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Application of digital image analysis method to study the gelatinization process of starch/ sodium chloride solution systems

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ABSTRACT

The digital image analysis, integral optical density (IOD) method, combined with “the model of response difference of crystallite change (MRDCC)” and “the model of gelatinization process (GP)”was applied to further study the gelatinization of corn starch, potato starch, and pea starch in different concentrations of NaCl solutions. It was found that the addition of non-aqueous media not only affected the gelatinization temperature, but also impacted on the whole gelatinization process and effected differently at different stages of gelatinization. NaCl solution of 1, 3 and 4 mol/L (M) rose the GP curve of corn starch while 2 M had little effects on it; for potato starch, it seemed that NaCl gave little efforts on GP curve while 1 M rose the GP curve at the relative biggest degree; for pea starch, it performed two stage gelatinization process in different concentration of NaCl solution, this may due to the resultant force of heat, plasticizer, solute and expansion power and more studies were needed to reveal the gelatinization mechanism. Compared with the loss of crystalline structure (long-ordered structure), the disappearance of the molecular helix-coil structure (short-ordered structure) contributed greater to the DSC enthalpy, which could be deduced from the GP model.

1. Introduction

Gelatinization is one of the most important properties of starch and the most common use of natural phenomena in our daily life and production. During the industrial application, starches will be in a variety of medium systems, including various salts, sugars, proteins and lipids, etc. Starch granules are subjected to different thermal conditions and other unit operations that result in granules with differing stages of partial and total gelatinization; these collectively influence the physicochemical properties of the products (Ratnayake & Jackson, 2006).

Gelatinization is the phase transition process of starch under the common function of thermoplastic agent and heat; meanwhile, different source of starch experiences huge difference in the phase transition process. Therefore, the theory based on a specific starch may not be able to applied to other types of starch; it is impossible to explain the phase transition process of different starch with a general theory. The real mechanism of starch phase transition is determined by the combination of starch type and solvent, as a result, understanding the effects of various medium are particularly important for food processing and similar operations where starch is subjected to various hydrothermal conditions for long periods during heat processing or cooking (Ratnayake, Otani, & Jackson, 2009).

The effect of sodium chloride (NaCl) on starch gelatinization has been studied by many researchers using a wide range of techniques. For example, light microscopy (Rumpold & Knorr, 2005), differential scanning calorimetry (Ahmed, 2012; Gonera & Cornillon, 2002; Jane, 1993), rheological measurements (Ahmed, 2012), scanning electron microscopy (Koch & Jane, 2000), dielectric measurements (Bircan & Barringer, 1998), light transmission (Sandstedt, Kempf, & Abbott, 1960), RVA (Moreira, Chenlo, & Torres, 2011), and NMR spectroscopy (Chiotelli, Pilosio, & Le Meste, 2001; Gonera et al., 2002; Jane, 1993), With different parameters, these methods can characterize the effect of salts on starch gelatinization properties from different perspectives. Based on polarizing microscopy, the IOD method was of advantage compared with the previous traditional methods, such as counting the particle number and calculating polarization area methods, because it was the product of two parameters: optical density and area, which would be a response of both light intensity and area of birefringence light. Therefore, the gelatinization degree

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(DG) of the starch granule especially together with the partially gelatinized zone could be detected and quantitatively analyzed. The IOD method could dynamically measure starch crystallization, truly and accurately reflecting the state of the starch gelatinization process under a specific temperature with higher accuracy and sensitivity. (Li, Xie, Yu, & Gao, 2013) The model of response difference of crystallite change (MRDCC) was a new characterization of the crystallization change degree in the starch gelatinization process. It characterizes that the starch gelatinization speed change with the temperature (Li et al., 2013). Compared with the result of DSC technique, MRDCC is more sensitive and accurate due to it could detect the subtle expansion in the pre-gelatinized stage. It seems that the single peak in DSC method was corresponded to the combination of crystalline helix–helix dissociation and the reduction of the molecule helix-coil transition, while the gelatinization degree measured by IOD method mainly corresponded to the helix–helix dissociation.

