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Properties of lotus seed starch–glycerin monostearin complexes formed by high pressure homogenization

Bingyan Chen, Shaoxiao Zeng, Hongliang Zeng, Zebin Guo, Yi Zhang, Baodong Zheng *

College of Food Science, Fujian Agriculture and Forestry University, Fuzhou, Fujian 350002, China
Fujian Provincial Key Laboratory of Quality Science and Processing Technology in Special Starch, Fujian Agriculture and Forestry University, Fuzhou 350002, China

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ABSTRACT

Starch-lipid complexes were prepared using lotus seed starch (LS) and glycerin monostearate (GMS) via a high pressure homogenization (HPH) process, and the effect of HPH on the physicochemical properties of LS-GMS complexes was investigated. The results of Fourier transform infrared spectroscopy and complex index analysis showed that LS-GMS complexes were formed at 40 MPa by HPH and the complex index increased with the increase of homogenization pressure. Scanning electron microscopy displayed LS-GMS complexes present more nest-shape structure with increasing homogenization pressure. X-ray diffraction and differential scanning calorimetry results revealed that V-type crystalline polymorph was formed between LS and GMS, with higher homogenization pressure producing an increasingly stable complex. LS-GMS complex inhibited starch granules swelling, solubility and pasting development, which further reduced peak and breakdown viscosity. During storage, LS-GMS complexes prepared by 70–100 MPa had higher Avrami exponent values and lower recrystallization rates compared with native starch, which suggested a lower retrogradation trendency.

1. Introduction

Starch is an abundant, inexpensive, renewable, and fully biodegradable natural material and is the major form of carbohydrate storage in plants. It is generally composed of essentially linear amylose and highly branched amylopectin with α-D-glucopyranose as the structural unit (Ann-Charlotte Eliasson, 2004). Amylose is well known to form inclusion complexes with small ligands such as fatty acids, alcohols and aromatic compounds, and this has been ascribed to a conformational change from a coiled to a single left-handed helical structure (Rodríguez & Bernik, 2014). Amylose helices can then pack together to form a crystalline V-type structure. The V-amylose complex has been suggested to play an important role in modifying the functionality of starchy food, reportedly retards retrogradation (Tufvesson, Skrabanja, Björck, Elmståhl, & Eliasson, 2001), changes the rheological behaviour (Shah, Zhang, Hamaker, & Campanella, 2011), lowers the digestion rate of starch (Mei, Zhou, Jin, Xu, & Chen, 2015), and even to create a new delivery system to protect volatile and sensitive ligands, such as phenols and unsaturated fatty acids (Karunaratne & Fan, 2016; Zabar, Lesmes, Katz, Shimoni, & Bianco-Peled, 2010). Changes in functionality are of interest to the food industry and for human nutrition. Although interactions between amylopectin and lipids have been investigated, only a small proportion of amylopectin forms V-type complexes and this is dependent on short branch chain length and steric hindrance (Heinemann, Escher, & Conde-Petit, 2003).

To date, various methods have been employed to form starch-lipid complexes, and their properties differ depending on the methods used. In the dimethyl sulphoxide (DMSO)-based synthesis method, a high complex index was obtained due to modifications to starch that took place in the non-heterogeneous system. However, organic solvents are volatile, which limits their applications in food industrial processes. When lipid and starch dispersion are heated in water-based synthesis systems, complexes are formed as soon as starch begins to gelatinize. (Fanta, Kenar, & Felker, 2015) reported that steam jet-cooking resulted in the formation of corn starch-fatty acid spherulites at different operating conditions, the size and morphology of which depends on the fatty acids used and the cooling rate. During extrusion cooking, V-type inclusion complexes are formed between flours and both saturated and unsaturated fatty acids, the formation of complexes by extrusion cooking is strongly influenced by feed moisture (De Pilli, Derossi, Talja, Jouppila, & Severini, 2011). A greater number of pure amylose-fatty acid complexes are formed in excess water during high hydrostatic pressure treatments due to starch gelatinization

* Corresponding author at: College of Food Science, Fujian Agriculture and Forestry University, Fuzhou 350002, China.
E-mail address: zbdfst@163.com (B. Zheng).

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at room temperature (Le Bail et al., 2013). However, marked differences in resistance to high pressure can occur in different starch samples from various botanical sources. For example, B-type starch is more resistant to pressure than A-type starch, and B-type complex maybe formed at higher pressures and after longer exposure times that are economically unviable. In recent years, high-pressure homogenization (HPH) technology has been increasing applied to the field of starch-lipid complex research. This green technology is also economically viable for starch modification. During the HPH process, intense mechanical effects such as high pressure can degrade the starch polymers to release more amylose. Simultaneously, HPH facilitates the rapid dispersion of lipids with low water solubility and enhances the chances of reaction between lipids and amylose (Meng, Ma, Cui, & Sun, 2014). Some studies have reported the formation of starch-lipid complexes from regular, waxy, high-amylose corn starch following HPH-treatment (Kenar, Compton, Little, & Peterson, 2015; Lesmes, Barchechath, & Shimoni, 2008). However, there is little information on the gelatinization and retrogradation properties of starch-lipid complex formed by HPH-treatment.

