Carbohydrate Polymers 115 (2015) 112–121

Contents lists available at ScienceDirect

Carbohydrate Polymers

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

Effect of ripening and heat processing on the physicochemical and rheological properties of pepper pectins

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ARTICLE INFO

Article history:
Received 15 February 2014
Received in revised form 7 August 2014
Accepted 10 August 2014
Available online 2 September 2014

Keywords:
Polysaccharides
Cell-wall materials
Functional properties
Physicochemical characteristics
New pectin sources

ABSTRACT

Water-, chelator-, and alkali-soluble pectins were isolated from raw and heat-processed Jalapeño peppers (green and red) and their physiochemical and rheological properties were determined. The yield, tristimulus color, degree of methyl esterification, monosaccharide composition, molecular weights distribution, and protein content depended on ripening and heat processing. The viscosity properties of pectins were independent of ripening. The water-soluble pectin was the most abundant pectin. Pectins from grilled peppers showed the lowest L^* values. The alkali-soluble pectin showed the highest protein content. The content of xylose, rhamnose, and mannose in pectins was highly altered by tested factors. The degree of methyl esterification of pectins ranged from 26.8 to 91.6%. The peak M_w of the main fraction of tested pectins was sequentially reduced by ripening and heat processing. Pectins from raw peppers showed the best viscosity properties.

1. Introduction

The world demand for pectin is annually growing by 4–5%. Pectin is used as thickening, gelling, emulsifying, and texturizing agent in food, pharmaceutical, and cosmetic products (Mesbahi, Jamalian, & Farahnaky, 2005; Chan & Choo, 2013). Citrus peels and apple pomace remain as the most important sources of commercial pectin. New pectin sources will be necessary in the future to satisfy the pectin demand. Jalapeño peppers might be an alternative source of these polysaccharides since they are highly cultivated, have a very low commercial value, unmarketable Jalapeño peppers are highly available due to their short shelf life, pepper wastes from seed-production industry are highly available, and the growers are demanding new uses and adding-value strategies for this crop (Ornelas-Paz et al., 2012). However, little is known about the pectin content in peppers and its characteristics. The pectin content in peppers depends, in a first instance, on genotype, ranging from 1.7 to 10.4% (DWB) (Arancibia & Motsenbocker, 2004; Bernardo, Martínez, Álvarez, Fernández, & López, 2008). However, the yield of pectin from a specific pepper genotype can be altered by ripening and heat processing. Bernardo et al. (2008) demonstrated that the pectin content of Fresno de la Vega and Benavente-Los Valles peppers decreased (7–23%) during ripening. Priya, Prabha and Tharanathan (1996) reported a considerable reduction (55%) of pectin content in Bell peppers during ripening. The ripening also modifies the chemical properties of pepper pectins, but this has been scarcely studied. Arancibia and Motsenbocker (2006) demonstrated that the degree of esterification of pectin from Tabasco peppers decreased from 70 to 40% during ripening and inferred a high poligalacturonase-mediated pectin depolymerization associated with pectin methylesterase activity. The neutral monosaccharide content in pectins from Bell pepper decreased by 47% during ripening, with the losses of galactose (Gal) (68%) and arabinose (Ara) (43%) being the most distinctive (Priya et al.,

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http://dx.doi.org/10.1016/j.carbpol.2014.08.062
0144-8617/

1996). Similar losses (42–56%) of neutral monosaccharides were reported for pectin from other genotypes of sweet and pungent peppers during ripening (Gross & Sams, 1984). Other changes in the physicochemical properties of pepper pectin as a function of ripening remain unknown.

The study of the effect of heat processing of peppers on yield and physicochemical properties of their pectins is scarce. Gu, Howard, and Wagner (1999) demonstrated that the total pectin content in several genotypes of Jalapeño peppers was reduced (11–19%) after heat processing and storage for 10 days. Similarly, Gallardo-Guerrero, Pérez-Gálvez, Aranda, Mínguez-Mosquera, and Hornero-Méndez (2010) found that hot-forced air drying decreased (30–45%, approximately) the pectin content in red peppers. Howard, Burma and Wagner (1997) demonstrated that blanching (50 °C, 15–60 min) and subsequent pasteurization (75 °C, 5 min) did not alter the esterification degree of pectin from Jalapeño peppers; however, these treatments decreased the content of several pectin fractions by 30–49%. Gallardo-Guerrero et al. (2010) found that the protopectin and soluble pectin are altered during hot forced-air drying of peppers. The increase of pectin solubility by some heat processing styles (pasteurization and blanching) has been hypothesized in peppers (Howard et al., 1997). Other heat-induced physicochemical changes in pepper pectin remain unknown.

