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Effect of single and dual hydrothermal treatments on the crystalline structure, thermal properties, and nutritional fractions of pea, lentil, and navy bean starches

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ABSTRACT

Pea, lentil and navy bean starches were annealed at 50 °C (70% moisture) for 24 h and heat-moisture treated at 120 °C (30% moisture) for 24 h. These starches were also modified by a combination of annealing (ANN) and heat-moisture treatment (HMT). The impact of single and dual modifications (ANN–HMT and HMT–ANN) on the crystalline structure, thermal properties, and the amounts of rapidly digestible starch (RDS), slowly digestible starch (SDS), and resistant starch (RS) were investigated. Birefringence remained unchanged on ANN but decreased on HMT. Granular swelling and amylose leaching decreased on ANN and HMT. Relative crystallinity, gelatinization enthalpy, and short-range order on the granule surface increased on ANN but decreased on HMT. Gelatinization transition temperatures increased on ANN and HMT. Gelatinization temperature range decreased and increased on ANN and HMT, respectively. ANN and HMT increased SDS and decreased RS levels in all starches. However, RDS levels increased on ANN and HMT in pea and lentil starches but decreased in navy bean starch. In gelatinized starches, ANN and HMT decreased RDS level and increased SDS and RS levels. Changes to crystalline structure, thermal properties and amounts of RDS, SDS, and RS were modified further on ANN–HMT and HMT–ANN.
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1. Introduction

Pulses are the edible seeds of certain leguminous plants that include pea (*Pisum sativum* L.), lentil (*Lens culinaris* L.), chickpea (*Cicer arietinum* L.), and navy bean (*Phaseolus vulgaris* L.). Pulses are rich in starch, protein and dietary fiber with significant amounts of vitamins and minerals (Almeida Costa, Queiroz-Monici, Machado Reis, & Oliveira, 2006; Tharanathan & Mahadevamma, 2003). The poor starch digestibility of pulses has been attributed to intact cell-wall structures enclosing starch granules, the presence of various antinutrients such as amylase inhibitors, phytates and polyphenolics, relatively high levels of amylose, and high content of viscous soluble dietary fibre components (Bravo, Siddhuraju, & Saura-Calixto, 1998; Hoover & Zhou, 2003; Tharanathan & Mahadevamma, 2003). Based on the rate of glucose release and its absorption in the gastrointestinal tract, starch is classified into rapidly digestible starch (RDS), slowly digestible starch (SDS), and resistant starch (RS) (Englyst, Kingman, & Cummings, 1992). RDS is the starch fraction that causes a sudden increase in blood glucose level after ingestion, and SDS is the starch fraction that is digested more slowly but completely in the small intestine. RS has been defined as the starch portion that cannot be digested in the small intestine, but is fermented in the large intestine. Pulses have been shown to contain significant amounts of SDS and RS (Chung et al., 2008; Sandhu & Lim, 2008).

Annealing (ANN) and heat-moisture treatment (HMT) modify physicochemical properties of starch, without destroying its granular structure. Both ANN and HMT involve incubation of starch granule in excess water (<65% w/w) or intermediate water (40–55% w/w) and at low moisture levels (<35% w/w), respectively, during a certain time period, at a temperature below the onset temperature of gelatinization but over the glass transition temperature (Jacobs & Delcour, 1998; Hoover & Vasanthan, 1994a, 1994b; Tester & Debon, 2000). Numerous studies have been carried out on the impact of ANN and HMT on starch physicochemical properties (Chung, Liu et al., 2009; Gunaratne & Hoover, 2002; Hoover & Manuel, 1996a, 1996b; Hoover & Vasanthan, 1994a, 1994b; Jacobs & Delcour, 1998; Jayakody & Hoover, 2008; Lan et al., 2008; Tester & Debon, 2000; Vermeylen, Goderis, & Delcour, 2006). The above studies have shown that ANN and HMT result in structural changes within the amorphous and crystalline regions to different extents, which in turn influence granular swelling, amylose leaching, pasting properties, gelatinization parameters, molecular structure, crystalline structure, and susceptibility towards enzyme and acid. However, only a few studies have reported the effect of the combination of HMT and ANN on starch structure and properties (Chung, Hoover et al., 2009; Stute, 1992). Our previous study (Chung, Hoover et al., 2009) showed that the molecular structure and physicochemical properties of corn starch (A-type unit cell)

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could be modified to varying extents by hydrothermal treatment. The extent of this modification was most pronounced on HMT, and when ANN starch was subjected to HMT (ANN–HMT). This implied that crystallite disruption on HMT has a greater impact on corn starch properties than crystalline perfection on ANN. Stute (1992) also studied the impact of ANN–HMT and HMT–ANN on the X-ray pattern, gelatinization, properties and sorption isotherm of potato starch (B-type unit cell). The results showed that structural alterations on ANN were reversible when ANN followed HMT or HMT followed ANN. The impact of ANN (Hoover & Manuel, 1996b) and HMT (Hoover & Vasanthan, 1994b) on pulse starches (C-type unit cell) showed that the extent of changes to starch structure and properties was different to that observed with starches containing pure A or B-type unit cells. This was attributed to the presence of higher amylose content (30–65%), C-type (mixed A and B unit cells) crystalline structure, and the presence of only trace quantities of internal lipids. It has been shown that in pea starch, the B-type unit cells are located mainly in the central part of the granule and are surrounded by the A-type unit cells (Bogracheva, Morris, Ring, & Hedley, 1998; Buleon, Gerard, Riekel, Vuong, & Chanzy, 1998). Zhou, Hoover, and Liu (2004) showed that the amount of B-type unit cells varies among pulse starches. Thus, the objective of this study was to investigate to what extent changes to crystalline structure, properties and nutritional fractions of pea, lentil and navy bean starches on ANN, HMT, HMT–ANN and ANN–HMT are influenced by amylose content, amylopectin structure, and B-type polymorphic composition.

