varying concentrations were prepared by adding an appropriate amount of corn starch powder to a beaker containing deionised water at a specific temperature adjusted (±0.1 °C) using a thermostated water-bath. The prepared dispersions were subjected to sonication using a 24 kHz Hielscher UP200H ultrasound device (Hielscher Ultrasonics, Germany) equipped with a titanium sonotrode (3 mm tip diameter) producing a nominal output power of 150 W at specified amplitudes and time durations according to the experimental design (Table 1). The device was powered with an automatic frequency tuning system which adjusts the radiation to optimal frequency to ensure that maximum power is transmitted to the reaction vessel. In order to eliminate the influence of varying energy volume densities, the volumes of sample slurries were kept constant (50 mL). The sonotrode was immersed into the sample vessel at a depth of approximately 2 cm. The sonication was performed in the pulsed mode (80% duty cycle) in order to reduce the formation of free radicals and heat generation and also to protect the sonotrode from any abrasion. To prevent any temperature rise by sonication, the sample vessels were held in the water bath during treatment. The sonicated samples were lyophilised, sieved (No. 70 mesh), and placed in air-tight containers before performing the experiments. The non-sonicated samples, denoted as 0% amplitude in the experimental design, were prepared in the same way as the sonicated samples, but with the transducer turned off.

2.3. Morphology

2.3.1. Light microscopy (LM)

A drop of water was placed on a glass slide, and starch samples were sprinkled into the drop and mixed in it. The suspension was covered with a glass cover slip and then observed and photographed using an Olympus BX41 microscope equipped with a DP12 digital camera (Olympus Corporation, Japan). The captured images were analysed for particle size using Image Pro Plus 6.0 software (Media Cybernetics Inc., USA). Granule size of samples was measured on at least 100 randomly selected granules from micrographs at five replicates and the mean value was reported (Jambrak et al., 2010).

2.3.2. Scanning electron microscopy (SEM)

SEM micrographs of starch samples were recorded using LEO 1450VP scanning electron microscope (LEO, Oberkochen, Germany). The starch samples were sprinkled onto double-sided adhesive tape attached to aluminium stubs, coated with gold–palladium (for 120 s) using a SC7620 sputter coater (Quorum Technologies, UK) and photographed in microscope at an accelerating voltage of 20 kV (Sujka & Jamroz, 2013).

2.4. Solubility and swelling power

Solubility and swelling power determinations of samples were carried out according to the method of Leach, McCowen, and Schoch (1959). Briefly, 1% w/v dispersions of samples were prepared in screw-cap tubes and heated in a thermostated water-bath for 30 min at 25, 45, 65, and 85 °C with continuous mixing at 5 min intervals using a vortex mixer. Afterwards, the tubes were cooled to ambient temperature and centrifuged at 2400 × *g* for 20 min. The sediments (m_2) and their supernatants were collected and dried at 105 °C until constant weight was achieved (m_3 and m_4, respectively). The solubility (*S*) and swelling power (*SP*) were calculated by the following equations, respectively.

$$S\ (\%) = \left(\frac{m_4}{m_1}\right) \times 100 \tag{1}$$

$$SP\left(\frac{\text{g water}}{\text{g starch}}\right) = \frac{(m_2 - m_3)}{m_3} \tag{2}$$

where m_1 is the initial sample weight.

2.5. Paste clarity

According to the method of Craig, Maningat, Seib, and Hoseney (1989), aqueous dispersions of starch samples (1% w/v) in screw-cap tubes were heated in a boiling (95 °C) water bath for 30 min with continuous mixing at 5 min intervals using a vortex mixer, and after cooling to room temperature, the transmittances of the pastes were measured at 650 nm using a Jenway 6105 UV–visible spectrophotometer (Jenway, Staffordshire, UK). The transmittances of the pastes stored at 4 °C were also determined daily for 5 consecutive days.

2.6. Relative degree of polymerisation (DP)

Relative degree of polymerisation of samples was determined as the ratio of reducing sugars measured using 3,5-dinitrosalicylic acid (DNS) (Miller, 1959) and total carbohydrate measured using phenol–sulphuric acid (Dubois, Gilles, Hamilton, Rebers, & Smith, 1956). The glucose was used as a standard for both methods.

2.7. Differential scanning calorimetry (DSC)

Thermal properties of samples were measured using a DSC 822e calorimeter (Mettler-Toledo, Switzerland). Starch samples (5 mg) were accurately weighed and transferred to aluminium pans. Appropriate amounts of deionised water were added to prepare 15% w/w concentration. Then, the pans were sealed and allowed to rest for 1 h at room temperature before performing the experiments. The scan was done from 20 to 200 °C at a rate of 10 °C/min. An empty pan was used as reference. Onset (T_o), peak