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Effect of processing time, temperature and alkali concentration on yield extraction, structure and gelling properties of corn fiber arabinoxylans

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ABSTRACT

The effect of time (2, 8 h), temperature (40, 60 °C) and NaOH concentration (0.25, 0.50 M) on yield extraction, hydroxycinnamic acid (HCA) concentration and gelling properties of corn fiber arabinoxylans (CFAX) were evaluated. The yield of arabinoxylans (AX) was higher at 8 h and 60 °C whereas alkali concentration did not influence this value. A negative correlation existed between AX yield and ester-linked HCA ($r = -0.7$). Stronger CFAX gels were formed at higher ester-linked ferulic acid concentrations (e-FA). Prediction equations and a range of desirable values of AX yield, e-FA and complex viscosity (η^*) were generated to select alkaline conditions. The selected conditions (6 h, 0.3 M NaOH, 60 °C) had the third highest AX yield (36.6%) whereas in in terms of e-FA and gel strength it had the fourth position (0.59 μg e-FA/mg AX dry basis (db), η^* 185 Pa s).

1. Introduction

Corn fiber (CF) is a low-value byproduct that is produced in high quantities from the corn wet-milling industry. It is mainly composed of pericarp and also contains cell wall materials from the endosperm. Based on previous studies, CF is a potential source of added-value arabinoxylans (AX) (Ayala-Soto, Serna-Saldívar, García-Lara, & Pérez-Carrillo, 2014a, 2014b; Carvajal-Millán et al., 2007; Kale, Pai, Hamaker, & Campanella, 2010; Kale, Hamaker, & Campanella, 2013; Lapierre, Pollet, Christine, Ralet, & Saulnier, 2001).

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 (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) and *p*-coumaric acid (*p*-CA). In addition to monomeric FA, dimers and trimer structures of this compound has been identified previously in AX extracted from wheat or corn bran (Lapierre et al., 2001; Funk, Ralph, Steinhart, & Bunzel, 2005). AX are subdivided into water-extractable (WEAX) and water-unextractable (WUAX) fractions. The insolubility of this polymer is related to the cross-linking between HCA and other cell-wall components (cellulose, proteins and lignin) (Chanliaud, Saulnier, & Thibault, 1995; Courtin & Delcour, 2002). Different alkaline or enzymatic treatments with a combination of processing time and temperature have been used for AX solubilization. The alkaline conditions are most suitable to extract corn fiber arabinoxylans (CFAX) due to their high-branched structure (A/X 0.5–0.85) nature (Lapierre et al., 2001).

AX can gel through the formation of covalent bridges and physical interactions between FA moieties, which are formed under oxidative coupled cross-linking with chemical conditions (O_2, H_2O) or catalyzed under the action of different enzymes such as laccase and peroxidase (Berlanga-Reyes, Carvajal-Millán, Lizardi-Mendoza, Islas-Rubio, & Rascón-Chu, 2011; Niño-Medina et al., 2009). AX possesses interesting functional applications such as adhesive, thickener, stabilizer, emulsifier, controlled release matrix and film former (Carvajal-Millán et al., 2007; Yadav, Moreau, & Hicks, 2007; Saeed, Pasha, Anjum, & Sultan, 2011). Furthermore, the soluble dietary fiber nature of AX imparts relevant health benefits such as control of diabetes mellitus, cardiovascular disorders and improve

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colon function due to its prebiotic nature (Rose, Patterson, & Hamaker, 2010; Saeed et al., 2011). Recent studies have demonstrated that AX also have activity against some types of cancers and immunological disorders (Cao et al., 2011; Ogawa, Takeuchi, & Nakamura, 2005; Zhou et al., 2010).

The functionality of arabinoxylans varies due to their polymeric nature, structural features (molecular weight, branching degree, HCA profile) and source of extraction mainly in terms of alkaline conditions (Saulnier, Sado, Branlard, Charmet, & Guillon, 2007). Carvajal-Millán et al., (2007) researched the effect of extraction time on AX yield from maize bran whereas Kale et al., (2013) reported the effect of time and alkali concentration on FA values and gelling properties. However, considering the potential applications of AX in the industry, it is necessary to optimize the alkaline extraction of fiber-rich coproducts by evaluating three important parameters: extraction time, alkali concentration and temperature. For this reason, the objective of the present study was to select the alkaline conditions to extract CFAX with the highest possible extraction yield, without negatively affecting the gelling capacity. The selection was performed by evaluating the effect of time, temperature and NaOH concentration on extraction yield, hydroxycinnamic acid (HCA) concentrations and gelling properties of resulting CFAX extracts. Furthermore, the relationship between AX structure and functionality was determined.

