

Extraction and Characterization of Hemicellulose from the Corn Fiber Produced by Corn Wet-Milling Processes

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The industrial processing of corn by wet-milling converts the hull layer into the byproduct known as corn fiber, which is combined with other materials (e.g., steep and fermentation liquids) to make corn gluten feed. Increased fuel ethanol production from corn will necessitate other uses of corn fiber. More than 30% of the corn fiber is in the form of a xylan (CFX) composed mainly of xylose and arabinose. Various procedures were examined for rapid extraction of CFX from corn fiber to obtain a purified material with good recoveries. Using 2% calcium hydroxide and precipitation with ethanol yielded a relatively pure CFX material but required long extraction times (16–20 h) and produced a hard, insoluble fiber residue. Extractions with 15% ammonium hydroxide yielded a rather impure CFX material and required similar long extraction times. Extraction with potassium hydroxide solutions were attempted using various combinations of time, temperature, and concentrations. Extraction with 2% potassium hydroxide at 70 °C for 4–6 h, coupled with calcium hydroxide treatment of the dissolved CFX, produced a highly pure CFX material composed of about 94% neutral sugars (34% arabinose, 52% xylose, 7% galactose, 8% glucose), 7% hexuronic acids, and 3% protein. The overall CFX yields were good (15%), and the residual fiber material still appeared physically suitable for use as a feed or for other uses.

Keywords: *Xylan; hemicellulose; corn fiber; corn hull; corn gum; corn xylan; arabinoxylan; pentosan*

INTRODUCTION

The processing of corn grain by wet-milling processes yields a variety of product streams that include starch, protein (gluten meal), oil, germ meal (spent flake), corn fiber (hulls or bran), and steep liquor. Almost all fuel ethanol is made by wet-milling industries that ferment the starch fraction and sell the other streams as by products to offset the costs of ethanol fermentation. The corn fiber is usually combined with spent fermentation and other liquids, dried down, and sold as a low-cost feed known as corn gluten feed. Whereas ethanol production from corn could be increased, it is questionable whether there would be comparable increased animal market demand for the corn gluten feed. Alternatively, corn fiber could be converted into or used as starting material for producing high-value products. For example, corn fiber contains at least 30% hemicellulose. This hemicellulose is water soluble, produces viscous solutions, and has properties similar to those of exudate gums and thus may have uses as a thickener in foods or as an adhesive (Whistler, 1993).

Corn fiber hemicellulose has been structurally and chemically analyzed and shown to be an arabinoglucuronoxylan (Montgomery et al., 1956a,b, 1957; Montgomery and Smith, 1957; Whistler and Corbett, 1955, 1956; Whistler and BeMiller, 1956). This corn fiber xylan (CFX) is a low molecular weight xylan that is extensively substituted with side chains of 4-*O*-methylglucuronic acid, arabinose, and a trisaccharide composed of arabinose, xylose, and galactose. CFX was first

isolated from corn fiber by strong hydroxide extraction (Wolf et al., 1953), but the preparations were deemed undesirable because of their dark brown colors. Rutenberg and Herbst (1957) used various combinations of sodium hydroxide or calcium hydroxide to extract CFX from corn fiber. Whereas calcium hydroxide extracting solutions yielded very clear CFX preparations, the procedures involved long extraction times and the recoveries were low. In the present work, these findings have been confirmed. Thus, the objectives of this work were to examine various other procedures for use in the development of rapid extraction of high-purity CFX from corn fiber that also resulted in a corn fiber residue that might retain value as an animal feed material or fiber product for food or other uses.

MATERIALS AND METHODS

Materials. Wet, fresh processed corn fiber was collected directly from the production facilities of Pekin Energy Co. (Pekin, IL) and immediately was returned to the laboratory. The corn fiber was divided into 10–12 kg portions that were placed in walk-in freezers and stored frozen (–20 °C) until used; freezing had no apparent effect on the extraction methods used. All chemicals used were of reagent grade or higher grade. Chemicals, biochemicals, and enzymes were purchased from Sigma Chemical Co. (St. Louis, MO) or from Aldrich Chemical (Milwaukee, WI).

