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Influence of high-pressure homogenization on functional properties of orange pulp

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ABSTRACT

The current work evaluated whether high-pressure homogenization (HPH) could functionalize orange pulp in terms of water holding and swelling capacity and rheological properties. The orange pulp particle size was gradually decreased by applying HPH at increasing pressure (200 and 800 bar) whereby the mechanical impact at 800 bar resulted in the appearance of a more homogeneous, smoother suspension with a twofold increase in yield stress. HPH also affected the pectin properties within the orange pulp cell walls. More specifically, HPH at 800 bar increased the relative presence of water-extractable pectin. By investigating subsamples containing particles with different sizes isolated from orange pulp before and after HPH, it became clear that particle size is inversely related to water holding capacity and the ability of the particle network to deform prior to flow. Especially highly disintegrated orange pulp material (<40 μm) contributes to water holding capacity and soft particle network behavior.

Industrial relevance: Orange pulp is of particular interest in the context of producing fiber-rich functional ingredients because of its large quantity available within the juice industry. High-pressure homogenization (HPH) at pressures higher than the ones usually applied in food industry seems required to functionalize orange pulp as HPH at 200 bar (a common pressure applied in the food industry) could not increase the relative presence of small particles, contributing to water holding capacity and soft particle network behavior, as substantially compared to HPH at 800 bar.

1. Introduction

Consumption of dietary fiber is linked to health benefits associated with bowel function, reduced risk of coronary heart diseases and type 2 diabetes and weight management (Hauner et al., 2012). Unfortunately, the average total dietary fiber consumption (14 to 29 g/day) is far below the recommended intake between 21 and 40 g/day (WHO/FAO, 2003). This recommendation could be achieved by increasing the consumption of fruits and vegetables: it is suggested to eat five to nine pieces a day. On the other hand, fiber-enriched food products could also play a key role in our diet. Fruit and vegetable by-products, mainly consisting of cell wall material rich in dietary fiber, can in this context be considered as potentially interesting functional ingredients. In order to create healthy, fiber-rich food products that consumers like to eat, functional food ingredients should not only possess desired nutritional properties but also particular technological functional properties in terms of hydration properties, surface activity and texturization. Compared to commonly-used hydrocolloid ingredients, the structure-forming properties of plant cell wall-derived fibers, currently available on the market, are however limited. Because of its large quantity available within the juice manufacturing industry, orange pulp is of particular interest in the context of producing fiber-rich functional ingredients. The limited functionality of orange pulp, when dispersed in water, however confines valorization pathways for this by-product.

Mechanical processing is known to alter the physicochemical properties of plant-based fiber suspensions. During mechanical processing, particle sizes are reduced to a certain micro-scale level enhancing physicochemical properties such as the water holding capacity (WHC), the swelling capacity, the oil holding capacity (OHC) and the cation exchange capacity (CEC). Mechanical processing techniques based on high-pressure processes, e.g. high-pressure homogenization (HPH) and microfluidization, have been shown more effective in

Abbreviations: AIR, alcohol-insoluble residue; CEC, cation exchange capacity; CEP, chelator-extractable pectin; DME, degree of methyl-esterification; FITC, fluorescein isothiocyanate; HF, hemicellulose fraction; HPH, high-pressure homogenization; LVE, linear viscoelastic; MM, molar mass; NEP, sodium carbonate-extractable pectin; OHC, oil holding capacity; PBS, phosphate buffered saline; UA, uronic acid; WHC, water holding capacity; WEP, water-extractable pectin.

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particle size reduction compared to conventional mechanical processing techniques such as ball milling and jet milling (Chau, Wang, & Wen, 2007). In these high-pressure valve homogenizers, the fluid is forced through a small gap between seat and valve creating elongational and turbulence stresses modifying the fluid. Alternative technologies such as steam explosion and supercritical CO_2 explosion have also demonstrated the ability to open up the structures of plant based biomasses (Narayanaswamy, Faik, Goetz, & Gu, 2011; Tanahashi, 1990). However, in the case of steam explosion a degradation of the hemicellulose was observed, which goes paired with a reduction in water binding capacity of the plant cell wall and an increase in brittleness of the structure (Ramos, 2003). These phenomena are more desired for the use of the treated biomasses in power plants rather than for improving their texturizing properties in an aqueous system. Supercritical CO_2 on the other hand shows promising results but still remains challenging in terms of cost effectiveness.

The particle size reduction obtained during HPH is shown to go hand in hand with changes in physicochemical properties but changes clearly depend on the microstructure of the plant material. Significant increases in the WHC, the swelling capacity, the OHC and the CEC due to mechanical processing were for example found for the insoluble fiber fraction of carrot pomace, for tomato paste suspensions, and to a lesser extent for carrot suspensions, whereas no clear changes in the WHC of apple sauce and potato pulp suspensions upon HPH were noticed (Bengtsson & Tornberg, 2011; Chau et al., 2007). The relation between changes in microstructural properties and changes in functional properties due to HPH are however not yet completely understood.