The modification of the chemical characteristics of pectin by ripening or heat processing induces alterations in its functional properties, such as the rheological behavior, gelling ability, and capacity to bind compounds (Sila, Smout, Elliot, Van Loey, & Hendrickx, 2006b). The viscosity of their solutions and the strength of their gels increase as their molecular weight increases (Leroux, Langendorff, Schick, Vaishnav, & Mazoyer, 2003; Yapo, 2009). Pectins with high degree of methyl esterification form gels when in acidified solutions containing a solute such as sucrose, while pectins with low degree of methyl esterification can produce gels with divalent cations (Yapo, 2009; Chan & Choo, 2013). The degree of methyl esterification of pectins also modulates their binding of cations (Khotimchenko, Kolenchenko, Khotimchenko, Khozhaenko, Kovalev, 2010). De-esterified pectins are less susceptible to heat-induced degradation and less soluble. The charge of the carboxyl groups of galacturonic acid (GalA) can be influenced by pH and ionic strength of the medium, altering the three-dimensional conformation of the polysaccharide and its ability to modify the viscosity (Lara, García, & Vendrell, 2006; Lamikanra & Watson, 2007). Leroux et al. (2003) found that some pectin fractions are able to improve the emulsification of oil in water, presumably as a consequence of their protein content, low degree of acetyl esterification, and low molecular weight. The aim of this work was to determine the physicochemical characteristics and viscosity of several pectin types isolated from green and red Jalapeño peppers before and after two common heat-processing styles.

2. Material and methods

2.1. Plant material and heat processing

Green and red Jalapeño peppers (*Capsicum annuum* L. var. *Marajá*) were harvested from a commercial orchard in Chihuahua, México. Only fruits free of blemishes and defects were included in the experiment. The fruits were distributed in nine samples for each ripening stage. Three samples were boiled at 96 °C for 12.3 min. Another three samples were grilled on a hot plate at 210 °C for 13.2 min while the remaining three samples were used as untreated controls. Each sample was individually subjected to pectin extraction process. Individual fruits (raw and processed) were characterized for tristimulus color, dry matter content, firmness, and biometrical characteristics.

2.2. Preparation of the alcohol-insoluble residue (AIR)

The peduncle of peppers was removed. The raw peppers were blanched at 96 °C for 2.5 min before pectin extraction. Triplicate pepper samples (500.0 g) from each treatment were individually homogenized to puree in a kitchen blender (Taurus, Model Robot 180) for 5 min and suspended in ethanol (96%, v/v) in a ratio of pepper puree to ethanol of 1:4 (w/v). The suspension was subjected to a more intense homogenization until total disruption of pepper tissue (Homogenizer Ika T18 Basics; IKa Works Inc.). The mixture was stirred for 2 h at 25 °C and then maintained overnight at 4 °C. The mixture was filtered using a Whatman paper (No. 3) and the retained solids were recovered. The solids were suspended in acetone (ratio of 1:3, w/v), stirred for 2 h at 25 °C, and filtered again. This step was repeated six times until complete discoloration of solids. The residue was lyophilized, weighed for yield calculation, and pulverized using a mortar and pestle. The dried AIR was stored in N_2 atmosphere until pectin extraction.

2.3. Sequential extraction of pectic polysaccharides

Pectic polysaccharides were extracted according to Roeck, Sila, Duvetter, Van Loey, and Hendrickx (2008), with slight modifications. The AIR was subjected to sequential extraction of pectin with distilled water (96 °C for 5 min), 0.05 M *trans*-1,2-diaminocyclohexane-*N*,*N*,*N*′,*N*′-tetraacetic acid monohydrate (CDTA) in 0.1 M potassium acetate (pH 6.5, 6 h at 28 °C), and 0.05 M Na_2CO_3 containing 0.02 M $NaBH_4$ (16 h at 4 °C and then 6 h at 28 °C). The ratio of AIR to extracting solution was always 1:100 (w/v). The pH of all filtrates and retained solids was neutralized (pH 7.0) with NaOH or acetic acid. Pectic polysaccharides were precipitated from each filtrate by adding 3 volumes of ethanol, and the suspensions were maintained overnight at room temperature. The pectin was recovered by centrifugation (12,000 × *g*, 5 min at 4 °C) and filtration throughout a Whatman paper No. 541. The final residues obtained after pectin extraction process were washed with ethanol (3 L) and then recovered by filtration. Pectins and final residues were lyophilized, weighed, pulverized, and stored under N_2 atmosphere. Pectins and final residues were named according to their solubility as water-, chelator-, and alkali-soluble pectin (WSP, CSP, and NSP) and insoluble fibers (IF), respectively. These materials were not subjected to further purification processes and therefore they might contain other polysaccharides. Pectins were characterized for their physicochemical (yield, tristimulus color, degree of methyl esterification, monosaccharide composition, molecular weights distribution, and protein content), and rheological (viscosity of their solutions) properties.

2.4. GalA content and degree of methyl esterification

The GalA was liberated from pectin and quantified according to Ahmed and Labavitch (1978) and Filisetti-Cozzi and Carpita (1991). Pectin samples (5 mg) were subjected to acid hydrolysis for 40 min, using concentrated H_2SO_4 (2 mL) and water (1 mL). The volume of the reaction was adjusted to 10 mL using water, and then aliquots of each hydrolyzate (400 μL) were mixed with 4 M potassium sulfamate (40 μL, pH 1.6) and 75 mM sodium tetraborate in concentrated H_2SO_4 (2.4 mL), and maintained at 96 °C for 20 min. After cooling, 80 μL of *m*-hydroxydiphenyl solution (0.15% 3-phenylphenol in 0.5% NaOH) or 0.5% NaOH (control reaction) were added and the GalA content was colorimetrically determined at λ = 525 nm using a 6405 Jenway UV/Vis spectrophotometer (Jenway Ltd., Essex, UK). The GalA quantification was performed using a calibration curve constructed with three independent sets of dilutions of pure D-galacturonic acid.