Chemical Analyses. Neutral sugar compositions of corn fiber and xylans were determined by hydrolyzing samples in 2.0 M trifluoroacetic acid for 60 min at 100 °C and measuring the component sugars as alditol acetate derivatives (York et al., 1986). Total pentose sugar content of samples was determined according to the orcinol method (Schneider, 1957) and uronic acid content according to the dimethylphenol

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Table 1. Properties of Corn Fiber Xylans Isolated by Calcium Hydroxide and Ammonium Hydroxide Extractions^a

preparation	2C-A	15NH-A	15NH-BK	15NH-BKC
1st extract	2% Ca(OH) ₂	15% (NH ₄)OH	15% (NH ₄)OH	15% (NH ₄)OH
2nd extract			0.5% KOH	0.5% KOH
3rd extract				2.5% Ca(OH) ₂
total neutral sugars (%) ^b	80.1	64.5	68.8	90.0
distribution of neutral sugars (%)				
arabinose	36.3	31.6	33.8	35.7
xylose	56.6	58.1	57.8	56.8
galactose	7.3	8.6	7.4	6.6
glucose	0.5	1.6	0.6	0.6
orcinol sugars (%) ^b	76.8	65.1	70.9	86.2
hexuronic acids (%) ^b	7.7	5.5	8.0	6.9
protein (%) ^b	2.1	7.2	4.6	2.4
recovery (%) ^c	14.0	11.1	8.2	5.1

^a See text for details of extraction procedures. ^b Expressed as percent of dry weight of xylan recovered. ^c As percent weight of initial corn fiber based on orcinol sugar content of corn fiber and recovered xylan.

procedure (Scott, 1979). Protein contents of samples were estimated according to the method of Lowry et al. (1951).

Destarching of Corn Fiber. Corn fiber contains ~50% moisture, and while the dry weight, nonstarch composition of corn fiber remains fairly constant, the starch content varies from ~15 to 30% (dry wt) depending on the particular milling operation and processing rate. With most batches, the starch content is 18–22%. To prevent coextraction of starch with xylan, corn fiber was first destarched. About 3 kg (wet wt) of corn fiber was quickly thawed by mixing it into 8 L of hot (70 °C) distilled water and stirred for 30 min at room temperature. After filtering through two layers of cheesecloth, the fiber was re-extracted with hot water as above, filtered again, and then resuspended in 6 L of 5 mM citrate–phosphate buffer (pH 4.5) containing 0.6 unit/mL of glucoamylase. After incubating 12 h at 55 °C, the mixture was filtered through cheesecloth, extracted again with hot water, filtered, and then divided into 400 g (wet wt) portions and frozen until needed.

Xylan Extraction and Isolation. The routine procedures used to isolate CFX generally included the following steps. About 400 g (wet wt) of destarched corn fiber was stirred into 2 L of 70 °C extracting solution (see Table 1) and incubated for 4–5 h at 70 °C with occasional stirring with small amounts of solution additions to maintain a low viscosity. After cooling to 30–35 °C, the mixture was divided into 200 mL portions and centrifuged (20 °C; 20 min; 18000g). The supernatant fluid was decanted, and the pellet was resuspended in 100 mL of distilled water and centrifuged as before. The supernatant fluids were pooled and adjusted to pH 4–4.5 with 6 N HCl with rapid stirring. After another 5–10 min of stirring, the flocculent precipitate was removed by centrifugation as before. Three volumes of cold (4 °C) 95% ethanol was slowly added with stirring to the supernatant fluid. After another 5 min, the stirring was stopped and the formed precipitate allowed to settle out for another 10–15 min. The bulk of the fluid was decanted and discarded. The precipitate was collected by low-speed centrifugation (20 °C; 10 min; 5000g). The crude, rubber-like xylan pellets were cut into small (3–5 mm square) pieces and either freeze-dried to dryness or processed further.

