Journal of Cereal Science 70 (2016) 275–281

Contents lists available at ScienceDirect

Journal of Cereal Science

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

Changes in the structure and gelling properties of maize fiber arabinoxylans after their pilot scale extraction and spray-drying

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ARTICLE INFO

Article history:
Received 10 March 2016
Received in revised form
18 May 2016
Accepted 6 July 2016
Available online 9 July 2016

Keywords:
Arabinoxylans
Pilot scale
Spray-drying
Gelation

ABSTRACT

The effects of pilot scale extraction and spray-drying on structure (molecular weight, hydrodynamic radius, branching and phenolic compounds) and gelation capacity of maize fiber arabinoxylans (AX) were assessed. A control extract was obtained at laboratory scale and freeze-dried (ctrl-AX). The AX obtained at pilot level were either freeze-dried (AX (−)) or spray-dried (AX (+)). Total carbohydrates, molecular weight and sugar linkages were not affected by the scale extraction. Ctrl-AX formed more viscous solutions (3.5 times), had higher ester-linked ferulic acid (e-FA) (1.5 times) and smaller hydrodynamic radius (1.3 times) compared to those extracts obtained at the pilot scale. All AX gels showed elastic behavior in creep-recovery tests, but as indicated by their measured complex viscosity ($\eta^* = 32–40$ Pa s) and maximum creep compliance (Jcmax = 0.4–0.06 1/Pa) extracts obtained from pilot scale were weaker compared to ctrl-AX ($\eta^* = 216$ Pa s, Jcmax = 0.01 1/Pa). A better understanding on the relationships between the AX structure and its gelling properties was obtained; likewise, useful aspects to consider for scaling up AX extraction from maize fiber are presented in this manuscript.

1. Introduction

Arabinoxylans (AX) are non-starch polysaccharides mainly localized in the endosperm cell walls, aleurone layer and the pericarp of cereal grains. Their structure is mainly constituted of a linear β-(1,4)-D-xylopyranose backbone and L-arabinofuranose residues as side chains on O-2 and/or O-3 positions (Carvajal-Millán et al., 2007). Some of the arabinose moieties are ester-linked on O-5 positions to hydroxycinnamic acids (HCA) such as ferulic acid (FA), *p*-coumaric acid (*p*-CA) and dimers ad trimers of ferulic acid (Lapierre et al., 2001).

AX exhibit different functional properties (water solubility, viscosity, gelling and hydration properties) according to their physicochemical characteristics, which are strongly related to their structural features such as molecular weight, degree of branching and HCA profile (Saulnier et al., 2007). It has been also reported that AX structure is influenced by the extraction treatment, which include acid, enzymatic, alkaline and mechanical treatments, and purification methods (ultrafiltration, chromatography, centrifugation) (Zhang et al., 2014).

Some functional properties of AX are relevant to many applications, which include their use as adhesives, thickeners, stabilizers, emulsifiers, controlled release matrix and film former agents (Carvajal-Millán et al., 2007; Saeed et al., 2011; Yadav et al., 2007). Furthermore, the soluble dietary fiber nature of prebiotic AX imparts relevant health benefits such as control of diabetes mellitus, cardiovascular disorders and improve colon function (Rose et al., 2010; Saeed et al., 2011). Recent studies have demonstrated that AX also exert activity against some types of cancers and immunological disorders (Cao et al., 1993; Ogawa et al., 2005; Zhou et al., 2010). Based on these results, AX have potential application in both the food and pharmaceutical industries. Therefore, it is

Abbreviations: AX, arabinoxylans; AX (−), arabinoxylans from maize fiber extracted at the pilot scale and freeze-dried; AX (+), arabinoxylans from maize fiber extracted at the pilot scale and spray-dried; ctrl-AX, arabinoxylans from maize fiber extracted at the laboratory scale and freeze-dried; DLS, dynamic light scattering; e-FA, esterified ferulic acid; FA, ferulic acid; G′, elastic modulus; G″, viscous modulus; HCA, hydroxycinnamic acids; J, compliance; Jcmax, maximum creep compliance; Jer, elastic recovery compliance; Jvr, viscous recovery compliance; *p*-CA, p-coumaric acid; η^*, complex viscosity; τ, stress; γ, strain.

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http://dx.doi.org/10.1016/j.jcs.2016.07.005
0733-5210/

necessary the development of feasible processes to extract AX at pilot and industrial levels. Nonetheless, only a few studies have dealt with pilot and industrial scale extraction of AX (Annison et al., 1992; Mansberger et al., 2014).

In the present study, the effect of pilot plant extraction and spray-drying on the structure (molecular weight, hydrodynamic radius, branching degree and phenolic compositions) and gelling properties of maize fiber AX have been evaluated. The AX extraction from a laboratory process followed by freeze-drying were used as benchmark.

2. Material and methods

2.1. Extraction source

A commercial fiber from white maize was obtained from a wet-milling procedure applied by Mexstarch Industry Sapi de C.V. (Sinaloa, México). The CF was ground in a Wiley mill equipped with a 0.5 mm mesh, and the resulting milled samples placed in sealed plastic bags and stored at 4 °C until use.

2.2. Extraction and experiment design

Maize fiber AX were extracted under alkaline conditions (0.3 M sodium hydroxide) for 6 h at 60 °C as reported Ayala-Soto et al. (2016). Three types of AX samples having different extraction conditions and drying processes were evaluated. The samples are named as follow:

- **Ctrl-AX**: Arabinoxylans from maize fiber extracted at the laboratory scale and freeze-dried.
- **AX (−)**: Arabinoxylans from maize fiber extracted at the pilot scale and freeze-dried.
- **AX (+)**: Arabinoxylans from maize fiber extracted at the pilot scale and spray-dried.

