Isolation, characterization and the functional properties of cellulosic arabinoxylan fiber isolated from agricultural processing by-products, agricultural residues and energy crops

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ABSTRACT

Water insoluble cellulosic arabinoxylan fibers were prepared from low-valued agricultural processing by-products, agricultural residues and energy crops by removing the alkali-extractable hemicelluloses and processing the alkali insoluble material under high temperature and high shear conditions. The yield of these fibrous plant materials varied from 14.3 to 59.90 % and most of them contained more than 90% insoluble dietary fiber. Sugar composition and glycosyl-linkage analysis show that they are composed of cellulose and arabinoxylans indicating that they have a typical cellulosic arabinoxylan structure. They have

very high water holding capacity varying from 6.374 to 74.33 g/g (water/fiber), indicating their usefulness in many food system applications as moisture management aids and texturizing agents. Their ORAC values vary from 352 to 1560 μ mole Trolox/100 gram showing that they have the ability to provide antioxidant activity in foods, as well as offering other functionality and health-promoting benefits of dietary fiber.

Keywords: Insoluble fiber, agricultural processing by-products, water holding capacity, antioxidants, rheology.

1. Introduction:

Lignocellulosic materials are abundant and renewable biological resources, which contain numerous components that could be valuable products if they are economically isolated and marketed. Along with the three major polymeric components, namely cellulose, hemicellulose, and lignin, lignocellulosic materials also contain other minor components, such as extractives (phenolics, lipids, etc.), pectins and proteins (Zhang et al., 2004 and Fengel et al., 1984). Corn fiber/bran, a renewable resource available in a large quantity, can be a good source of valuable consumer products. Corn fiber makes up about 5 to 10 wt. % of the total weight of the corn kernel. Primarily, it consists of residual starch (10 to 20 wt. %), hemicelluloses (40 to 50 wt. %), cellulose (15 to 25 wt. %), phenolic compounds (3 to 5 wt. %), protein (5 to 10 wt. %) and some oils (Wolf et al., 1953, Chanliaud et al., 1995, Yadav et al., 2006, 2007 a, b, 2010). Byproducts obtained from agricultural processing operations, such as corn stover, rice straw and cereal brans, are rich in lignocellulose. Fibers isolated from these sources could be useful in numerous applications across a wide range of industries, including the food, biofuels, cosmetics, medicine, packaging, construction and animal feed sectors (Reddy and Yang, 2005). The food industry, in particular, is a large potential market for products made from lignocellulosic biomasses, which consist of nutritionally beneficial dietary fibers with good functional properties. Dietary fiber-based ingredients are often used as viscosity building additives and bulking agents in foods, which impart food products with desirable mouth feel and texture while decreasing fat and calorie-content and providing nutritional benefits in the form of increased satiety, improved blood glucose and cholesterol levels, reduced gut transit times and stool bulking (Gray and Tainter 1941; Faivre and Boutron 1991; Reddy et al., 1989; Buttriss and Stokes, 2008), among others. The preparation of insoluble dietary fiber-based bulking agents from corn bran and oat

hulls has been reported previously (Inglett, 1998, Inglett and Carriere, 2001). These materials were reported to have high water holding capacity and viscosity, and serve as fat replacers in food formulations.

In this study, highly functional insoluble fibers were isolated from many agricultural sources using an improved process consisting of very high shear in the presence of alkali, and their chemical composition and rheological and functional properties were thoroughly evaluated. These cellulosic arabinoxylan fibers show great promise as health promoting and highly functional bulking and viscosity building agents for use in the food and non-food industries.

2. Materials methods

2.1. Materials

Plant materials were obtained from various sources as follows: Corn bran (Bunge Milling, Inc., St. Louis, MO), Corn stover (Lawrence Shrawder, a Private Farm, Kempton, PA), Rice fiber (SunOpta Ingredients, Cedar Rapids, IA), Wheat bran (Deibel Laboratories, Bethlehem, PA), Wheat straw (Hougar Farms LLC, Coatesville, PA), Switch grass (ARS lab at University Park, PA), Miscanthus (University of Illinois at Urbana-Champaign, Champaign, IL), Sugarcane bagasse (Southern Regional Research Center, ARS, USDA, New Orleans, LA), Sorghum brans (Nu Life Market, Scott City, KS), Barley hulls (Osage Bio-Energy, Glen Allen VA), Barley straws (Foundation Seed Farm of the Virginia Crop Improvement Association in Mt. Holly, Virginia) and Carrot pomace (Grimmway Farms, Bakersfield, CA). They were ground to a 20-mesh particle size using a Wiley mill. Termamyl[®] was a gift from Novozymes, Inc., (Davis, CA). Sodium hydroxide and hydrochloric acid were obtained from Sigma-Aldrich (St. Louis, MO). All chemicals were reagent grade.