2. Materials and methods

2.1. Materials

Pea (*P. sativum* L.) cultivar (*1544-8*, yellow cotyledon) and lentil (*L. culinaris* L.) cultivar (*CDC Plato*, yellow cotyledon) were obtained from the Crop Development Centre, University of Saskatchewan, Canada. Navy bean (*P. vulgaris* L.) cultivar (*AC Compass*) was provided by the Department of Plant Agriculture, University of Guelph, Canada. Pulse seeds were ground to flour using a cyclone mill (A10 analytical mill, Tekmar Co., Cincinnati, OH) and passed through a screen with 125 μm openings. Pea, lentil, and navy bean starches were isolated from milled flours using the procedure of the Chung et al. (2008).

2.2. Hydrothermal treatment

For annealing, native pulse starch slurries (70% moisture) were incubated at 50 °C for 24 h in a water bath. At the end of the incubation period, samples were centrifuged (2000 g) for 10 min and supernatant was decanted. The annealed starches were washed once with deionized water and air dried at 40 °C. For heat-moisture treatment, starch samples were weighed into glass containers (125 ml). The moisture content of starch was adjusted to 30% by adding the appropriate amount of distilled water. The containers were sealed, kept for 24 h at ambient temperature, and then placed in a forced air oven at 120 °C for 24 h. Afterwards the containers were opened, and the starch samples were air-dried to uniform moisture content (~10%). For dual modification, annealed starch samples were subjected to heat-moisture treatment (ANN–HMT) and heat-moisture treated starch samples were subjected to annealing (HMT–ANN).

2.3. Apparent amylose content and amylopectin chain length distribution

Apparent amylose content of native pulse (pea, lentil and navy bean) starches was determined by a colorimetric method (Williams, Kuzina, & Hlynka, 1970). Isoamylase debranching of whole starch accompanied by high performance anion exchange chromatography with pulsed amperometric detection (HPAEC-PAD) was used to determine the branch chain length distribution of native pulse starches (Liu, Gu, Donner, Tetlow, & Emes, 2007).

2.4. Polarized light microscopy

Birefringence of native and modified pulse starch granules was observed under polarized light with a binocular microscope (DME, Leica Canada, Mississauga, ON, Canada) equipped with real time viewing (Micropublisher 6.0, QImaging, Surrey, BC, Canada). The images were recorded at the same magnification (400×) for all starch samples (1.0% starch suspension).

2.5. Swelling factor (SF) and amylose leaching (AML)

SF and AML of native and modified pulse starches when heated at 80 °C in excess water were measured according to the method of Tester and Morrison (1990), and Chung et al. (2008), respectively. SF was reported as the ratio of the volume of swollen granules to the volume of the dry starch and AML was expressed as percentage of amylose leached per 100 g of dry starch.

2.6. X-ray diffraction

X-ray diffraction analysis of native and modified pulse starches (~10% moisture content) was conducted using a Rigaku RPT 300 PC X-ray diffractometer (Rigaku-Denki Co., Tokyo, Japan) operated at 40 kV and 100 mA with scanning range of 3–35° and scan speed of 2.0°/min. The degree of relative crystallinity was quantitatively estimated following the method described by Nara and Komiya (1983) using the Origin 6.0 software (Microcal Inc., Northampton, MA). The 'B' polymorph content was calculated using the method described by Zhou et al. (2004).

2.7. Fourier transform infrared (FT-IR) spectroscopy

FT-IR spectra of native and modified pulse starches were obtained with a Digilab FTS 7000 spectrometer (Digilab USA, Randolph, MA) equipped with a thermoelectrically cooled deuterated triglycine sulfate (DTGS) detector using an attenuated total reflectance (ATR) mode. For each spectrum, 128 scans were recorded at room temperature at a resolution of 4 cm^{-1}. Spectra were baseline-corrected and then deconvoluted over the range of 1200–800 cm^{-1}. A half-band width of 15 cm^{-1} and a resolution enhancement factor of 1.5 with Bessel apodization were used. The amplitudes of absorbance for each spectrum at 1022 and 1047 cm^{-1} were noted and the ratio of 1047 cm^{-1}/1022 cm^{-1} was calculated per sample to estimate the degree of order of starch granules at the surface (Sevenou, Hill, Farhat, & Mitchell, 2002).

2.8. Differential scanning calorimetry (DSC)

Gelatinization characteristics of native and modified pulse starches were measured by a differential scanning calorimeter (2920 Modulated DSC, TA Instruments, New Castle, DE). Starch (12 mg, db) was placed in a high-volume pan and distilled water (28 μl) was added with a microsyringe. The sample pan was sealed, equilibrated at room temperature for 12 h, and then heated from 5 to 180 °C at a heating rate of 10 °C/min. An empty pan was used as a reference. The onset (T_o), peak (T_p), and conclusion (T_c) temperatures and enthalpy of the gelatinization endotherm (ΔH) were determined. Gelatinization temperature range (T_c–T_o) was calculated as the temperature difference between T_c and T_o.

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