Food Hydrocolloids 74 (2018) 349–357

Contents lists available at ScienceDirect

Food Hydrocolloids

journal homepage: www.elsevier.com/locate/foodhyd

Small-granule starches from sweet corn and cow cockle: Physical properties and amylopectin branching pattern

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A R T I C L E I N F O

Article history:
Received 3 July 2017
Received in revised form
21 August 2017
Accepted 22 August 2017
Available online 29 August 2017

Keywords:
Small-granule starch
Sweet corn starch
Cow cockle starch
Physical properties
Amylopectin branching

A B S T R A C T

Small-granule starches (SGS) attract growing interests with their unique structure and functionalities, such as fat replacement and texture modifications. To explore new resources of SGS, sweet corn starch (SCS) and cow cockle starch (CCS) were studied for their physical properties and branching pattern, with normal corn starch, waxy corn starch, normal rice starch, and waxy rice starch as comparisons. Hydrothermal and wide-angle X-ray powder diffraction analyses showed the much lower content of ordered structure (melting enthalpy of 5.6 J/g) and lower crystallinity (28%) for SCS than for normal corn starch (14.4 J/g and 34%, respectively). The rapid viscosity analyzer analysis showed very low viscosity for SCS through the pasting process and high resistance of CCS against the shear force. The results of β-limit dextrin structure analyses showed highly branched clusters of SCS (about 15 glucan chains per cluster) and a lack of long inter-cluster chains that connect 3 or more clusters. In contrast, CCS displayed a branching pattern similar to that of corn starches, however, with fewer short intra-cluster chains. It was considered that the low crystallinity and viscosity of SCS were attributed to its high branching, which reduced the formation of crystalline structure during the genesis of starch and decreased the interactions among swollen granules. On the other hand, the submicron size made CCS granules less susceptible to the shear force applied during pasting, leading to negligible breakdown. Using SCS and CCS as models, this study connected the physical properties and branching pattern of SGS, thus supporting their potential applications.

1. Introduction

Starch is an important ingredient in the food industry due to its multiple functionalities in food systems. Starch functionalities are closely associated with the size, composition, and glucan structure of individual granules. In general, the granular size of starches from rice, amaranth, and cow cockle are much smaller than that of maize, potato, and wheat (Lindeboom, Chang, & Tyler, 2004). In recent years, small-granule starches (SGSs) have attracted growing attentions due to their potentials in providing smooth texture (Wani et al., 2012), fat replacement (Malinski, Daniel, Zhang, & Whistler, 2003), and controlled delivery (Gonzalez-Soto, de la Vega, García-Suarez, Agama-Acevedo, & Bello-Pérez, 2011).

Cow cockle starch (CCS) exists in the seeds of cow cockle (*Saponaria vaccaria* L.), an annual weed found in United States and Canada (Goering, Eslick, Watson, & Keng, 1966). Recently, efforts have been made to cultivate cow cockle as an industrial crop in North America (Willenborg & Johnson, 2013). Starch isolated from cow cockle seeds showed homogeneously small granules (~0.3–1.5 μm) (Biliaderis, Mazza, & Przybylski, 1993), possibly the smallest granules that have been reported. However, only limited information is available for CCS, such as its granular stability, low pasting temperature, and low setback upon gelatinization and pasting (Biliaderis et al., 1993; Goering & DeHaas, 1972). Its unique property is likely associated with the submicron granules (Biliaderis et al., 1993). Furthermore, there is still a substantial lack of knowledge on the branching structure of CCS.

Phytoglycogen and starch are two primary carbohydrate polymers in *sugary-1* maize mutant (James, Robertson, & Myers, 1995), a traditional sweet corn. Previous studies have shown the small granular size of sweet corn starch (SCS) (Jane, Kasemsuwan, Leas, Zobel, & Robyt, 1994; Wang, White, Pollak, & Jane, 1993). Early studies on SCS revealed its high amylose content and several

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http://dx.doi.org/10.1016/j.foodhyd.2017.08.025