2.2. Standard proximate analyses

Protein ($N \times 6.25$) and ash contents of all samples were determined using AACC Approved Methods 46-30 and 08-01 respectively (AACC International, 1995). Crude fat content was determined using the ANKOM XT10 Extractor with hexane as the extracting solvent. Starch content was determined using a total starch assay kit (Megazyme, Inc., Wicklow, Ireland) based on the use of thermostable α -amylase and amyloglucosidase (McCleary et al., 1997).

2.3. Dietary fiber determination

Insoluble dietary fiber (IDF), soluble dietary fiber (SDF), total dietary fiber (TDF) and neutral detergent fiber (NDF) in CAF were determined following the standard A2000 automated fiber analyzer procedure (ANKOM Technology, 2011).

2.4. Preparation of cellulosic arabinoxylan fiber

The plant material (200 g) was added to mechanically stirred 1150 mL hot water (85 °C). The pH was adjusted to about 6.8 by adding 50% sodium hydroxide solution. 2 g of α -amylase (Termamyl®) was added and stirred for 1 hour to hydrolyze starch. The pH of the slurry was raised to 11.5 by adding 50% sodium hydroxide and stirred using a mechanical stirrer at 85 °C for an additional 30 minutes to completely deconstruct the material. During the reaction, pH was maintained at 11.5 by adding 50% NaOH and the reaction volume was maintained by adding water as needed to compensate water loss due to evaporation. The hot slurry of the deconstructed material was immediately sheared using a high speed Polytron (PT 10/35 GT) equipped with 12 mm probe (Kinematica Inc., Bohemia, NY) at 10,000 rpm for 1 hour. The solid residue was separated from the reaction mixture by centrifugation at 14,000 X g for 10 minutes, suspended in 2 L boiling water and stirred using a mechanical stirrer for 5 minutes. The hot suspension was again sheared at 10,000 rpm for 5 minutes. The sheared material was allowed to cool at room temperature and centrifuged at 14,000 X g for 10 minutes to separate the solid, and hot water washing and shearing cycles were repeated till a clear supernatant was obtained. For most of materials two repetitions (three total washings) were needed to get a clear supernatant. The final solid residue was collected, suspended in water and dried by drum or spray drying.

2.5. Determination of sugar composition

The sugar composition of CAF samples was determined using National Renewable Energy Laboratory Analytical Procedure (Chiu et al., 2008). Briefly, 0.3 g sample was suspended in 3 mL 72% (w/w) sulfuric acid in a test tube, mixed and incubated at 30 °C for 1 h with intermittent stirring every 5 to 10 minutes. The sample suspension was transferred into a 100 mL Pyrex glass bottle using 84 mL water, which also diluted the acid to 2.48%. The bottles containing samples and standard sugars were autoclaved at 121 °C for 1 h. After cooling to room temperature, aliquots of 5 ml were taken and their pH was adjusted between 5 and 7 followed by centrifugation. The supernatant was filtered and the filtrate was analyzed for sugars using Agilent 1200 HPLC that included a BioRad Aminex HPX-87H column at 60 °C and an RI detector. The mobile phase consisted of isocratic 5 mM H₂SO₄ eluent at a flow rate of 0.6

mL/min. The quantification of each sugar was done using the calibration curve of standard sugars prepared under the same conditions.