Lotus (*Nelumbo nucifera Gaertn.*), a perennial aquatic plant, is widely cultivated in China, India, South Korea and Japan (Kim & Shin, 2012). Lotus seed is an excellent source of carbohydrate, which is used in the production of traditional confectionery products and food additives (Geng, Chen, & Yimin, 2008). In our previous study, the starch content of lotus seed was ~500 g/kg starch and the amylose content was relatively high at 40%, (w/w) starch (Zeng, Zheng, Lin, & Zhuo, 2009). Lotus seed starch is therefore suitable for starch-lipid complexation. Currently, no studies have focused on the physicochemical properties of lotus seed starch-lipid complex, especially in gelatinization and retrogradation.

Therefore, the objective of this study was to investigate the physicochemical properties of starch-lipid complexes, which were prepared using lotus seed starch (LS) and glycerin monostearate (GMS) via a high pressure homogenization (HPH) process. The physicochemical properties were characterized by morphology, crystalline structure and thermal stability. Furthermore, swell power, solubility, pasting and retragradation properties of LS-GMS complexes were studied and compared.

2. Materials and methods

2.1. Materials

Lotus seed starch (Green Field Fujian Food Co., Ltd., Fujian, China) was isolated as previously described (Zhang, Zeng, Wang, Zeng, & Zheng, 2014). Lotus seed starch contained, on average, 9.25% (d.b) moisture, 0.37% (d.b) lipid, 0.28% (d.b) protein and 0.3% (d.b) ash. The amylose/amylopectin content of lotus seed starch was 40 /60, as measured using a amylose/amylopectin assay kit purchased from Nanjing chemical Co. (Nanjing, China). All other chemical reagents used in this study were of analytical grade.

2.2. Preparation of starch–glycerin monostearin complexes

The raw starch (25 g) was suspended in 500 mL ethanol solution to completely remove organic-soluble components, especially lipids, and was then washed several times with distilled water until the starch was free of colour. The defatted starch was dried in an air oven at 40 ± 1 °C for 16 h. Glycerin monostearin (5% weight based on defatted lotus seed starch) was added to the defatted starch slurry (25 g, 5%, w/w), mixed in a water bath at 40 °C with constant stirring for 5 min, then homogenized in a high-pressure homogenizer (GYB 40-10S, Donghua Technology Co., Ltd., Shanghai, China) at 20–100 MPa for 20 min. Simultaneously, a Cooling water circulating device was used to prevent the temperature rise of starch slurry in homogenization process. The homogenized dispersion was allowed to cool without stirring at room temperature. Samples were collected by centrifugation (4500*g*, 10 min) and washed twice with a 50% water ethanol mixture to move uncomplexed GMS. The final precipitates (LS-GMS) were freeze-dried and ground with a laboratory-scale grinder to pass through a 100-mesh sieve.

2.3. Determination of the complex index (CI)

CI was determined in order to investigate the extent of complex formation as previously described (Kawai, Takato, Sasaki, & Kajiwara, 2012). Approximately 0.3 g of starch sample was accurately weighted and suspended in 5 mL of water, and the dispersion was heated in a boiling water bath for 20 min. After cooling, the dispersion was centrifuged (3000*g*, 10 min) and 50 μL of the supernatant was mixed with 4 mL of iodine solution (0.1% (w/w) I_2 and 2% (w/w) KI in deionized water) in a 10 mL test tube that was gently vortexed. Absorbance (ABS) values of samples and controls lacking GMS were measured at 690 nm with a UV–VIS spectrophotometer. CI was calculated using the following equation:

$$CI\ (\%) = 100 \times (ABS_{\text{control}} - ABS_{\text{sample}})/ABS_{\text{control}}$$

2.4. Fourier transformed infrared (FTIR) spectroscopy

FTIR spectra of LS-GMS samples were obtained using a Tensor 27 FTIR spectrometer (Bruker, Karlsruhe, Germany). Spectra were scanned at room temperature between 4000–400 cm^{-1} with an accumulation of 32 scans and a resolution of 4 cm^{-1}.

2.5. Scanning electron microscopy (SEM)

SEM images of LS-GMS samples and controls lacking GMS were obtained using a field emission scanning electron microscope (JSM-6360LV, JEOL, Tokyo, Japan) in low vacuum mode at an accelerating voltage of 20 keV. The approximate diameter of complex was measured from the SEM images.

2.6. Wide angle X-ray diffraction (XRD)

LS-GMS sample powers were performed with a D/MAX 2200PC X-ray diffractometer (Rigaku Corporation, Tokyo, Japan) operating at 40 kV and 200 mA with Cu kα radiation ($k = 1.5406$ Å). X-ray diffraction patterns were obtained between 0–35° 2θ at a scanning speed of 8°/min. The relative crystallinity was calculated as the area ratio of the crystalline sharp peak over the total area using peak-fitting software (Origin-version 8.1, Microcal Inc., Northampton, MA, USA), and was calculated using the following equation:

$$Relative\ crystallinity\ (\%) = 100 \times A_C/(A_C + A_a)$$

where A_c is the crystallinity area observed by X-ray diffraction and A_a is the amorphous area.

2.7. Differential scanning calorimetry (DSC)

Thermal properties of LS-GMS samples were analyzed using a differential scanning calorimeter (DSC-200FC, NETZSCH, Selb, Germany). LS-GMS samples (2 mg) were mixed with 5 μL of deionized water and hermetically sealed in a stainless steel pan. After equilibrating for 12 h at room temperature, the samples were scanned between 20–150 °C at a rate of 10 °C/min. Calculated values for onset temperature (T_o), peak temperature (T_p), end temperature (T_e) and enthalpy (ΔH) were recorded from the LS-GMS melting enthalpy.