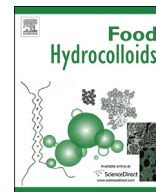


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Emulsion stabilizing properties of pectins extracted by high hydrostatic pressure, high-speed shearing homogenization and traditional thermal methods: A comparative study

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ABSTRACT

In the previous studies, two novel methods for pectin extraction, using high hydrostatic pressure (HHP) and high-speed shearing homogenization (HSHE), were developed with higher efficiency, less time and energy consumptions than traditional thermal extraction (THE); however, many functional properties of these pectins are still unknown. To explore the influence of extraction methods (HHP, HSHE and THE) and pH on the emulsion stabilizing properties of these pectins, many parameters, including zeta potential, apparent viscosity, light microscopy, and particle mean diameters of pectins or their emulsions (1% pectin with 42.8% refined soybean oil), were firstly determined compared with those of two commercial pectins (AP and SP). The results revealed that extraction methods have important influence on viscosities of pectins and their emulsions. The emulsions prepared by HHP extracted pectin have the smallest particle mean diameters (8.96–11.02 μm) and best emulsifying stability (100%) after centrifugation assay or 3 weeks of storage at 4 °C with the pH in the range of 3–5. The emulsions prepared by HSHE and THE extracted pectins have a similar particle mean diameters and emulsifying stabilities better than those made with AP and SP. The molecular weight distributions and morphological features, determined by HPSEC-RID and AFM, then indicated their difference at molecular level and their interactions. From the results, it could be concluded that emulsion stabilizing properties of pectin were greatly influenced by their physicochemical properties, including viscosity, molecular size and their interactions.

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1. Introduction

Many food products, including milk, cream, beverages, dressings, dips, sauces, batters and desserts, etc., are oil-in-water (O/W) or water-in-oil (W/O) emulsions that consist of small lipid droplets dispersed in a continuous aqueous/oil medium, which can make and maintain the necessary textures and tastes of foods (Akhtar, Dickinson, Mazoyer, & Langendorff, 2002; Guzey, Kim, & McClements, 2004). Emulsions, however, are thermodynamically unstable systems that are prone to destabilization through a variety of different physicochemical processes, including gravitational separation, flocculation, coalescence, or Ostwald ripening (Guzey et al.,

2004). One of the most important and widely used methods for improving the stability of O/W or W/O emulsions is to utilize emulsifiers (Dagleish, 2006; Dickinson, 2009; Neiryck, Van der Meeren, Bayarri Gorbe, Dierckx, & Dewettinck, 2004), of which food hydrocolloids from natural resources are the most commonly used materials, including protein, pectin, gums etc., for facilitating the formation, improving the stability, and preparing desirable quality of the emulsions (Dickinson, 2003; McClements, 2004; Nakauma et al., 2008; Thanasukarn, Pongsawatmanit, & McClements, 2004).

Pectin, a family of complex heteropolysaccharides consisting predominantly of partially methoxylated galacturonic acid residues, is extensively distributed in almost all of the fruits and vegetables as the structural unit of fresh cells and the junction between the cells (Christiaens et al., 2011; Jolie et al., 2012; Ridley, O'Neill, & Mohnen, 2001; Thakur, Singh, & Handa, 1997). Its structure is based

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on 1, 4-linked α -D-galacturonic acid, interrupted by L-rhamnose residues with side-chains of neutral sugars (mainly D-galactose and L-arabinose) (Mohnen, 2008; Ridley et al., 2001). Pectin, as a well-known food additive, is widely used as thickener, emulsifier and stabilizer in a variety of food, pharmaceutical and cosmetic products (Thakur et al., 1997). Pectin has been extracted from various agricultural byproducts, including orange peel, apple pomace, lemon and sugar beet pulp, specifically, the orange peel and apple pomace are abundant and contain high levels of pectic polysaccharides (Fishman, Chau, Cooke, & Hotchkiss, 2008; Wang et al., 2007; Yeoh, Shi, & Langrish, 2008). The most commonly used methods for pectin extraction currently were direct boiling by water acidified with mineral acid (the so-called traditional thermal/acid extraction) under the pH, temperature, and duration conditions generally in the range of 1.3–3, 60–100 °C, and 20–360 min, respectively (Liu, Shi, & Langrish, 2006; Yeoh et al., 2008). Due to a long period of heating under strong acid condition, the extracted pectin undergoes thermal/acid degradation, which could result in undesired changes in physicochemical and functional properties of pectin (Koubala et al., 2008). Currently, new technologies, including ultrasonication, high hydrostatic pressure, microwave and super-high frequency electromagnetic field, are employed in pectin extraction process to give an increased extraction yield, higher quality, less time or energy consumptions than traditional thermal extraction (Fishman et al., 2008; Guo et al., 2012; Kratchanova, Panchev, Pavlova, & Shtereva, 1994; Wang et al., 2007; Yeoh et al., 2008). Fishman, Chau, Hoagland, and Ayyad (2000) demonstrated that microwave heating within 3 min at moderate pressure (50 ± 2 psi) can effectively decrease the thermal/acid degradation and significantly increase the weight-average molar mass and intrinsic viscosity of pectin. Our previous study also confirmed that pectin extracted by high hydrostatic pressure has a significant higher intrinsic viscosity and viscosity-average molecular weight than those extracted by traditional thermal methods (Guo et al., 2012).