Salts have been shown to have a significant effect on the gelatinization and rheological properties of starches generally, and it has been found that they could cause an elevation or depression of the gelatinization temperature, T_p (Evans & Haisman, 1982; Paredes-Lopez & Hernández-Löpez, 2006; Rumpold et al., 2005; Wootton & Bamunuarachchi, 1979)and gelatinization enthalpy $\triangle H$ (Evans et al., 1982; Jane, 1993; Maaurf, Che Man, Asbi, Junainah, & Kennedy, 2001; Wootton et al., 1979), and similarly might increase or decrease the rate of gelatinization degree (Katsuta, 1998). Jane (Jane, 1993) reported that the mechanism of starch gelatinization in salt solutions could be attributed to: (1) structure-making and structure-breaking effects on water and (2) electrostatic interactions between salts and hydroxyl groups of starch. Finally, a plasticizing effect of salt has also been envisaged. According to the observation of Ahmad (Ahmad & Williams, 1999), it could be divided into two factor to understand the effect of salts on starch gelatinization: (1) the effect of salts on polymer–solvent interactions, which are influenced to a greater extent by the anions; (2) the interaction of cations with the hydroxyl groups of the starch molecules to form complexes that disrupt polymer chain aggregation. On the one hand, ions of high charge density (e.g., Ca^{+2},$SO_4{}^{-2}$) have strong electrostatic interactions with water molecules (structure-makers), and thus reduce the fraction of free water and increase the viscosity of the solution. On the other hand, ions with low charge densities (e.g., I^-, SCN^-, $IO_4{}^-$), known as structure-breakers, decrease the viscosity of the aqueous solution by breaking or weakening hydrogen bonds between water molecules, and thus increase the fraction of free water (Chiotelli et al., 2001). Because of the electronegative nature of starch (Donnan potential), (Oosten, 1982) anions tend to repel starch –OH groups and stabilize starch granules. Cations, on the other hand, attract starch –OH groups and destabilize starch granules. The repulsion or attraction is proportional to the charge density of the ion. At a given salt concentration, the driving force of the anions exceeds the repulsing force of the Donnan potential and anions enter the granule and start the gelatinization process by rupturing the hydrogen bonds between the starch molecules (Chiotelli et al., 2001).

Nevertheless, the underling mechanisms implicated in gelatinization of starch in the presence of salts are still not well understood; and the theory based on a specific starch may not be able to fully applied to other types of starch. The objective of this investigation was, therefore, to undertake a comprehensive study of the effect of NaCl on the gelatinization of corn (A-type), potato (B-type) and pea (C-type) starch, by combining information from hot-stage light microscopy and differential scanning calorimetry; meanwhile, the digital image analysis technique, integral optical density (IOD) method is employed in this work.

2. Materials and methods

2.1. Materials

Native starches used in this study were of food grade. Corn starch (type A) was purchased from Guangdong Pengjin Industrial Ltd (Guangdong, China), content: corn starch84% (protein 0.3%, ash 0.3%, lipid 0.87%, amylose 26.8%, phosphate 0.02%). Potato starch (type B) was obtained from Haotian Group Ltd (Jiangsu, China), content: potato starch81% (protein 0.1%, ash 0.35%, lipid 0.1%, amylose 21%, phosphate 0.08%). Pea starch (type C) was purchased from Yantai Oriental Protein Ltd (Shandong, China), content: pea starch86% (protein 10.4%, ash 2.5%, lipid 1.5%, amylose 43%, phosphate 0.03%). Chemicals, NaCl and solvents used in this work were of analytical grade.

2.2. Hot stage-light microscopy

1,2,3 and 4 mol/L (M) of NaCl solution was configured under 25 °C. NaCl-starch dispersions (starch: distilled water in NaCl solution = 1:4, dry basis) equilibrated for 2 h were sealed between two glass cover slip using Dow Corning 732 Sealant before replaced in the hot stage (model THMS600, Linkam, Britain).

Each specimen in the hot stage were observed under a polarization microscope (model BHS-2, Olympus Vanox, Japan) equipped with a digital camera, which can display live video of birefringence granules in a real time. A temperature programmer was connected with the hot stage to control the heating progress from 40 °C to 80 °C at a rate of 2 °C/min. Live pictures were captured every 5 °C when blow 60 °C, while 2 °C above 60 °C. Each image (2048 × 1536 pixels) was saved as 12bits TIFF image file, without compression.

2.3. Differential scanning calorimeter (DSC)

Gelatinization parameters of starch were detected using a Perkin-Elmer DSC 8000 (Norwalk, CT, USA) equipped with a refrigerated cooling system and Pyris™ operation software (Perkin-Elmer). Melting point and enthalpies of indium were used for temperature and heat capacity calibration.

The ratio of starch to distilled water and heating scan program was made the same as it in the hot stage experiment. NaCl-starch slurry (starch: distilled water in NaCl solution = 1:4, dry basis) equilibrated for 2 h. The nitrogen access speed was 20 mL/s. The spectrum of starch gelatinization enthalpy was analyzed by the Pyris Software accompanied with the DSC instrument. DSC measurement of each specimen has been performed in triplicate, and the results are presented as the mean.

2.4. Degree of gelatinization (DG)

(1) IOD method:

The IOD value of each digital picture was calculated by the Image-pro plus 5.0 software as described by Li et al. (2013).

The DG based on the IOD value (DG_I) was calculated as defined by Qian Li:

$$\text{Background correction}: \quad C = A - B \tag{1}$$

$$DG_I\% = (1 - C/C_0) \times 100\% \tag{2}$$

Where A is the original IOD value (IOD value calculated from the original digital image), B is the background IOD value (IOD value calculated from the original digital image when all of the birefringence disappeared), and C_0 is the initial real IOD value (IOD value of