



# Effects of inulin with different degree of polymerization on gelatinization and retrogradation of wheat starch



Denglin Luo<sup>a,b,\*</sup>, Yun Li<sup>a</sup>, Baocheng Xu<sup>a,b</sup>, Guangyue Ren<sup>a,b</sup>, Peiyan Li<sup>a,b</sup>, Xuan Li<sup>a,b</sup>, Sihai Han<sup>a,b</sup>, Jianxue Liu<sup>a,b</sup>

<sup>a</sup> College of Food and Bioengineering, Henan University of Science & Technology, 471023 Luoyang, Henan Province, China

<sup>b</sup> Henan Engineering Research Center of Food Material, 471023 Luoyang, Henan Province, China

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## ABSTRACT

The effects of three types of inulin, including FS (DP ≤ 10), FI (DP of 2–60) and FXL (DP ≥ 23), on the gelatinization and retrogradation characteristics of wheat starch were investigated. As the concentration of inulin added into starch increased, the gelatinization temperature increased whereas the breakdown value decreased, and the value of setback first decreased and then increased slightly. The three types of inulin with lower concentrations (<15%) all showed obvious suppression effects on the short-term retrogradation of wheat starch. After 7 days of storage, the three types of inulin showed a significant suppression of starch retrogradation in the addition range of 5–7.5%. They can all inhibit amylose retrogradation, but accelerate amylopectin retrogradation. Inulin with lower DP has stronger effects on the starch retrogradation. Generally, the three types of inulin can all retard the retrogradation performance of wheat starch to some extent in the long-term storage.

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## 1. Introduction

As a dietary fiber from the nature, inulin is a fructan varying in length from 2 to 60 fructose units linked by β-(2→1) glycosidic bonds. It is found in variety of vegetables, grains and fruits, usually extracting from *Chicory* or *Jerusalem artichoke* (Terkmane, Krea, & Moulai-Mostefa, 2016). Many studies have proved that inulin has many health benefits such as nourishing beneficial intestinal bacteria, reducing the risk of gastrointestinal diseases, regulating blood glucose, promoting absorption of minerals and enhancing immune system (Shoaib et al., 2016). Apart from its nutritional benefits, inulin can be used as a modifier in the formulation of foods for technological reasons, due to its excellent water-holding capacity, white color, powder characteristic similar to flour or starch, and good gel texture properties (Gao, Brennan, Mason, & Brennan, 2016; Kuntz, Fiates, & Teixeira, 2013).

According to the differences in degree of polymerization (DP), inulin is usually divided into three groups, namely short-chain (DP ≤ 10), native (DP 2–60) and long-chain (DP ≥ 23) types. The rheological and thermal properties of inulin molecules vary widely with temperature and concentration, as well as the chain length

(Bot, Erle, Vreeker, & Agterof, 2004). Short-chain inulin shows more water-soluble and water-retention properties than native and long-chain ones at lower temperatures, while long-chain inulin displays higher viscosities, and better and stronger gel formers at lower concentrations (Meyer, Bayarri, Tárrega, & Costell, 2011). Therefore, in order to enhance handling properties or nutritional values of products, inulin is used to alter texture or substitute fat according to its DP-sensitive gel forming and viscous behavior (Colla & Gamlath, 2015; Meyer et al., 2011). It has exhibited positive influence on the rheological properties of dough, texture and mouthfeel in dairy products, and texture and stability as a fat replacer in meat products (Shoaib et al., 2016). In these products starch as a main ingredient played important roles as thickener or gelling agents to afford a suitable viscosity or texture.

Several papers focused on the effect of inulin on thermodynamic and rheological properties of starches including potato and corn starches. Krystyan, Ciesielski, Khachatryan, Sikora, and Tomasik (2015) reported that viscoelastic properties and firmness of starch and short-chain inulin gels were largely dependent on their concentrations, which had obvious differences at 6% starch plus 25% inulin compared to control samples. 10% inulin addition could result in viscoelastic changes of mixture gels in comparison with pure starch gels. It was observed that phase inversion from a waxy maize starch-continuous system to an long-chain inulin-continuous system happened at a total polymer concentration

\* Corresponding author at: College of Food and Bioengineering, Henan University of Science & Technology, 471023 Luoyang, Henan Province, China.

E-mail address: [luodenglin@163.com](mailto:luodenglin@163.com) (D. Luo).

$\geq 30\%$  (w/w, w.b.) in rheological properties (Zimeri & Kokini, 2003b). The glass transition temperature suggested that no interactions or plasticizing effects existed between them. When the partial replacement of potato starch by pectin and inulin with different degrees of polymerization, it would significantly modified thermal and rheological properties of starch gels, and the consistency coefficient and both moduli were influenced by the average DP and concentration of inulin added (Witczak, Witczak, & Ziobro, 2014).

Recently ten years, the applications of inulin in wheat doughs have received considerable attention (Liu, Luo, Chen, Xu, & Liu, 2016; Salinas, Zuleta, Ronayne, & Puppo, 2016). The addition of inulin to wheat flour can prolong the time of dough development and stability, increase crumb hardness and value of the quality indexes, and decrease water absorption and bread volume (Karolini-Skaradzinska, Bihuniak, Piotrowska, & Wdowik, 2007; Peressini, 2009; Peressini, Page, & Lagazio, 2016). Poinot et al. (2010) found that inulin could shorten the bread baking time without any impact on the quality of bread. Gallagher, O'Brien, Scannell, and Arendt (2003) explored the application of fructo-oligosaccharide in biscuit dough and found an improvement in the quality and shelf-life of biscuit. Inclusion of inulin in an extruded flour-based product lowered dough consistency and elasticity due to a lubricating effect of sugars and oligosaccharides, and different kinetics of starch gelatinization. Short-chain inulin had a greater effect than long-chain inulin (Peressini, Foschia, Tubaro, & Sensidoni, 2015). However, there is scarce information about how inulin affects the gelling and thermal properties of wheat starch. As an important component of wheat flour, starch directly influences the processing properties of dough and the quality of final products to some extent. Hence, a better understanding of the rheological behaviour of inulin-wheat starch blend may help us to design desired formulations. The objective of this work is to investigate the impact of inulin addition on the pasting, gelling and retrogradation characteristics of wheat starch.