2.6. Determination of glycosyl-linkage composition

The sugar linkage composition of the CAFs was determined by gas chromatography-mass spectrometry (GC-MS). The sample was permethylated, depolymerized, reduced and acetylated. The resulting partially methylated alditol acetates (PMAAs) were analyzed by GC-MS (Heiss *et al.*, 2009; York *et al.*, 1986). Briefly, about 1 mg of the sample was suspended in 200 µl Dimethyl Sulfoxide (DMSO) and stirred for 3 days, followed by treatment with sodium hydroxide (15 min) and methyl iodide (45 min) at room temperature, repeated twice. The permethylated polymer was hydrolyzed with 2 M TFA (2 hours in a sealed tube at 121 °C), reduced with NaBD₄ and acetylated using acetic anhydride/TFA. The resulting partially methylated alditol acetates (PMAAs) were analyzed by GC-MS (Agilent 7890A GC interfaced to a 5975C mass selective detector in electron impact ionization mode). The separation was performed on a 30 meter Supelco SP-2331 bonded phase fused silica capillary column.

2.7. Determination of water holding capacity

The water holding capacity of CAF was determined following the published procedure (Elhardallou & Walker, 1993) and AACC method 88-04 (AACC, 1995) with some modification. Briefly, 0.5 g CAF sample in triplicates was weighed in a polypropylene centrifuge tube with screw cap. To each tube, 24.5 mL distilled water were added and the sample was sheared using a high speed polytron at 10,000 rpm for 2 minutes and at 15,000 rpm for 1 minute. The tubes were placed on shaker at room temperature and shaken at a moderate speed for about 24 hours. Then they were centrifuged at 1,000g for 15 minutes. After decanting the excess water, the tubes were inverted to completely remove any residual water. Each tube was weighed, and the amount of water held was calculated by subtracting the weight before water treatment and the water holding capacity (WHC) of the sample was calculated using the following equation:

$$WHC = \frac{g_{water}}{g_{sample}} \times 100$$

where g_{water} (g) and g_{sample} (g) are the weight of water held by the sample and the original weight of dry sample respectively.

2.8. Determination of antioxidant activity

Antioxidant activity of the CAF samples was determined using the ORAC assay, and expressed as Trolox equivalents/100 g sample. The ORAC values of CAF were determined using the published procedure (Huang et al., 2002; Ou, B., et al., 2002).

2.9. Rheological studies

Rheological properties of CAF samples were measured using a rotational rheometer (Anton Paar Modular Compact Rheometer 102, Anton Paar GmbH, Ashland, VA) with a concentric cylinders geometry. The samples were prepared by shearing 4 % w/w suspensions of the CAF in water at 15000 rpm for 3 min followed by 10000 rpm for 2 min using a Polytron mixer (Kinematica Inc., Bohemia, NY). Flow curves of the samples were developed by measuring viscosity at different shear rates ($0.13 - 130 \text{ s}^{-1}$). Power Law fitting of the flow curves was carried out, using Microsoft Excel, to determine flow consistency index and flow behavior index.

3. Results and discussion

3.1. Preparation of cellulosic arabinoxylan fiber

Cellulosic arabinoxylan fiber (CAF) was prepared from ground plant materials including agricultural processing byproducts such as sorghum bran, corn bran, wheat bran, rice fiber, barley hulls, sugarcane bagasse, carrot pomace, agricultural residues such as corn stover, wheat straw, barley straw etc. and energy crops such as switchgrass and miscanthus according to the scheme shown in Fig. 1. Alkali-soluble hemicelluloses were removed by mechanically stirring the de-starched plant materials in alkaline solution (pH ~ 11.5) at 85 °C. The alkali insoluble CAF was recovered from the deconstructed reaction mixture by centrifugation. The recovered material was re-suspended in hot water and sheared using high speed homogenization. The suspension of the solid material in hot water and its heating and shearing were repeated till a clear supernatant was seen. Then the final solid residue was collected, suspended in water and dried by drum or spray drying to obtain the final product “CAF”.

The amount of CAF isolated from different plant materials, following the scheme given in Figure 1, varied from 14.30 to 59.90% (Table 1). Rice fiber had the highest percent of CAF, which may be attributed to prior alkaline treatment by the manufacturer. Corn stover, wheat straw, miscanthus, sugarcane bagasse, and barley straw yielded more than 40% CAF, making them all promising feedstocks for commercial-scale production. The remaining plant materials gave less than 20% yield, but could potentially be used for commercial CAF production if they are readily available, especially if the hemicellulose fractions can also be utilized.

