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# Comparisons of three modifications on structural, rheological and functional properties of soluble dietary fibers from tomato peels

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ABSTRACT

Alkaline hydrogen peroxide (AHP), hydrochloric acid-ethanol (HAE) solution and enzymatic hydrolysis (EH) were used in three different modification methods to improve the extraction yield of soluble dietary fibers (SDF) from waste tomato peel. For each of the methods, named A-SDF, H-SDF, and E-SDF, the molecular mass, monosaccharide composition, surface charge, FT-IR, morphological characteristics, and rheological behavior were determined to demonstrate the gelling mechanisms. Compared with the original SDF (O-SDF), the three modified fiber samples displayed lower molecular mass and zeta potential. Specifically, A-SDF showed the lowest molecular mass and zeta potential, $1.30 \times 10^6$ Da, and −39.5 mV, respectively, which could account for the results that A-SDF with $Ca^{2+}$ presented the strongest gelling property and excellent capacity of glucose adsorption *in vitro*. A-SDF and H-SDF exhibited similar capacities in binding bile acids, which were higher than that of E-SDF and O-SDF. In conclusion, AHP could be regarded as the optimal modification method to improve the extraction yield and functional properties of SDF extracted from tomato peel.



## 1. Introduction

Tomato (*Lycopersicon esculentum*) is grown worldwide for its edible fruits. Thousands of tomato cultivars are grown worldwide, and annual global production of tomatoes has reached more than 160 million tons including fresh market and process tomatoes (Namir, Siliha, & Ramadan, 2015). Tomato waste, including seeds, skin, and pulp, has typically been used as animal feed (Grassino et al., 2016a). Tomato peels can be utilized as a cheap source of soluble dietary fiber (SDF), which is regarded as a functional ingredient with high value (Grassino et al., 2016b). According to a recent study by Zhai, Gunness, and Gidley (2016), the SDF from plants could decrease lipid digestion and exhibit a better effect on adjusting the blood glucose compared with insoluble dietary fiber (IDF) (Ma & Mu, 2016a).

Many methods have been used to change the ratio of dietary fiber with the aim of higher availability and better functional properties. Common modification methods including chemical, physical, and enzymatic hydrolysis were used to extract SDF from plants for food application (Liu et al., 2016a). According to a previous study by Maes and Delcour (2001), alkaline hydrogen peroxide (AHP) could react with lignin and hemicellulose to form water-soluble molecules with lower molecular mass. Hydrochloric acid-ethanol (HAE) solution has been shown to cause the mild degradation of cellulose and hemicellulose and improve the porous structure of IDF to obtain more soluble polysaccharides (Sun, Tang, Dong, & Li, 2013). Enzymatic hydrolysis (EH) was also an efficient method, with celluclast 1.5L and viscozyme L chosen to conduct EH due to their high xylanolytic and cellulolytic activities (Wikiera, Mika, Starzyńska-Janiszewska, & Stodolak, 2015).

In this study, the effects of AHP, HAE, and EH on the SDF extracted from tomato peel were compared using structural, rheological, and functional property data. The monosaccharide composition, molecular mass, zeta potential, FT-IR, and SEM were determined to illustrate effects on the structural and morphological characteristics. Bile acid holding and glucose adsorption capacities were measured to evaluate the functional properties of three modification methods. The results provide a scientific basis for enhancing the usefulness of waste tomato peel in the food industry.

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## 2. Materials and methods

### 2.1. Materials

Tomatoes were purchased from a local supermarket in Shanghai, China. Arabinose (Ara), fructose (Fru), fucose (Fuc), galactose (Gal), galacturonic acid (Gal A), glucuronic acid (Glu A), mannose (Man), rhamnose (Rha), xylose (Xyl), and 3α-hydroxysteroid dehydrogenase were purchased from J&K Scientific Ltd (Beijing, China). Nitro blue tetrazolium, chenodeoxychholic acid, cholic acid, diaphorase from *Clostridium kluyveri*, cholestyramine resin, MES monohydrate and glucose (Glu) were purchased from Sigma-Aldrich (St.Louis, MO, USA). A total dietary fiber kit (α-amylase, amyloglucosidase, protease) was purchased from Megazyme (Bray, Co. Wicklow, Ireland). Dextrans of different molecular mass were purchased from the National Institute for Food and Drug Control (Beijing, China). All other analytical reagents were analytical grade.

### 2.2. Modification of tomato peel fiber

Fresh tomatoes were peeled and dried in drying oven at 55 °C with continuous hot air for 12 h. Then, the dry peels were ground into a powder (60 mesh). The conditions of the modification method for AHP were 5 mL/100 mL $H_2O_2$ at pH 10, solid-liquid rate as 1 g: 15 mL, and reaction time of 1 h. The conditions of HAE were a HCl-EtOH rate of 1 mL: 9 mL, solid-liquid rate of 1 g: 12 mL, and reaction time as 1 h. The modified conditions of EH were 0.5 mL/100 g celluclast 1.5L and viscozyme L at pH 4.5, and a solid-liquid rate of 1 g: 10 mL for 2 h at 50 °C. The following steps were used for each of the three modification methods. After reaction, the HAE suspension was neutralized with NaOH (1 mol/L). The AHP and EH suspensions were neutralized by HCl (1 mol/L). Then, the solid matter was filtered with water-washing. Finally, the solid matter was dried at 55 °C for 12 h.

