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The effect of high speed shearing on disaggregation and degradation of pectin from creeping fig seeds

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ABSTRACT

The effect of high speed shearing (HSS) on disaggregation and degradation of pectin from creeping fig seeds was investigated. It was found that disaggregation and degradation occurred during the whole shearing process. When pectin solution was sheared at 24,000 rpm for less than 8 h, degradation happened but disaggregation was dominant during this period. After 8 h, degradation became obvious, however, a small amount of aggregates remained even after 24 h treatment, indicating that HSS may not eliminate aggregates efficiently. The presence of aggregates is one of the most probable causes for the inaccurate determination of molecular weight of pectin. A new method was proposed for calculating more accurately the molecular weight based on the change of the reducing sugar content and the variation of molecular weight. Determination of unsaturated uronide and FT-IR spectra analysis indicated that neither β-elimination nor demethoxylation occurred during the HSS, and no new functional group was formed during the HSS process.

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1. Introduction

Pectin is a polysaccharide extracted from cell walls and middle lamella of plants. It has been extensively used in food and pharmaceutical industries. In the food industry, pectin is commonly used as a gelling agent, thickener, texturizer, emulsifier, and stabiliser to modify properties of food systems. One of the greatest difficulties in the characterisation of pectin is that pectins are highly prone to aggregation in solution, and problems have been identified in several studies on pectin size and conformation (Lopes da Silva & Rao, 2006). In addition, the presence of aggregates may be one of the most probable causes of inaccurate determination of its molecular weight. Considerable success in producing homogeneous solutions of pectin and similar materials has been reported using kinds of "physical" methods. Such techniques include microwave treatment (Ratcliffe, Williams, Viebke, & Meadows, 2005), sonication (Geresh, Adin, Yarmolinsky, & Karpasas, 2002), and the application of heat at an elevated pressure (Wang, Wood, Cui, & Ross-Murphy, 2001). Shearing is also an effective technique that can increase the energy of the component polymer chains and destroy the supramolecular aggregates. However, the effect of shearing for a short period is not enough to change the properties or the change is undetectable. On the other hand little is known about the effect of long time high speed shearing (HSS) on the disaggregation of pectin up to now.

When HSS is evaluated for its efficiency to reduce the aggregates, one inevitable concern is whether this method causes degradation of the polysaccharide, i.e. cleavage of glycosidic bonds in polymer chains. Ultrasonic treatment, homogenisation (including dynamic high pressure microfluidisation), and extrusion are frequently used food processing techniques. All of these processes have been reported to cause the degradation of pectin, and shearing stress was considered as a major mechanical force within these processes (Chen et al., 2012; Corredig & Wicker, 2001; Ralet & Thibault, 1994; Tiwari, Muthukumarappan, O'Donnell, & Cullen, 2010). Very few studies have evaluated the effect of shearing on the degradation of pectin. Mechanical shearing has been shown to be able to generate enough energy to disrupt the polymers. For example, amylopectin molecules were degraded under minimal shear conditions such as gentle agitation (Han & Lim, 2004), whereas according to Silvestri and Gabrielson (1991) a polymer may be mechanically degraded if the number of passes through a conventional capillary viscometer is sufficient. Distinguishing degradation from disaggregation is always an indispensable step when scientists evaluate the stability of polymers.

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Pectin isolated from creeping fig seeds (CFS) is low methoxylated and has high galacturonic acid content, molecular weight, and intrinsic viscosity (Liang et al., 2012). These properties would enable the effect of HSS to be detected easily. Therefore, CFS pectin was chosen in this study for investigating the effect of HSS up to 24 h on the elimination of aggregates and/or degradation of pectin. A new method based on the change of molecule weight and reducing sugar content induced by HSS was also proposed for the more accurate calculation of the molecule weight of those pectins containing a high number of large aggregates.

2. Theoretical considerations

It has been reported that the degradation reaction of a polymer by mechanical shearing follows a first-order reaction (Harrington & Zimm, 1965; Tsai, Tseng, Chang, Hsu, & Chen, 2010). A relationship between the reducing sugar content and the average molecular weight was deduced in our previous paper (Chen et al., 2012):

$$C_t - C_0 = (1/M_t - 1/M_0)K \quad (1)$$

where C_t and C_0 (mol L^{-1}) is the concentration of reducing sugars at time t and 0, respectively. M_t and M_0 (g mol^{-1}) is the average molecular weight of the polysaccharide at time t and time 0, respectively. K is rate constant (g L^{-1}).