3.2. Proximate composition and dietary fiber analysis

Table 2 shows the proximate composition of CAF isolated from all sources. The ash content in these samples was less than 3% except corn stover (7.29%) and sugarcane bagasse (9.90%). Their protein content varied from 0.56 to 2.20 except CAF from sorghum bran, which was rich in protein (5.37 to 23.82%). CAF from sorghum bran had 0.91 to 5.42% residual starch but for all other samples the starch content was less than 0.36%. None of the samples contained significant amounts of fat. As expected, the samples were very rich in neutral detergent fiber (95.50 to 99.75%). The only exception to this was the CAF from sorghum bran, which had a greater amount of residual starch which could explain the lower fiber content. In most of the samples, more than 90% of the fiber present was insoluble dietary fiber. CAF from corn bran, corn stover, sorghum bran (black milled), sorghum bran (burgundy milled), barley hulls/straws and carrot pomace showed some soluble dietary fiber which could be due to presence of some water soluble carbohydrate. It is clear that CAFs isolated from all the plant materials were almost pure dietary fiber. They are predicted to be non-caloric in human diets, and should be very useful for making non-caloric food products.

3.3. Sugar composition of CAF

Table 3 shows that the neutral sugars present in CAF from all plant materials are glucose, xylose and arabinose, clearly demonstrating that it has a typical cellulosic arabinoxylan structure. While it is known that the exhaustive extraction of arabinoxylans from cereal cell walls using alkali is not achieved even by very harsh alkaline treatments (Maes and Delcour, 2001), the insoluble residue was previously thought to contain primarily glucose (cellulose) (Inglett and Carriere, 2001) with little to no arabinose and xylose. The amount of glucose varied from about 34.78- 73.87%, while xylose and arabinose contents ranged from 11.69-31.97%, and 2.04- 13.17% respectively. The true structure of this cellulosic arabinoxylan fiber with respect to the type of association, if any, between the cellulose and arabinoxylan polymers, is not currently known. However, the presence of significant amounts of arabinoxylan in spite of the rigorous purification process used (Figure 1) suggests the presence of very strong covalent or non-covalent associations between the two polymers.

3.4. Glycosidic-linkage composition of CAF

The glycosidic-linkage composition of CAF isolated from five selected sources (corn bran, rice fiber, wheat straw, miscanthus and sugarcane bagasse) were analyzed (Table 4). CAF from corn bran contained 55.5 % of (1→4)-linked glucose residues, representing cellulose. Most of

xylose residues were present as (1→4)-linked xylopyranose (Xylp) backbone, which was highly branched on its C-2 and C-3 positions. The total percent of unbranched and branched (1→4) linked Xylp was 17.4 %. The presence of significant amounts of terminal arabinose, xylose, galactose and glucose (5.1, 5.2, 2.9 and 6.1 % respectively) indicates that the arabinoxylan in corn bran CAF has a highly branched structure, which may contribute to some of its functional properties such as water holding capacity and viscosity. Table 4 also shows the glycosidic-linkage analysis results for CAF from rice fiber, wheat straw, miscanthus and sugarcane bagasse showing results similar to CAF from corn bran with 53.3 to 66.8% of (1→4)-linked glucose residue and about 23.7 to 32.8% of (1→4) linked Xylp residue as the major sugars. It is clear that as in CAF from corn bran, the major portion of carbohydrate polymer in all these CAF samples have a cellulose-like sugar backbone structure. It also demonstrates that though it has a cellulose-like sugar backbone, it does not have a fully cellulose-like structure. It also contains a high percentage (23.7 to 32.8%) of xylan in addition to glucose and some arabinose. Unlike CAF from corn bran, the Xylp present as (1→4)-linked backbone in CAF from these four sources are not highly branched as indicated by their low percent of terminal arabinose, xylose and glucose. The percentage of sugars linked at carbons 2, 3 or 6 position in all of these four samples are lower than CAF from corn bran, which further confirms that they do not have a very highly branched structure. From these results, the nature of the association between two main carbohydrate polymers [(1→4)-linked Xylp (xylan) and (1→4)-linked glucan] is still not clear. The less branched structure of arabinoxylan polymer in these materials than that in corn bran CAF may be associated with the lower water holding capacity and viscosity of their suspensions as discussed in following sections.