After the 6 h alkaline treatment with 0.3 M sodium hydroxide (1:15 w/v) at 60 °C, the extraction process of AX at the laboratory level was followed as previously reported by Ayala-Soto et al. (2016) and performed three times. The resulting extracts were freeze-dried at 0.036 mbar and −50 °C for the condenser temperature (Freeze Drier 4.5, LABCONCO, Kansas City, MO).

Regarding the samples obtained from the pilot scale process, triplicates of 100 g of maize fiber were suspended in 0.3 M sodium hydroxide (1:15 w/v agitated at 150 rpm) during 6 h at 60 °C by using a glass beaker (capacity 3 L) covered with aluminum foil. The temperature was controlled through a magnetic stirring hot plate (Thermo Scientific, USA). Following, the suspension was centrifuged at 4500 g for 15 min at 20 °C (IEC CL40R, Thermo Scientific, France) and the resulting supernatant acidified to pH 4 with 3 N hydrochloric acid. The precipitated hemicellulose A was separated through centrifugation (4500 g for 15 min at 20 °C) and the acidified supernatant was mixed with 96% ethanol (55% v/v) in order to enhance the precipitation of hemicellulose B. The solution was allowed to precipitate at 4 °C overnight. The ethanol supernatant was separated using a peristaltic pump (mini-pump variable flow, Fisher Scientific, USA). A second precipitation of the AX extract was performed with 55% ethanol to decrease the content of ethanol soluble compounds in the extract. The content of ethanol in the precipitate was evaporated using rotary evaporation under vacuum at 80 mbar at 55 °C and 100 rpm (Rotavapor R-220 Büchi, Switzerland). During this process water was added to decrease the high viscosity of the extract. In preparation for spray and freeze-drying, the solids of the AX solution was adjusted to 2.9% ± 0.3%. The freeze-drying conditions were the same to the applied at laboratory level. The spray-drying conditions were set with inlet and outlet temperatures of 190 °C and 90 °C, respectively and a flow rate of 570 mL/h (Spray drier 311S YAMATO Scientific Co, Japan).

2.3. Molecular weight distribution and hydrodynamic radius

Molecular size distributions of AX were determined using high performance size-exclusion chromatography equipped with multi-angle light scattering and refractive index detectors (HPSEC-MALS-RI) as previously described by Rumpagaporn et al. (2015). AX solutions (0.1% w/v) were filtered beforehand through a 1.5 μm cut-off membrane filter and injected (100 μL) into a Sephacryl S500HR (Amersham Biosciences, Piscataway, NJ, USA) column. A 0.02% sodium azide solution pumped at a flow rate of 1.3 mL/min was employed as the eluent. Detectors used were the Optilab rEx refractive index detector and Dawn Heleos II multi-angle light scattering detector equipped with a 658 nm GaAs laser diode (Wyatt Technology Corporation, Santa Barbara, CA, USA). Data were collected and analyzed using the ASTRA software (version 5.3.4.10) from Wyatt Technology (A value dn/dc = 0.136 was used) (Rumpagaporn et al., 2015).

Dynamic light scattering (DLS) was used to determine the hydrodynamic radius of the AX solutions (2 mg/mL). Measurements were carried out using a light scattering instrument ALV/CG-3 equipped with a Light Scattering Electronic and Multiple Tau Digital Correlator ALV/LSE-5004 (Langen, Germany). Correlation data was obtained by 3 runs of 20 s for each sample, reading at 10° increasing angles from 30° to 150° at a temperature of 22 °C. Cumulative and regularized fittings were used to obtain the particle size distributions and hydrodynamic radius by using the CONTIN 2DP algorithm, which was present in the ALV data analysis software.

2.4. Chemical analysis

The protein and lipids (methods 978.02 and 945.16, AOAC, 1992) content were determined in the final AX extracts.

A colorimetric assay was employed to determine the content of pentosans (arabinose and xylose) in AX samples (Douglas, 1981). Briefly, D-(+)-xylose was used to construct the calibration curve at concentrations of 0.1, 0.2, 0.3, 0.5, 0.75 and 1 mg/mL. The colorimetric reagent was prepared beforehand by mixing 5 mL of phloroglucinol in absolute ethanol (20% w/v) with 110 mL of glacial acetic acid, 2 mL of hydrochloric acid and 1 mL of glucose solution in water (1.75% w/v). Triplicate 2 mL aliquots of each standard dilution were mixed with 10 mL of the colorimetric reagent. Immediately, the tubes containing the mixes were placed in a boiling water bath for 25 min. The samples were removed, cooled in an ice bath for 1 min, and immediately placed in a water bath adjusted to room temperature for 1 min. The tubes were removed, laid horizontally, and covered with aluminum foil. After 10 min, the absorbance of the samples was read at 552 and 510 nm using a Genesys 10S UV−Vis spectrophotometer (Thermo Scientific, Milford, MA). The absorbance reading at 510 nm was subtracted from that at 552 nm to remove the influence of hexoses. Concerning to experimental samples, 2 mL of aqueous solutions of AX samples (1 mg/mL) were prepared to perform the procedure described above for standard dilutions. Values of AX content in samples were determined interpolating the equation derived from the standard xylose curve. The total pentosan content in samples was determined in triplicates and the values were reported as percentages (g xylose/100 g extract in dry weigh basis (db)).

The composition analysis of neutral sugars (xylose, arabinose, glucose and galactose) was performed according to the methodology described by York et al. (1985). Alditol acetate derivatives in