## 2. Materials and methods

### 2.1. Materials

Three types of commercially available inulin with different average DP: Fibruline® S30 (FS, average DP  $\leq 10$ , inulin content  $\geq 90\%$ , 7.6% of glucose, fructose and sucrose mixture), Fibruline® Instant (FI, DP: 2–60, inulin content  $> 86\%$ , 14% of glucose, fructose and sucrose mixture) and Fibruline® XL (FXL, average DP  $\geq 23$ , inulin content  $\geq 94.5\%$ , no glucose, fructose and sucrose) were obtained from Cosucra (Belgium). Wheat starch (87.5% of dry matter) was purchased from Daily Source Food Co., Ltd (Wuxi, China). Amylose and amylopectin standards extracted from potatoes were obtained from Blue Season Technologies Co., Ltd (Shanghai, China).

### 2.2. Experimental methods

#### 2.2.1. Pasting characteristics

Wheat starch (10 g) was dispersed into 100 mL deionized water to obtain suspensions before inulin powder was added to reach 2.5% to 20% of inulin in wheat starch (w/w), where inulin amounts were selected according to Liu et al. (2016). The pasting characteristics of the suspensions were analyzed using a Brabender visco-graph (Brabender GmbH & Co. KG, Germany). Each measurement was performed twice. And heating-cooling cycles were carried out at a constant shear speed of 150 rpm. The samples were heated from 30 to 95 °C with a heating rate of 1.5 °C/min and held at 95 °C for 30 min, and then cooled down from 95 to 50 °C at a rate of 1.5 °C/min and held at 50 °C for another 30 min (Li et al., 2016).

#### 2.2.2. Thermal analysis

Suspensions containing 30% wheat starch were prepared with deionized water followed by inulin powder addition in the same manner as above, where 30% starch was determined according to the value of retrogradation enthalpy measured for the accuracy. Their thermal properties were determined using a DSC-1 device (Mettler-Toledo, Switzerland) equipped with a nitrogen cooling system. The thermal analyzer was calibrated with standard indium and zinc, and a standard aluminum crucible was also used in the tests.

Suspensions of the wheat starch and inulin mixture ( $20 \pm 1$  mg) were weighed and hermetically sealed in an aluminum pan, which was then heated from 20 °C to 120 °C with a heating rate of 10 °C/min. An empty aluminum pan was used as the reference. Subsequently, when the samples were naturally cooled to 25 °C according to the calorimeter, they were immediately transferred to a refrigerator and stored at 4 °C for 7 days. The starch retrogradation properties of the samples were measured by reheating the sample pans under the same conditions used for gelatinization (Witczak et al., 2014).

#### 2.2.3. Determination of the glass transition temperature

Suspensions with 5% wheat starch were prepared using deionized water prior to addition of inulin powder in the above method. The suspensions were pasted for 1 h at 95 °C, and then the pasting samples were dried to constant weight with air oven at 60 °C and transferred to a dryer to balance for 24 h at 25 °C before the test. DSC-1 (Mettler-Toledo, Switzerland) calibrated with indium and zinc was used to measure the glass transition temperature ( $T_g$ ) of the samples. The samples were heated from 20 to 140 °C at 10 °C/min. After the first cycle of heating, the samples were immediately cooled at 100 °C/min, which was used for rescanning within the same range. The glass transition temperature was reported from the second scan of each sample by measuring the midpoint of the shift on the differential scanning calorimeter (DSC) curve.

#### 2.2.4. X-ray diffraction patterns

Suspensions of 5% wheat starch were prepared with deionized water prior to inulin powder addition using the previous method. The suspensions were pasted for 1 h at 95 °C. Each pasting sample was divided into four subsamples. One subsample was immediately dehydrated using anhydrous alcohol, degreased using chloroform and then dried for 24 h at 60 °C before scanning. The other subsamples were separately stored at  $-22$  °C, 4 °C and 25 °C for 7 days, and then scanned after the aforementioned dehydration, derosination and drying procedures. The degree of relative crystallinity was quantitatively determined according to the method of Zhang et al. (2014).

The X-ray patterns of the samples were obtained with copper, nickel and foil filtered, and then  $K\alpha$  radiation was performed using a diffractometer (D-8 Advance, BRUKER-AXS). The diffractometer was operated at 27 mA and 50 kV. The diffraction angle ( $2\theta$ ) was scanned from 5° to 50° in 0.05° increments with 2 s counting time (Xijun, Haiqi, Haibo, Liu, & Lin, 2015).

#### 2.2.5. Determination of amylose and amylopectin contents

The standard curves of amylose and amylopectin were prepared according to Jin's method (Jin, 2009). The samples were determined as follows: A starch sample of 100 mg with inulin was weighed, and chloroform was used to remove fats and oils. Then, 10 mL of 0.5 mol/L potassium hydroxide (KOH) was added and the mixture was heated to 95 °C within 30 min. After sample dispersion, it was cooled to room temperature and then transferred to a volumetric flask and diluted to 50 mL with deionized water. Afterwards, 2.5 mL of sample solution was taken and 20 mL of distilled water was added before adjusting the pH value to 3.5 with

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