The glycosyl-linkage analysis of CAF from carrot pomace was also performed (data not shown), and was found to be significantly different than that of the other CAFs. It contains a high percentage of (1→4)-linked glucose residues (67.8%), but its (1→4)-linked Xylp content (5.1%) was lower than CAFs from other sources. It also showed a low amount of terminal sugars and less sugars linked at carbons 2, 3 and 6 positions, showing less branched structure than CAF from corn bran.

3.5. Water holding capacity

Table 5 shows the water holding capacity (WHC) of CAF from the various sources. WHC is known to vary depending upon the carbohydrate composition, branching and molecular structure of the fiber. All of the CAF samples studied showed high water holding capacity, with remarkable variation in the WHC depending upon source and also on the process of drying from

its slurry into solid form. The WHC of these fibers varied from 6.374 to 34.81 g/g (water/fiber), with rice fiber CAF showing the lowest WHC. The WHC of CAF from corn bran was much higher than the CAF from all other sources, possibly owing to the unique highly branched structure of its constituent polymers. The drying process to make CAF also had an effect on its WHC as seen in drum and spray dried material from corn bran. The WHC of spray dried CAF from corn bran was much higher (74.33 g/g) than the drum dried product (32.767 g/g). This could be due to the difference in the particle morphology caused by the two drying techniques, leading to better hydration in the spray dried CAF particles than the drum dried material. This difference was also apparent in the rheological properties of the CAF samples, discussed in section 3.7.

3.6. *Antioxidant activity*

Lignocellulosic materials are known to contain significant amounts of phenolic compounds, which can contribute to antioxidant activity in dietary fibers isolated from these materials. Table 6 shows the ORAC values of CAF isolated from different sources. It is clear that all the CAF samples retained a considerable amount of phenolic compounds, leading to significant antioxidant activity in the range of 326 to 1560 $\mu\text{mol Trolox}/100$ gram sample. There is some evidence that the sugar composition, linkages and structural features of polysaccharides may also influence their antioxidant properties (Lo et al., 2010; Chattopadhyay, et al., 2010; Rao et al., 2006). Arabinose in the side chains and unsubstituted and/or monosubstituted xylose on CAF may also contribute to antioxidant activity. Drum dried CAF from corn bran had the highest ORAC value of 1560 Trolox/100 grams (Table 6). A higher ORAC value of drum dried corn bran CAF than the spray dried material from the same source shows that there is an effect of drying method on ORAC value. The reason for this is unknown but may be related to accessibility of phenolic compounds on the fiber. CAFs from wheat bran, sorghum brans, switchgrass, barley hulls and rice fiber had ORAC values above 600 $\mu\text{mole Trolox}/100$ grams. The ORAC value in the CAFs from the remaining materials was less than 500 $\mu\text{mole Trolox}/100$ grams, which is still significant from the nutraceutical perspective. Thus it is clear that CAFs from these plant sources have the ability to provide antioxidant activity in foods in addition to other health-benefits as an insoluble, bulking dietary fiber.

3.7. *Rheological properties*

All CAF suspensions showed very high viscosity at 4% w/w and even at lower concentrations (data not shown). The apparent viscosity of the samples at a shear rate of 1.3 s^{-1} ranged widely

from 0.418 Pa.s for rice fiber CAF to 29.1 Pa.s for the spray dried corn bran CAF. This wide range is probably a result of the different sugar compositions of the materials as well as the branching of the constituent arabinoxylan polymers. In general, the samples with higher arabinoxylan content and more branching showed higher viscosity values. This data correlated well with the WHC of the samples, with the samples that had high WHC showing correspondingly high viscosity.

All the samples also showed shear thinning behavior (Table 7), with flow consistency index (K) values ranging from 0.727-36.45 Pa.sⁿ and flow behavior index (*n*) values ranging from 0.25-0.5. The low values of *n* indicate that all of the CAF samples showed a significant degree of shear thinning, implying that even though they are highly viscous materials at low shear rates, they lose much of their viscosity at higher shear rates. Such high degree of shear thinning can be an advantage in food processing operations, where these CAF suspensions can be used to impart high viscosity to the product while still being easy to pump at high shear rates.