2. Materials and methods

2.1. Extraction source

A commercial corn fiber (CF) from white maize was obtained through the 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 samples were placed in plastic bags and stored in a freezer until use.

2.2. Experiment design of arabinoxylans (AX) extraction

A 2^3 complete factorial design was applied to evaluate the alkaline processing conditions on the parameters specified. The variables were time (2 or 8 h), temperature (40 or 60 °C) and sodium hydroxide concentration (0.25 M or 0.50 M). These parameter levels were selected according to preliminary studies.

During alkaline extractions all samples were agitated at 150 rpm and then centrifuged at 17,000 g, for 15 min at 20 °C (IEC Central MP4R Needham Heights, Ma). Supernatants were taken and adjusted to pH 4 with 3N hydrochloric acid. Then samples were centrifuged again (17,000 g, 15 min at 4 °C) in order to separate a pellet rich in hemicellulose A, and the acidified supernatant was treated with absolute ethanol (addition of 55% v/v). The alcoholic solution was left overnight at 4 °C in order to enhance the precipitation of hemicellulose B, fraction rich in AX. The precipitate was recovered by centrifugation (10,000 g, 10 min at 20 °C) and air-dried (50 °C, 1 h) to volatilize ethanol from pellets. Finally, the resulting extracts were freeze-dried at 0.036 mbar and −50 °C (Freeze drying 4.5, LABCONCO, Kansas City, MO). The amount of extracts was expressed as percentage on dry basis relative to the weight of CF.

2.3. Determination of AX content using the phloroglucinol colorimetric assay (PCA)

A colorimetric assay was employed to determine the content of pentosans (arabinose and xylose) in corn fiber (CF) and CFAX (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, 5 mg of corn fiber was added to 2 mL of water and 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)). Extraction yield of AX was estimated as the percentage of AX content in alkaline extracts with respect to the AX content in CF (39.74%). The purity of extracts was determined as the percentage of AX content in their respective extracts.

2.4. Quantification of hydroxycinnamic acids

Total and free hydroxycinnamic acids (HCA) such as *p*-coumaric acid (*p*-CA), ferulic acid (FA), dehydrodimers (di-FA) and dehydrotrimers of ferulic acid (tri-FA) were identified and quantified according to the protocol described previously by Ayala-Soto et al., (2014a). Sample preparation of total HCA concentration consisted in an alkaline treatment (2 N NaOH at 35 °C for 2 h), followed by acidification to pH 2 with 4 N hydrochloric acid. The compounds were extracted twice with diethyl ether (500 μL). Ethyl ether phases were evaporated with nitrogen flush and dried extracts were suspended with 500 mL 50% methanol (HPLC grade). To free forms of HCA, 1 mL of 1% hydrochloric acid in methanol (MeOH-HCl 1%) was added to 20 mg of AX extracts. The extraction was performed during 2 h on a rotatory shaker (200 rpm) at 25 °C, and the resulting extracts were centrifuged (IEC Central MP4R, Needham Heights, MA) at 10,000 g for 10 min at 20 °C. Supernatants were evaporated with nitrogen flush and the phenolic bran extracts were dissolved in 500 μL of 50% MeOH (HPLC grade). Each sample was filtered through a 0.45 mm (syringe) and then injected (5 μL) into the column of the HPLC equipped with a photodiode array (PDA) detector. Linear gradient elution was performed by HPLC-water acidified with trifluoroacetic acid (pH 2) and acetonitrile, at a flow rate of 0.6 mL/min at 25 °C (Chemstation for LC Copyright© Agilent Technologies, 1990–2003). Peak identification and quantification of FA and *p*-CA was based on retention times of authentic standards and their calibration curves at 320 nm. Values were reported as micrograms of *p*-CA or FA per milligram expressed in dry basis (μg *p*-CA/mg db or μg FA/mg db). The identification of di-FA and tri-FA peaks was performed analyzing UV spectra (at 320 nm) and mass spectra using HPLC-TOF-MS at the conditions described above for HPLC-PDA. The sum of di-FA and tri-FA was reported as FA equivalents per milligram expressed in dry weight basis (μg FAE/mg db). Total HCA were reported as μg HCA/mg db. Ester-linked compounds were determined by subtracting the values of free from total HCA. The percent composition of e-HCA was calculated with respect to the total amount of extracts.