Many studies currently have investigated the emulsifying or emulsion stabilizing properties of pectin in O/W emulsions (Akhtar et al., 2002; Al-Hakkak & Al-Hakkak, 2010; Dickinson, 2009; Drusch, 2007; Leroux, Langendorff, Schick, Vaishnav, & Mazoyer, 2003; Neiryneck et al., 2004). Leroux et al. (2003) demonstrated that citrus pectin and beet pectin can efficiently reduce the interfacial tension between oil and water phase in the emulsions, however, their emulsion stabilizing properties of pectin varies due to the contents difference of acetyl groups, proteins and calcium ion. Akhtar et al. (2002) also confirmed that depolymerized pectins with different molecular weights have different emulsion stabilities in the rapeseed oil/gum arabic emulsion. In our previous study, we developed two novel extraction technologies for the extraction of pectin from the peels of pomelo with higher yield, less time and energy consumptions than the traditional thermal extraction (Guo et al., 2012). However, the applications of these pectins in food as a thickener, emulsifier or stabilizer have neither been investigated nor compared with traditional thermal extracted pectin or commercial pectins.

The purpose of the present study was to investigate the thickening, emulsion stabilizing properties of pectins extracted by high hydrostatic pressure (HHP) and high-speed shearing homogenization (HSHE) compared with pectin extracted by traditional thermal extraction (THE) in O/W emulsion. Many physicochemical properties and morphological features, such as apparent viscosities, droplet-size distributions, micrographs and stabilities, of the pectins or their emulsions with different pH before or after the storage were first determined to explore the differences of emulsion stabilizing properties among different pectins. The influences of molecular structure, morphological feature and extraction method on

emulsion stabilizing properties of pectins were then investigated to interpret their interactions.

2. Materials and methods

2.1. Materials and chemical reagents

Honey pomelo (*Citrus grandis* Osbeck) cultivated in Guanxi, Fujian province, was purchased from a local market of Beijing in November 2011. The collected peels of pomelo (ca. 1.5 kg) were first cut into pieces and steamed at 100 °C for 2 min to inactivate the enzymes using a Vertical Heating Pressure Steam Sterilizer (LDZX-50KBS, Shen'an Medical Instrument Factory, Shanghai, China) under the ambient pressure, the drying process was then carried out by a vacuum freeze dryer (LGJ-25C, Four-ring Science Instrument Co., Ltd, Beijing, China) with the shelf temperature at 18 °C for about 24 h until the water content was reduced to ca. 8%. The dried sample was then milled by an electric grinder (HY-04A, Beijing Huanya Tianyuan Machine Technology Co., Ltd, Beijing, China) and filtrated using a filter sieve (ca. 60 meshes). Finally, the sample was vacuum-packed and stored at 4 °C for the subsequent extraction. Two types of commercial citrus pectins, including SP9135 (Sigma–Aldrich, St. Louis, USA) and AP102 (Andre Pectin Co. Ltd, Yantai, China), extracted by traditional thermal methods with industrial scale, were purchased from Beijing Biodee Biotechnology Co., Ltd (Beijing, China) and Andre Pectin Ltd (Yantai, China), respectively.

All chemical reagents, including ethanol, hydrochloric acid, citric acid, sodium citrate, sodium azide, etc, used in the study were analytical grade and purchased from Lanyi reagent company (Beijing, China).

2.2. Apparatus

HHP extraction was carried out using a pressure-assisted thermal high hydrostatic pressurization unit (CAU-HHP-700-6, Baotou Kefa High Pressure Food Processing Inc., Inner Mongolia, China) with a cylindrical pressure chamber capacity of 7 L. Distilled water was used as the pressure-transmitting medium. The rate of pressure increase was about 130 MPa/min and the pressure release time was about 2 s.

HSHE extraction was carried out by an analytical system (JHBE-50, Jinnai Sci-tech Development Ltd, Zhengzhou, China) contains rotating cutter, high-speed motor, lifting system, control system and sample container. The rotation speed of the high-speed motor could be regulated in the range of 0–10,000 rpm by controlling the extraction voltage from 0 to 220 V.

2.3. Preparation of pectins

The pectin extracted by HHP, HSHE and THE were based on the methods of the previous investigations with slight modification (Guo et al., 2012; Kratchanova, Pavlova, & Panchev, 2004). Extraction solvent (pH = 1.24) was prepared by distilled water and 0.5 mol/L HCl, the solid to liquid ratio was 1:50. HHP extraction was carried out at the pressure of 500 MPa, temperature of 55 °C and the pressure-holding time of 10 min. THE extraction was carried out at 82 °C for 80 min using a reciprocating shaker bath. While, HSHE extraction was carried out with the extraction voltage of 156 V and extraction time of 240 s following a preheating process at 85 °C for 5 min. After extraction, the crude filtrate was precipitated using double volumes of 95% (v/v) ethanol and kept overnight without stirring at 4 °C. The precipitated pectin was then filtered by a filter cloth (400 meshes), and washed three times (5 min each time) using absolute alcohol to remove the monosaccharides, disaccharides and other impurities (Masmoudi et al., 2008). The

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