It is interesting to note that the *n* value was affected by the drying method, with drum dried and spray dried corn bran CAFs showing *n* values of 0.5 and 0.25 respectively. It is likely that the difference in particle morphology of the materials had an effect on the particle size and surface characteristics in the swollen state, leading to different levels of inter-particle friction and particle orientation in the flow field.

In addition to the difference in apparent viscosity, K and *n* values, the drying method also seems to affect the hydration of the particle and the effect of shearing on the viscosity of the suspension. Figures 2(a and b) show comparisons of the flow behavior of 4 % suspensions of drum dried and spray dried CAF from corn bran and sorghum bran (Burgundy milled), prepared with and without shearing. It is clear that, in both cases, the drum dried CAF did not show high viscosity when prepared without any shearing (empty squares). When these samples were sheared for 3 minutes at 15000 rpm followed by 2 minutes at 10000 rpm, the viscosity increased significantly (filled squares). The spray dried CAF from both sources showed very high viscosity (equivalent to drum dried samples with shear) even without shearing (empty triangles). All samples were allowed to hydrate for at least 3 hours before measurement of viscosity. When the spray dried samples were sheared, the viscosity increased even further (filled triangles). Thus it is clear that the drum dried CAF samples required shearing along with hydration in order to give high viscosity. Spray dried samples, on the other hand, showed high viscosity even with no shearing if they were allowed to hydrate sufficiently. Shearing of these spray dried samples lead to even higher viscosities which could not be obtained with drum dried samples at the same level of shearing. Thus, drying method has a significant effect on the rheological properties of

CAFs, with spray dried samples showing more shear thinning and higher viscosity even without shearing as compared to drum dried CAF.

4. Conclusion

Cellulosic arabinoxylan fiber (CAF) is a highly functional value-added product that can be prepared with high yields (14.3 to 59.90 %) from many low value agricultural processing by-products, agricultural residues and energy crops. The CAFs consisted almost entirely of dietary fiber, making them suitable as non-caloric bulking and texturizing agents in food products. These water insoluble materials are rich in glucose and also contain significant amounts of xylose and arabinose, in the form of arabinoxylan polymers, accounting for almost half of the total carbohydrate in some cases. The cellulose and arabinoxylan polymers are strongly associated, but nature of the association is not clear. CAF from corn bran had a highly branched structure, and also showed the highest ORAC value among all the samples. Corn bran CAF also gave the highest values of water holding capacity (WHC) and viscosity. CAFs from other sources also showed high values of WHC and viscosity, and they all showed shear thinning flow behavior. The method used for drying the CAF (drum drying versus spray drying) had a significant effect on the WHC, viscosity and flow behavior; with spray dried CAF showing higher WHC and higher viscosity even without shearing as compared to drum dried CAF from the same source. Owing to their high functionality and non-caloric nature, CAFs produced from low-valued agricultural by-products are a very interesting and commercially valuable class of health-promoting, insoluble dietary fiber based bulking agents, viscosifiers and texturizing agents for use in the food industry and beyond.

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7. Tables

Table 1: Isolation of CAF from Plant Materials (% Yield, dry weight basis) ^a

<u>CAF Sources</u>	<u>% Yield</u>
Corn bran	20.05
Corn stover	40.20
Rice fiber	59.90
Wheat bran	14.30
Wheat straw	53.10
Switchgrass	32.70
Miscanthus	56.70
Sugarcane bagasse	52.00
Sorghum bran (Black)	25.84
Sorghum bran (Sumac)	38.50
Sorghum bran (Burgundy)	19.32
Barley hulls	26.85
Barley straws	49.75
Carrot pomace	14.80

^aWeight percent based on de-starched biomass

Table 2: Proximate Composition and Dietary Fiber Content of CAF Isolated from Plant Materials (d.w.b.)