The objective of the current work was to evaluate whether HPH could be used to functionalize orange pulp. Hereto, orange pulp was homogenized using different pressure levels (200 and 800 bar) and the resulting microstructural (particle size analysis and microscopy analysis) and functional properties (WHC, swelling volume and rheological properties) were determined and compared to the initial orange pulp properties. In order to evaluate the role of changes in cell wall polysaccharides in changes of functional properties induced by HPH, the alcohol-insoluble residue (AIR) of the samples was analyzed in terms of pectin extractability, degree of methyl-esterification (DME), neutral sugar composition and molar mass distribution. In the second part of this work, subsamples with different particle size ranges were prepared from the non-homogenized and the high-pressure homogenized orange pulp. These subsamples were studied in terms of microstructural and functional properties in order to better understand the functional properties of high-pressure homogenized orange pulp.

2. Materials and methods

2.1. Orange pulp

The orange pulp fibers were from Brazilian oranges, cultivar Valencia. They were from the 2010 harvest and provided by Fischer S/A, a Brazilian orange juice company. The pulp, obtained after juice extraction, was pasteurized for food safety reasons and for inactivation of intrinsically present pectin methylesterase. The pulp was subsequently poured in 180 kg vessels and frozen. The vessels were shipped by boat to Europe for the present study under frozen conditions. Moisture content of the starting pulp was about 3.4%. The samples were thawed at ambient conditions to avoid structural damage to the pulp, diluted to 2% dry matter content with standardized tap water (1.00 g NaCl and 0.15 g $CaCl_2$ in 1 L reverse osmosis water with a conductivity of 2.2 ± 0.1 mS/cm at 25 °C), blended and high-pressure homogenized. The high-pressure homogenization was performed with a NS1001L-Panda 2k equipped with a single R-valve (Niro Soavi). The R-valve, which is preferably used in the context of cell rupture, consists of a cylindrical/flat type of impact head, a small impact ring and a passage head with sharp angles. A homogenization condition commonly used in the food industry was used (200 bar) as well as an elevated pressure level of 800 bar, which is a compromise between industrial process capacity and the intended increased product functionality. Control samples, also further called 'blended samples', underwent the same preparation procedure but no HPH step was applied in this case. Samples were frozen with liquid N_2 and stored at −40 °C until further analysis (after thawing at room temperature).

2.2. Isolation of the subsamples

Orange pulp samples were loaded onto a wet-sieving column (Vibratory Sieve shaker AS200, Retsch) in order to isolate relevant particle fractions, further called 'subsamples'. Sieves with pore sizes of 250, 125, 80 and 40 μm were used. The material that was not retrieved on the smallest sieve (40 μm) was centrifuged (30 min at 12,500 *g*, Beckman Coulter centrifuge) in order to obtain subsamples that contained the smallest material. The ranges that are used to refer to the different fractions are the pore sizes of the sieves used during the isolation process.

2.3. Particle size analysis

The particle size distribution of the samples was analyzed by laser diffraction (Mastersizer, Malvern). A refractive index of 1.56 was used for the cell wall particles. From the particle size analysis, different diameters can be obtained: (i) D(v, 0.1), D(v, 0.5) and D(v, 0.9) respectively represent the maximum particle diameter below which 10%, 50% or 90% of the sample volume exists, (ii) D[4,3] shows the volume based mean diameter and (iii) D[3,2] indicates the surface area based mean diameter.

2.4. Microscopy analysis

Microscopy pictures of diluted samples (~1:10) were taken using a light microscope (Olympus BX-41). In addition, immunofluorescence using the pectin-specific antibody JIM7 was performed. Hereto, the samples were incubated with the primary antibody JIM7 (fivefold diluted in phosphate buffered saline containing 3% milk powder (MPBS); PlantProbes, Leeds, UK) for 1 h and 30 min at room temperature. After primary labeling, samples were washed with PBS by centrifugation (3 times 5 min at 22 °C and 400 *g*; Microfuge 22R Centrifuge, Beckman Coulter, Germany). For the visualization of JIM7, secondary labeling with an anti-rat Ig antibody coupled to fluorescein isothiocyanate (FITC) (Nordic Immunology, Tilburg, The Netherlands) was used. The secondary antibody was diluted 1/20 in 3% MPBS. After a final washing step with PBS, samples were mounted in an anti-fade agent (Citifluor, Agar Scientific, Stansted, United Kingdom) on glass slides and examined with the Olympus BX-41 microscope equipped with epifluorescence illumination. Immunolabeling experiments were carried out at least in duplicate.

2.5. Water holding capacity

The orange pulp fiber dispersions were diluted to a 1% concentration (on a dry weight basis) in standardized tap water by using a 4-bladed propeller fitted on a RWD 20 digital IKA stirrer (set at 900 rpm for 10 min) in a 400 mL glass beaker with a diameter of 7.8 cm. Subsequently, the dispersions were transferred into 50 mL centrifuge tubes and centrifuged at 3500 *g* for 5 min. The WHC was calculated from the gravimetric measurement of supernatant and packed hydrated fiber. All analyses were performed in duplicate.

2.6. Swelling volume

The orange pulp fiber suspensions were diluted to a 1% concentration (on a dry weight basis) in standardized tap water by using a 4-bladed propeller fitted on a RWD 20 digital IKA stirrer (set at 900 rpm for 10 min) in a 400 mL glass beaker with diameter of 7.8 cm. The dispersions