For further purification, the xylan pieces were completely dissolved in 400–600 mL of 0.5–2% (w/v) KOH by stirring at 60–70 °C for 12–16 h. The xylan was then recovered from this solution using the same HCl pH adjustment, centrifugation, and ethanol precipitation steps as described above. Alternatively, impurities were removed from the dissolved xylan by slowly adding Ca(OH)₂ to the solution to a final concentration of 2% (w/v). After stirring for 1–2 h at 70 °C and then cooling to room temperature, the mixture was centrifuged (20 °C; 10 min; 18000g) to remove the insoluble Ca(OH)₂ absorbant material. The xylan was recovered from the supernatant fluid using the same HCl pH adjustment, centrifugation, and ethanol precipitation steps described previously. After freeze-drying, the xylan was ground to a fine powder by brief treatment with a coffee bean grinder.

Analyses of Xylans. Xylans were spectrally analyzed by dissolving the powdered material to final concentration of 2%

Table 2. Physical Properties of Corn Fiber Xylan Preparation

preparation	color (dry)	color (2% solution)	absorbance	
			520 nm	600 nm
2C-A	white	clear	0.27	0.25
15NH-A	dark tan	cloudy, particulate	1.80	1.54
15NH-BK	light brown	slightly opaque	1.00	0.79
15NH-BKC	cream	clear	0.11	0.14
10K-BK	tan	slightly opaque	0.61	0.43
10K-BC	cream	clear	0.08	0.04
2K-A	light brown	cloudy, particulate	2.22	2.08
2K-BC	off white	clear	0.17	0.13
0.56K-A	tan	cloudy	0.49	0.20
0.56K-BC	off white	clear	0.26	0.19

(w/v) in distilled water, using stirring and heating to 70 °C for 2–4 h, if necessary. Absorbance and spectral scans of these solutions were done using a Hewlett–Packard 8452A diode array spectrophotometer. Moisture content of xylans, corn fiber, and other samples was determined by use of a model 1 Denver moisture analyzer (Omnimark Instrument Co., Tempe, AZ).

RESULTS AND DISCUSSION

Corn Fiber Composition. Corn fiber streams obtained directly from wet-milling processes do not vary greatly in chemical composition, except for starch content. Wolf et al. (1953) found the starch content to be ~9–23%, whereas we have found that most of our samples are in the range of 15–20%. The nonstarch components included 10–13% protein, 20–22% cellulose, 3–4% uronic acids, 2–3% oil, and ~2% ash. The corn fiber neutral sugar content varied from 40 to 48%, mostly due to the starch changes. Consequently, the percent distribution of sugars varied: 21–28% arabinose, 30–41% xylose, 4–6% galactose, and 25–38% glucose. Assuming all of the first three sugars and the uronic acids constituted the hemicellulose component of the initial fiber, then an average of 37% of the fiber was in the form of xylan. This number agrees well with previously published values (Wolf et al., 1953; Kies et al., 1982; Osborn and Chen, 1984).

Calcium Hydroxide Extraction of Xylan. On the basis of example three of Rutenberg and Herbst (1957), corn fiber was extracted with 2% (w/v) calcium hydroxide for 16 h at 70 °C. The resultant xylan, 2C-A, was relatively free of protein and other impurities (Table 1). The almost complete absence of glucose indicated the lack of starch. The 2C-A xylan was white in color (Table 2) and dissolved easily in water, and viscous solutions (2% w/v) were visually almost clear and lacked yellowness as indicated by the low optical densities at 520 and

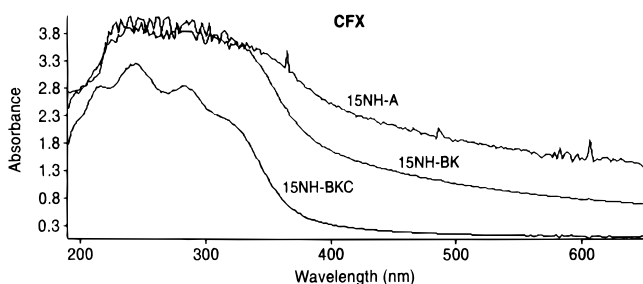


Figure 1. Spectra of CFX isolated by 15% ammonium hydroxide extraction of corn fiber. Material was extracted by ammonium hydroxide (15NH-A) and then reprecipitated after dissolving in 0.5% potassium hydroxide alone (15NH-BK) or also with calcium hydroxide treatment (15NH-BKC).