CAF Sources	Ash	Protein	Starch	NDF	Crude Fat	ISD	SDF	TDF
Corn bran	1.54	0.56 ± 0.00	0.26 ± 0.01	92.58 ± 0.35	0.21	92.44	4.09	96.54
Corn stover	7.29	1.29 ± 0.00	0.16 ± 0.08	96.92 ± 0.10	0.10	95.30	0.89	96.20
Rice fiber	2.25	0.91 ± 0.05	0.21 ± 0.04	95.11 ± 0.72	0.04	98.63	0.00	98.63
Wheat bran	1.05	1.72 ± 0.08	0.36 ± 0.03	90.48 ± 0.41	0.00	92.03	0.00	92.03
Wheat straw	2.27	1.75 ± 0.00	0.05 ± 0.03	99.75 ± 0.21	0.36	97.55	0.00	97.55
Switch grass	1.35	1.90 ± 0.04	0.09 ± 0.02	98.22 ± 0.31	0.48	99.45	0.00	99.45
Miscanthus	1.08	0.56 ± 0.05	0.16 ± 0.03	98.71 ± 0.15	0.14	99.61	0.00	99.61
Sugarcane bagasse	9.90	1.91 ± 0.00	0.06 0.04	98.55 ± 0.41	0.25	90.82	0.00	90.82
Sorghum bran (Black)	1.76	16.49 ± 0.09	2.51 ± 0.05	87.06 ± 0.14	0.40	77.88	1.98	79.86
Sorghum bran (Sumac)	1.07	23.82 ± 0.23	5.42 ± 0.24	70.04 ± 0.85	0.28	77.62	0.00	77.25
Sorghum bran (Burgundy)	1.63	5.37 ± 0.06	0.91 ± 0.09	94.70 ± 0.18	0.28	91.80	9.44	101.8 9
Barley hulls	1.00	0.89 ± 0.04	0.09 ± 0.08	96.54 ± 0.29	0.15	98.51	4.15	102.6 5
Barley straws	1.04	0.66 ± 0.03	0.11 ± 0.04	99.34 ± 0.39	0.44	97.57	6.84	104.4 1
Carrot pomace	1.79	2.20 ± 0.07	0.22	95.50 ± 0.59	0.70	94.70	1.4	96.20

Table 3: Sugar composition of CAF Isolated from Plant Materials

CAF sources	Sugar content (Wt. %)			
	Glucose	Xylose	Arabinose	Total
Corn bran (Drum Dried CAF)	52.18	31.56	12.29	96.03
Corn bran (Spray Dried CAF)	51.60	31.08	12.00	94.68
Corn stover	66.87	21.9	2.23	91.00
Rice fiber	64.67	20.47	2.04	87.18
Wheat bran	34.78	31.97	13.17	79.92
Wheat straw	60.72	23.30	2.69	86.71
Switch grass	59.65	24.02	2.81	86.48
Miscanthus	60.74	21.39	2.33	84.46
Sugarcane bagasse	57.99	23.33	2.31	83.63
Sorghum bran (Black)	46.34	14.03	10.48	70.85
Sorghum bran (Sumac)	54.79	11.69	7.87	74.35
Sorghum bran (Burgundy)	59.34	17.10	11.49	87.93
Barley hulls	52.89	31.66	5.74	90.29
Barley straws	66.70	21.74	2.52	90.96
Carrot pomace	73.87	14.46	2.38	90.71

Table 4: Glycosyl linkage composition of CAFs from different sources

Glycosyl residue linkage	Relative peak area (%)				
	Corn bran CAF	Rice fiber CAF	Wheat straw CAF	Miscanthus CAF	Sugarcane bagasse CAF
t-Araf	5.1	1.2	2.4	1.8	1.7
t-Xylp	5.2	0.9	1.1	0.8	0.8
2-Araf	2.1	0.2	0.1	0.1	0.1
t-Glcp	6.1	2.0	1.5	1.8	0.8
3-Araf	2.0	0.1	0.1	0.1	0.1
t-Galp	2.9	0.2	0.2	0.1	0.1
3-Xylp & 4-Arap or 5-Araf	0.8	0.1	0.2	0.2	0.2
4-Xylp	5.9	23.7	26.1	24.3	32.8
3-Glcp	0.7	n.d.	0.7	0.4	1.0
3-Galp	0.3	n.d.	0.1	n.d.	n.d.
4-Manp	0.7	0.1	n.d.	0.2	0.2
4-Galp	n.d.	n.d.	n.d.	0.1	0.1
6-Glcp	0.1	n.d.	0.1	n.d.	0.1
4-Glcp	55.5	66.8	60.3	65.2	53.3
2,4-Xylp	n.d.	n.d.	0.8	0.6	1.1
3,4-Xylp	7.7	1.7	2.9	2.3	3.3
3,4-Glcp	0.8	0.8	0.9	0.6	1.0
2,4-Glcp	0.4	0.7	0.9	0.5	1.1
4,6-Glcp & 2,3,4-	3.8	1.2	1.6	1.1	2.1