In addition, Eq. (1) also equals to

$$C_{t2} - C_{t1} = (1/M_{t2} - 1/M_{t_1})K \quad (2)$$

where C_{t2} and C_{t1} is the concentration of the reducing sugars at time $t2$ and $t1$, respectively. M_{t2} and M_{t1} is the average molecular weight of the polysaccharides at the corresponding time. Therefore, if a series or at least one pair of accurate molecular weight and reducing sugars content can be obtained for the degraded samples where no aggregates or aggregates exist at negligible amount, K can be calculated from the linear relationship of Eq. (2). Then the Mw of other samples containing large aggregates can be more accurately calculated using Eqs. (1) and (2).

3. Materials and methods

3.1. Pectin preparation

The CFS pectin was extracted and purified according to the water extraction procedure used in our previous study (Liang et al., 2012). The galacturonic acid content and degree of methoxylation (DE) of CFS pectin was 87.65 ± 1.47% and 14.02 ± 1.30%, respectively.

3.2. High speed shearing (HSS) treatment

A 2 mg/mL CFS pectin solution was hydrated and completely dissolved in deionised water for 12 h with continuous mild magnetic stirring (400 rpm). Then, the solution was treated with Ultra-Turrax T25 disperser (IKA-Werke, Staufen, Germany) at 24,000 rpm for 0–24 h. This device consists of a rotor within a stationary stator, works at speed up to 25,000 rpm by using the rotor–stator principle, and permits the continuous operation for more than 24 h. Due to the high circumferential speed, the medium to be processed is drawn axially into the dispersion head and then forced radically through the slots in the rotor–stator arrangement. The high speed and minimal gap between the rotor and stator produce extremely strong shear forces (IKA, 2013). This equipment has been successfully used by Chen, Huang, Tsai, Tseng, and Hsu (2011) to study the degradation kinetics of chitosan induced by shearing treatments.

During shearing treatment, the solution was kept in ice water bath to eliminate the effect of temperature. Then, samples were taken every 4 h and subsequently analysed for intrinsic viscosity, particle size, molecular weight, and reducing sugars content. Parts of the solution were lyophilised for Fourier transform infra-red (FT-IR) and scanning electron microscopy analysis. The solution without shearing treatment was used as a negative control in the study.

3.3. Determination of intrinsic viscosity

The intrinsic viscosity ($[\eta]$) of the pectin solutions treated and untreated by HSS was measured at 25.0 ± 0.1 °C, using an Ubbelohde dilution viscometer (diameter = 0.52 mm), which was suspended in a thermostatic water bath under precise temperature control. Four millilitres of pectin solution were applied to this test. The sample was manually diluted with solvent after generating at least three flow time readings at each concentration. The intrinsic viscosity ($[\eta]$) was estimated by plotting η_{sp}/c against c according to Huggins' equation, and extrapolation of the curves to "zero" concentration.

$$\frac{\eta_{sp}}{c} = [\eta] + K_1[\eta]^2 c \quad (3)$$

where c is the concentration, K_1 is Huggins constant, and η_{sp} is specific viscosity.

3.4. Determination of particle size and its distribution

Dynamic light scattering (DLS) has been reported to be an effective approach to study the aggregation behaviour of macromolecules in dilute solutions (Li, Wang, Cui, Huang, & Kakuda, 2006). The DLS determinations of pectin solutions were performed using a laser particle size analyzer (Nicomp 380 ZLS, PSS Co., Santa Barbara, USA). The solutions were diluted to a concentration of 0.5 mg/mL with deionised water, and all measurements were carried out at 25 °C (Chen et al., 2012).

3.5. Determination of molecular weights

The molecular weight of pectin samples was determined by a high performance size exclusion chromatography (HPSEC) system (Chen et al., 2012). The system consists of an Agilent 1200 pump unit, an automatic injector (Agilent Technologies, Waldbroon, Germany), a refractive index (RI) detector (Brookhaven Inc., New York, USA), and a linear mix column with a guard column. The columns were maintained at 40 °C. Pectin solutions were diluted to 0.5 mg/mL, and then filtered through 0.45 μm filters before injection. A solution of 0.05 M $NaNO_3$ containing 0.02% NaN_3 was used as mobile phase, while the elution rate was 0.7 mL/min. Dextrans of T-10, T-40, T-70, T-150, T-500, T-1000, and T-2000 were used as standards to construct a standard curve.

3.6. Determination of reducing sugar content

The reducing sugars were measured following a modified 3,5-dinitrosalicylic acid (DNS) assay described by Miller (1959). Briefly, 1.5 mL of the DNS reagent consisting of 3,5-dinitrosalicylic acid (6.5 g), sodium hydroxide (20 g), sodium sulphite (5 g), phenol (5 g) and Rochelle salt (185 g) in 1000 mL of distiled water, was added to 2 mL of the sample. The mixture was heated for 5 min accurately, and adjusted to a final volume of 10 mL. The absorbance of the mixing solution at 540 nm was determined with UV–Vis spectrophotometer (UV-2500, Shimadzu, Kyoto, Japan), using galacturonic acid to create the calibration curve. The analysis was carried out in triplicate.

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