600 nm (Table 2). Spectral analyses of these solutions confirmed these findings and indicated peak absorbances at 284 and 238 nm. Extensive dialysis of these 2C-A xylan solutions against distilled water did not appear to alter their physical properties, but the 238 nm peak was shifted to 234 nm.

While producing relatively pure xylan with virtually no color, the calcium hydroxide extraction had several disadvantages. Only ~14% of the corn fiber was recovered as xylan. This yield was only ~38% of starting corn fiber xylan content. Extending the extraction time to 20–24 h did not appreciably increase the amount of xylan recovered, whereas shortening the extraction times decreased yields from 14 to 4% with 4–6 h extraction times. Increasing the extraction temperatures to 85–90 °C increased the xylan yields slightly, but resultant tan xylan produced straw-colored solutions. Second, the extracted corn fiber yielded a hard residue that was difficult to resuspend and contained the insoluble calcium hydroxide. Even with gentle (55–75 °C) drying, the residue formed a hard, brittle cake, suggesting it may not be suitable as an animal feed (a normal use of corn fiber) and it could possibly pose disposal problems. To overcome these difficulties, development of other extraction methods was undertaken.

Ammonium Hydroxide Extraction of Xylan. As another gentle means of alkaline extraction, corn fiber was treated with ammonium hydroxide solutions at 70 °C. Initial experiments indicated little, if any, xylan was extracted with 2–5% solutions with treatment times of 4–6 h. Significant xylan was extracted using a 15% solution for 20 h at 70 °C as ~11% of the corn fiber or ~30% of the available xylan was recovered (Table 1). These suspensions were cloudy, produced no clear-cut absorbance spectrum (Figure 1), and eventually produced a precipitate. The 15NH-A xylan contained significant protein, and the low neutral sugar content (Table 1) suggested other materials had been coextracted.

When 15NH-A xylan was suspended in 0.5% potassium hydroxide and heated at 70 °C for several hours, the xylan dissolved to produce a clear brown liquid. Cooling to room temperature, adjusting the pH to 4.5, centrifuging, and then precipitating the supernatant liquid with 3 volumes of cold ethanol yielded the 15NH-BK xylan. This material was light tan in color and contained less protein but about the same amount of neutral sugars (Table 1). The 15NH-BK xylan dissolved in water with warming but still produced a cloudy, brownish-yellow solution that still had no clear-cut absorbance spectrum (Figure 1). The 15NH-BK xylan was then dissolved in 0.5% potassium hydroxide at 70

°C, and dry calcium hydroxide was slowly added to a final concentration of 2% (w/v) with stirring to this solution. After stirring for 2 h, the solution was cooled to room temperature and the calcium hydroxide removed by centrifugation. The supernatant fluid was adjusted to pH 4.5, and the resulting flocculent precipitate was removed by centrifugation. The final 15NH-BKC xylan was obtained by ethanol precipitation of the supernatant fluid. Similar to the 2C-A xylan, this final xylan had high neutral sugar and low protein contents (Table 1). The 2C-A and 15NH-BKC xylylans also had very similar proportions of neutral sugars. The 15NH-BKC xylan was cream colored, dissolved readily in water, and produced clear solutions having low optical densities at 520 and 600 nm. The spectra of 15NH-BKC solutions (Figure 1) were similar to those of 2C-A but had a lower spectrum peak at ~244 nm.

The corn fiber residue after ammonium hydroxide extraction was slightly sticky with a visible fiber texture present. The material could be readily resuspended with a Waring blender and could be gently dried to a flake-like cake. Chemical analysis of this material indicated it contained <2% protein and ~30% pentoses (orcinol positive material). Much of the removed protein probably was associated with the precipitate obtained with the pH 4.5 adjustment, as this material contained ~40–60% protein and 7–10% neutral sugars (data not shown). These results suggest the residual fiber material might still have some feed value or other usefulness. Whereas a highly purified xylan was obtained, the long extraction times plus the reprecipitation treatments needed suggested this ammonia extraction procedure was not suitable for practical purposes.