### 2.3. SDF extracted from modified tomato peels

The method of extracting SDF was based on a previous method (Kara, 2016) with some modifications. A 1.0 g of sample of dried peel was dispersed in 40 mL of MES-TRIS buffer (0.05 mol/L, pH 8.2 at 24 °C) by continuous stirring for 1 h 50 μL of α-amylase (3000 U/mL) solution was added to the suspension in a water bath above 95 °C for 35 min. Then, 100 μL of protease solution (50 mg/mL) was added at 60 °C for 30 min. Next, 100 μL of amyloglucosidase solution (3300 U/mL) was added at pH 4.5 and 60 °C with constant shaking for 30 min. The filter liquor was precipitated four times with volumes of 95% ethyl alcohols pre-heated at 60 °C. The precipitate was centrifuged, dissolved in distilled water, and removal of extra ethyl alcohol. Then the solution was freeze-dried to obtain the SDF.

### 2.4. Monosaccharide composition

#### 2.4.1. Hydrolysis of SDF

According to the method of Elboutachfaiti et al. (2017) with some modifications, the SDF was hydrolyzed. 2.0 mg sample was dissolved with 0.4 mL of distilled water and reacted with 0.4 mL of trifluoroacetic acid (TFA, 4 mol/L) for 3 h at 110 °C. Then, the excess acid was removed with constant nitrogen-blow. Dried samples were dissolved again with 1 mL of distilled water. Sample solutions were filtered through 0.45 μm filter membrane before use. A mixed solution with Glu, Gala, Ara, Fru, Rha, Xyl, Man, Fuc, Gal A and Glu A was prepared for standard curves.

#### 2.4.2. Ion chromatography

High performance anion exchange chromatography was used to analyze the monosaccharide composition of the SDF samples after acid hydrolysis. The detection equipment used was Dionex™ ICS-5000$^+$ (Thermo Scientific, Waltham, MA, USA) equipped with a CarboPacTMPA20 column and pulsed amperometric detection. Twenty-five μL of hydrolysis solution was injected automatically with a constant temperature of the column (30 °C) at the flow rate of 0.45 mL/min. The mixture eluant included distilled water (solution A), 50 mmol/L sodium hydroxide solution (solution B), and 0.5 mol/L sodium acetate solution (solution C). The eluant gradient was performed as follows: 95% solution A and 5% solution B (0–20 min); 85% solution A, 5% solution B and 10% solution C (20–30 min), and 100% solution B (30–40 min).

### 2.5. Measurement of molecular mass

The measurement of molecular mass was based on the technique used by Wang et al. (2016) with small modifications. The SDF sample (2 mg) was dissolved in 1 mL of distilled water and filtered through a 0.45-μm filter membrane. High performance liquid chromatography (HPLC, Agilent, USA) was equipped with a Shodex SUGAR KS-805 column (8 mm i. d. × 300 mm, Showa Denko, Tokyo, Japan) with RID-1A at 40 °C for analysis. 20 μL of solution was injected and eluted with distilled water at the flow rate of 0.8 mL/min for 25 min. The process of elution was maintained at 40 °C. Dextran with different molecular mass of 1.80, 2.50, 4.60, 7.10, 133.80, and 200.00 kDa were used to calculate the molecular mass of the SDF.

### 2.6. Measurement of zeta potential

The zeta potential of the SDF was measured by the method published by Feng, Dou, Alaxi, Niu, and Yu (2017). A Zetasizer Nano ZS90 (Malvern, UK) was used to evaluate the zeta potential of the SDF aqueous solution (2 mg/mL).

### 2.7. Fourier transformed infrared analysis (FT-IR)

Spectrum of the SDF was determined by a FT-IR spectrophotometer with an ATR accessory (Nicolet 6700, Thermo Fisherm Scientific, Waltham, MA, USA). The sample was thoroughly mixed with KBr by grinding and tableting. The wavenumbers ranged from 4000 to 400/cm with 32 scans and resolution of 4/cm.

### 2.8. Scanning electron microscopy

The microstructure and surface of the SDF samples were carried out by scanning electron microscopy (SEM) (Sirion 200, FEI Company, Netherlands) at 5 kV. Freeze-dried SDF samples were placed on double-side tape and coated with a thin gold layer. Representative microscopes were taken at 2000×magnification.

### 2.9. Rheological properties

#### 2.9.1. Instruments

The rheological properties of SDF were tested using an AR-G2 rheometer (TA Instruments, New Castle, DE, USA). An SRS Peltier Circulator (TA Instruments, USA), used as a heat exchanger was equipped to change the temperature of the plate sensor system.

#### 2.9.2. Flow behavior

A-SDF, H-SDF and E-SDF with 4 g/100 mL concentration were dissolved in distilled water at 55 °C with constant stirring for 30 min. Then the solutions were stored at 4 °C for 12 h to