Xylp					
Total	100.0	100	100	100	100

n.d. – not detected

Table 5: Water Holding Capacity of CAF Isolated from Plant Materials

CAF Sources	Water Held/Sample(g/g)
Corn bran (Drum Dried CAF)	32.8 $\pm$ 1.0
Corn bran (Spray Dried CAF)	74.3 $\pm$ 4.5
Corn stover	17.7 $\pm$ 1.3
Rice fiber	6.4 $\pm$ 0.4
Wheat bran	13.9 $\pm$ 0.5
Wheat straw	23.7 $\pm$ 0.4
Switchgrass	19.8 $\pm$ 0.7
Miscanthus	16.8 $\pm$ 0.5
Sugarcane bagasse	17.3 $\pm$ 0.7
Sorghum bran (Black)	20.0 $\pm$ 0.7
Sorghum bran (Sumac)	20.1 $\pm$ 0.2
Sorghum bran (Burgundy)	32.8 $\pm$ 1.0
Barley hulls	13.8 $\pm$ 0.3
Barley straws	21.4 $\pm$ 0.6
Carrot pomace	22.9 $\pm$ 1.9

Data are average of triplicate analysis with $\pm$ SD.

Table 6: ORAC Value of CAF Isolated from Plant Materials

CAF Sources	ORAC Value (μmole Trolox/100 gram)
Corn bran (Drum Dried CAF)	1560
Corn bran (Spray Dried CAF)	868
Corn stover	362
Rice fiber	414
Wheat bran	954
Wheat straw	4.93
Switchgrass	649
Miscanthus	392
Sugarcane bagasse	427
Sorghum bran (Black)	633
Sorghum bran (Sumac)	7.92
Sorghum bran (Burgundy)	938
Barley hulls	679
Barley straws	326
Carrot pomace	352

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591 **Table 7:** Rheological properties (apparent viscosity and power law parameters) of 4 %
592 suspensions of CAF Isolated from Plant Materials

	Apparent viscosity at shear rate of 1 s^{-1} (cP)	Flow consistency index “K” (Pa.s ⁿ)	Flow behavior index (n)
Corn bran (Drum Dried CAF)	8500	10.45	0.50
Corn bran (Spray Dried CAF)	29100	36.45	0.25
Corn stover	1900	2.72	0.36
Rice fiber	418	1.04	0.41
Wheat bran	639	0.78	0.41
Wheat straw	7770	11.11	0.26
Miscanthus	1760	2.43	0.36
Sugarcane bagasse	5730	6.97	0.27
Sorghum bran (Black)	1030	1.49	0.46
Sorghum bran (Sumac)	7250	9.35	0.45
Sorghum bran (Burgundy)	4750	6.36	0.43
Barley hulls	540	0.727	0.38
Barley straws	3450	4.93	0.32
Carrot pomace	8620	13.32	0.26
Sugar-beet bagasse	11700	18.31	0.32

8. Figure captions

Figure 1 shows a generic scheme for the preparation of cellulosic arabinoxylan fiber (CAF).

Figure 2(a) shows the effects of drying technique and shearing on corn CAF viscosity:
Comparison of drum dried (DD) and spray dried (SD) CAF suspensions prepared with and
without shear.
Figure 2(b) shows the effects of drying technique and shearing on sorghum CAF viscosity:
Comparison of drum dried (DD) and spray dried (SD) CAF suspensions prepared with and
without shear. Data are average of three runs with error bars.

9. Figures

Figure 1

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638 **Figure 2(a)**

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641 **Figure 2(b)**

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Captions

Figure 1 shows a generic scheme for the preparation of cellulosic arabinoxylan fiber (CAF).

Figure 2(a) shows the effects of drying technique and shearing on corn CAF viscosity: Comparison of drum dried (DD) and spray dried (SD) CAF suspensions prepared with and without shear.

Figure 2(b) shows the effects of drying technique and shearing on sorghum CAF viscosity: Comparison of drum dried (DD) and spray dried (SD) CAF suspensions prepared with and without shear. Data are average of three runs with error bars.