Potassium Hydroxide Extraction of Xylan. Wolf et al. (1953) examined a variety of methods for extracting xylan from corn fiber using 0.5–3.0% sodium hydroxide, sodium carbonate, or combinations of these solutions, usually at temperatures of 90–121 °C. Good xylan recoveries were obtained, but required four to seven sequential, 1–2 h extractions. The isolated xylylans usually were tan and produced brown to yellow solutions, probably because of the compounds produced by the high temperatures used for extraction. As an alternative approach, attempts were made to extract corn fiber with 10% potassium hydroxide solutions at room temperature (~22 °C). Little xylan was extracted with incubation times of 4–6 h, but overnight extraction for 18–22 h solubilized much of the available xylan. This 10K-A xylan was dark tan in color and produced turbid, light brown aqueous solutions. After dissolving in 0.5% potassium hydroxide at 70 °C, adjusting the pH to 4.5, centrifuging, and precipitating with ethanol, the 10K-BK xylan was obtained. This material had a low neutral sugar content (Table 3) and produced cloudy, straw-colored solutions as indicated by the 520 and 600 nm absorbances (Table 2). However, dissolving the 10K-A xylan in 0.5% potassium hydroxide and treating with dry calcium hydroxide as described above for the 15NH-BKC xylan resulted in producing the 10K-BC xylan that was high in neutral sugars (Table 3), dissolved easily in water to give clear solutions, and possessed a distinct spectrum. The overall yield was ~54% of the initial corn fiber xylan.

To shorten the extraction times, corn fiber was treated at 70 °C with 2% potassium hydroxide for 4–6 h to extract the xylan. The 2K-A xylan obtained was a crude material similar to the 10K-A xylan but could be freed

Table 3. Properties of Corn Fiber Xylans Isolated by Potassium Hydroxide and Calcium Hydroxide Extractions^a

preparation	10K-BK	10K-BC	2K-A	2K-BC	0.56K-A	0.56K-BC
1st extract	10% KOH	10% KOH	2% KOH	2% KOH	0.56% KOH	0.56% KOH
2nd extract	5% KOH	2.5% Ca(OH) ₂		2.5% Ca(OH) ₂		2.5% Ca(OH) ₂
total neutral sugars (%) ^b	65.9	81.0	64.0	93.4	83.2	83.6
distribution of neutral sugars (%)						
arabinose	32.2	33.8	25.9	33.8	33.6	38.9
xylose	58.9	56.8	46.5	51.9	55.4	55.0
galactose	7.1	7.4	10.7	6.5	6.7	4.9
glucose	1.8	1.9	16.8	7.8	4.1	1.1
orcinol sugars (%) ^b	70.9	77.3	68.4	81.3	74.9	88.0
hexuronic acids (%) ^b	6.4	6.7	5.8	7.4	6.8	7.4
protein (%) ^b	4.7	4.7	4.0	2.6	4.5	2.2
recovery (%) ^c	27.9	20.6	18.5	15.4	13.3	8.0

^a See text for details of extraction procedures. ^b Expressed as percent of dry weight of xylan recovered. ^c As percent weight of initial corn fiber based on orcinol sugar content of corn fiber and recovered xylan.

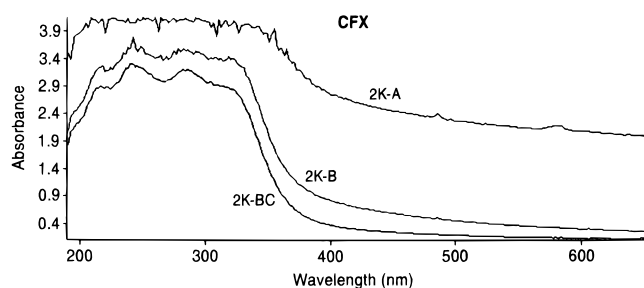


Figure 2. Spectra of CFX isolated by 2% potassium hydroxide extraction of corn fiber. Material was extracted by potassium hydroxide (2K-A) and then reprecipitated after dissolving in 0.5% potassium hydroxide alone (2K-B) or also with calcium hydroxide treatment (2K-BC).