Abbreviations

ANBPC	Average number of branches per cluster
AM	Amylose
AP	Amylopectin
ASG	Aggregated small granule
BLD	β-limit dextrin
CCS	Cow cockle starch
CL	Chain length
DMSO	Dimethyl sulfoxide
DSC	Differential scanning calorimetry
HCl	Hydrogen chloride
HMW	High molecular weight
HPSEC	High performance size-exclusion chromatography
IBD	Intra-cluster branch distance
ICL_{B2}	Internal chain length of B2 chain
IMW	Intermediate molecular weight
LG	Large granule
LBIM	Lightly branched intermediate molecule
LMW	Low molecular weight
MG	Medium granule
NaAc	Sodium acetate
NaOH	Sodium hydroxide
NCS	Normal corn starch
NRS	Normal rice starch
RI	Refractive index
RVA	Rapid viscosity analyzer
SCS	Sweet corn starch
SEM	Scanning electron microscopy
SG	Small granule
SGS	Small-granule starch
WCS	Waxy corn starch
WRS	Waxy rice starch

differences in the starch structure in comparison with normal corn starch (Wang et al., 1993). It was also revealed that SCS granules were hard to swell and low in melting enthalpy (Singh, Inouchi, & Nishinari, 2006; Tziotis, Seetharaman, Klucinec, Keeling, & White, 2005; Wang et al., 1993). In general, the SU-1 deficiency in sweet corn affects the structural and physicochemical properties of SCS, and there is need to better understand its granular and fine structure for potential industrial applications.

In this study, CCS and SCS were compared with normal and waxy rice starches and normal and waxy corn starches with regard to their morphological, thermal, pasting, and structural properties. The fine structures for both starches were determined to provide supporting information on their potential applications, such as fat replacement and texture modifications.

2. Materials and methods

2.1. Materials

Sweet corn (*sugary-1* variety) kernels were obtained commercially. Cow cockle seeds were purchased from Hangzhou Botanic Technology Co., Ltd., China. Waxy rice and normal rice were purchased from local grocery store. Waxy corn starch (WCS) and normal corn starch (NCS) were obtained from Cargill Inc. and National Starch and Chemical Company (now part of Ingredion), respectively. All chemical reagents used for the study were of analytical grade unless specified.

2.2. Methods

2.2.1. Extraction of crude starches

Sweet corn starch (SCS), waxy rice starch (WRS), and normal rice starch (NRS) were isolated using a modified alkaline extraction method. To start the extraction, 100 g of dry maize kernels or milled rice grains were ground into grits using a blender (Waring Laboratory Science, Torrington, CT) to pass through a 16-mesh sieve. The grits were mixed with 350 mL of 0.1% (w/v) sodium hydroxide (NaOH) solution and kept at 50 °C in a water bath for 30 min with constant agitation. The mixture was then homogenized using a blender at high speed for 4 min and passed through a 270-mesh sieve. The retained solids (by the sieve) were extracted again using another 350 mL of NaOH solution. The fractions permeated through the sieve were combined and centrifuged at 3000 × g for 15 min. The precipitate, as the crude starch material, was collected.

Cow cockle starch (CCS) was isolated according to the methods of Goering and Brelsford (1966) with modifications. Cow cockle seeds (100 g) were soaked in 350 mL 0.15 M lactic acid solution at 50 °C for 48 h and then ground using a blender. The slurry was passed through a 270-mesh sieve, and retained solid was again blended with 350 mL lactic acid solution and passed through the sieve. The permeated extracts were combined and allowed to stand for a short period of time (1–2 min) to decant hull pieces. The decanting process was repeated for three times before centrifugation (3000×g, 15 min) to obtain crude starch.

2.2.2. Purification of starches

Each crude starch material (about 50 g) was resuspended in 300 mL of NaOH solution (pH 10), agitated for 30 min, and centrifuged again. The precipitate was repeatedly washed with NaOH solution for four times, during which the proteinaceous material on the top of the precipitate was scrapped away using a spatula. Thereafter, the precipitate was resuspended in deionized water, neutralized to pH 7.0 using 1.0 M hydrogen chloride (HCl) solution, and centrifuged. The starch precipitate was further washed twice using deionized water (100 mL for each cycle) and finally using 100 mL of pure ethanol. The material collected was subjected to vacuum filtration and then dried overnight. All starch materials were stored in a desiccator at room temperature before use.

2.2.3. Chemical compositions of starches

The moisture content of each starch material was determined using the AACCI Method 44–15.02. Starch content was determined as described by AACCI Method 76–13.01. Nitrogen content was determined using the combustion method with a LECO model FP-2000 Nitrogen Analyzer (LECO Co., St. Joseph, MI), with its value multiplied by 5.75 to obtain the protein content. Apparent amylose content was determined by colorimetric method according to Morrison and Laignelet (1983).

2.2.4. Scanning electron microscopy

To image starch granules using scanning electron microscopy (SEM), each starch material was mounted on a circular metal stub with double-sided sticky tape and coated with platinum in a Cressington 208HR sputter coater. The specimens were observed using FEI NOVA nanoSEM Field Emission SEM (FEI, OR) under the voltage of 5.0 kV. Images were taken at magnification of 5000 ×.