of many impurities by simply redissolving in 0.5% potassium hydroxide and reprecipitating with ethanol as was done above for the 10K-A xylan. The resultant 2K-B xylan dissolved easily in water to give light, straw-colored solutions having a distinct spectrum (Figure 2). However, when the dry calcium hydroxide procedure was also used, a highly pure 2K-BC xylan (Table 3) was obtained; which produced clear solutions of low optical densities (Table 2) having a smooth spectrum (Figure 2). The overall yield of the 2K-BC xylan was ~40% of the initial xylan.

Further extraction experiments were done using even lower concentrations of potassium hydroxide. Using 0.56% solutions with 4 h extractions at 70 °C yielded the 0.56K-A xylan, which could be further purified by redissolving and treating with calcium hydroxide to produce the 0.56K-BC xylan. These two xylans were chemically (Table 3) and physically (Table 2) similar to the 2K-A and 2K-BC xylans, respectively. However, the overall low yield of only 22% of the initial corn xylan suggests this level of potassium hydroxide treatment would not be practical, particularly with large-scale processing.

The corn fiber residue obtained after xylan extraction with potassium hydroxide solutions to obtain the 10K-A or 2K-A preparation contained the bulk of the starting corn fiber material. The residue was somewhat sticky or amorphous, probably due to some contaminating extracted xylan, but the residue also retained a clearly visible fibrous component. After neutralization with dilute hydrochloric acid, the residue could be dried to a dark yellow-brown, somewhat hard material that was brittle and could be broken into small chips or readily ground to a powder. Approximately 40–46% of the residue could be accounted for as neutral sugars, with

a composition and distribution of sugars similar to those found for the untreated corn fiber (data not shown). A major difference was that the protein content was only ~2% compared to ~10% for the starting material. Most likely, much of the missing protein was present in the flocculent material collected when the alkaline extracts were adjusted to pH 4.5 prior to ethanol precipitation of the xylan. Analyses of this material indicated it contained about 60% protein and 4–10% neutral sugars, mainly in the form of arabinose and xylose (data not shown). The high-ethanol-containing liquid obtained after xylan precipitation was clear and dark yellow to brown in color. This liquid could not be dried to a powder but could be evaporated to leave a dark yellow, viscous material, suggesting the presence of oils, carotenoid-like pigments, and other lipid soluble materials.

Summary. Of the various methods examined for extraction of CFX from corn fiber, the procedures used to obtain the 2K-BC preparation (e.g., 2% potassium hydroxide, 70 °C, dry calcium hydroxide treatment) appear to be the most reasonable. The xylan obtained is highly chemically pure (Table 3) and yields clear (Figure 2) solutions. While further refinements in extraction parameters could lead to increased overall xylan recoveries, sufficient quantities can now be produced for research uses. Currently, we are using this CFX-2K-BC material as a model substrate for biochemical studies on the mechanisms and interactions of enzymes (xylanases, arabinofuranosidases, xylosidases) isolated from wild-type xylanolytic microorganisms and microorganisms harboring cloned genes.

As noted by Whistler (1993), CFX has potential applications in producing biofilms and in food uses as a thickener or replacement for other currently used gums. Because the isolation procedures developed here use common chemicals and do not produce any apparent hazardous waste streams, large-scale commercial production of CFX may be possible. Aside from the major use of corn wet-milling to produce starch for other uses, ~900 million gallons of fuel ethanol is currently made from other corn wet-milling processes that use at least 320 million bushels of corn. Assuming 4.5 lb of corn fiber per bushel (Gulati et al., 1996), 1440 million lb of corn fiber would be made as a byproduct. If only 20% of this corn fiber were used for CFX production with 15% recovery (Table 3), about 43 million lb of xylan is potentially available from fiber made in the conversion of corn to ethanol alone. Further studies are needed to evaluate the physiochemical properties of CFX and the costs of its production, because apparently the supplies of the starting raw materials seem quite adequate.

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