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An investigation of the efficacy of corn cob hemicellulose as a gas barrier and dry strength agent on previously uncoated linerboard

Zachary Witherspoon Western Michigan University, westernmichiganwitherspoon@gmail.com

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AN INVESTIGATION OF THE EFFICACY OF CORN COB HEMICELLULOSE AS A GAS BARRIER AND DRY STRENGTH AGENT ON PREVIOUSLY UNCOATED LINERBOARD

Zachary B. Witherspoon

April 2018

Abstract

Two types of xylan, water soluble and insoluble, were extracted from corn cobs and made into liquid coatings and applied to uncoated linerboard with a fine, #8 Meyer rod, with the intention of creating a gas barrier and increasing strength properties. The xylan coatings were plasticized with sorbitol and crosslinked with citric acid, and the crosslinking reaction catalyzed with sodium hypophosphite. A 1% konjac glucomannan comparison coating was created (with the same plasticizer and crosslinker) to serve as an alternative coating based on previous research in the department, but it failed as a paper coating due to the presence of glucomannan globules that created a wildly inconsistent coating. The coating that combined both types of xylan exhibited the best gas barrier properties, with little changes in physical properties, and drastic hygroscopic water vapor uptake. However, the coating made from pure isolated water-soluble xylan did nothing to create a gas barrier but mildly improved Mullen Burst strength, with little hygroscopic uptake.

AN INVESTIGATION OF THE EFFICACY OF CORN COB HEMICELLULOSE AS A MOISTURE BARRIER AND DRY STRENGTH AGENT ON PREVIOUSLY UNCOATED LINERBOARD

by

Zachary B. Witherspoon

A project submitted to the College of Engineering and Applied Sciences in partial fulfillment of the requirements for the degree of Bachelor of Engineering Paper Engineering: Process Western Michigan University April 2018

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Introduction

In the modern paper industry, there are many fillers and coating additives that are derived from petroleum. The crude oil price spikes of the previous decade made it very clear not just to drivers, but to manufacturers that the price of crude is subject to drastic changes with little to no warning. Whether this is brought on due to supply changes, international politics, or new regulations and taxes, it can make previously economically viable processes in any industry suddenly generate a new and uncomfortable deficit that may endanger the bottom line, in a moment's notice. Furthermore there are environmental consequences to moving large quantities of carbonaceous material that was previously sequestered underground and releasing it into economic circulation. Ultimately, these products either end up in the air as $CO₂$, in the water systems, or in the trash heap the size of Texas that is currently floating in the Pacific (ABC News, 2018). Therefore, it is in the best interest of the paper industry and the global economy as a whole to seek natural alternatives to solve problems that were previously forgotten about altogether with the global explosion of petroleum product usage.

Meanwhile, when one considers all of the biomass that sometimes goes to waste on farms of the world – wheat straw, maize stalks, bean stems, and many more – it is very quickly apparent that there is a remarkable abundance of unused plant matter on earth, and much of it just decomposes in the fields. Most of this plant matter contains lignin, cellulose, and hemicellulose as the major components. This investigation looks at hemicellulose, a catch-all term for many different variously branched polysaccharides that help to hold plants together via lignin-saccharidic bonds. The particular species of hemicellulose targeted in this study is xylan, an unusually linear hemicellulose present in high concentration in corn cobs.

The xylan examined in this project is subdivided into two groups: water-soluble and insoluble. Both of these subspecies are present in corn cobs and they can be separated, as insoluble xylan is soluble in highly-alkaline solution and insoluble at pH<7. This project will examine the effect of a coating containing both in emulsion, and compare that to a coating containing only the water-soluble portion.

In order to form an effective gas barrier from a branched polysaccharide like corn cob xylan, the intention is to induce a polysaccharide lattice network via conversion of the many freely available primary and secondary OH groups into ester and ether linkages via the addition of a polycarboxylic acid as a sort of bridge structure between branched chains. An illustration of this concept is in Figure 1.

Figure 1. Mechanism of action of SHP-catalyzed citric acid hemicellulose crosslinking (Salam, Venditti, Pawlak, & Tahlawy, 2011)

Citric acid has been shown to be an effective crosslinker for polysaccharides, especially when catalyzed with sodium hypophosphite (Wang, Ren, Li, Sun, & Liu, 2014). Even without the hemicellulose, citric acid crosslinking has already been shown to increase the strength properties of corrugated board in high-humidity environments via the same mechanism on cellulosic hydroxyl groups in paper fibers (Widsten, Dooley, Parr, Jaworski, & Suckling, 2014).

Each coating formulation will also need a plasticizer, as hemicellulose alone is quite brittle when used in any sort of film formation applications. Sorbitol, a polyol used as a sweetener in sugar-free gum, was chosen as it was found to be more effective than alternatives at increasing the flexibility of hemicellulose films (Hansen & Plackett, 2008).

As a basis of comparison, a coating using glucomannan from konjac root will be prepared and tested. Glucomannan is another type of hydrophilic hemicellulose and it will be substituted in place of the xylan in a coating with exactly the same plasticizer and crosslinker. This comparison is being made because currently glucomannan is widely available in pure powder form as it is used as a dietary fiber supplement, and it has been shown to have good film formation properties in this department in the past (Ma, Pekarovicova, & Fleming, 2017).

The final coatings will not be hydrophobic, and thus will be readily recyclable as most recycling operations use a pulper similar to a giant blender that feeds bales of old corrugated cardboard or mixed waste into a massive quantity of water as its first step. Many strength additives to cardboard (like traditional starch sizing) can make the substrate water resistant which in turn can make recycling much more energy intensive to reslurry the material or even worse, impossible. That is problematic considering the United States recycled 89% of its corrugated cardboard in 2013 (United States Environmental Protection Agency, 2016).

The hemicellulose strength and gas barrier effects will be tested with TAPPI standard methods and compared to uncoated identical material, including PPS porosity, Mullen burst, and tensile index.

If the results are strong enough, further research may investigate economic viability of a full-scale production.

Goal

The goal of this experiment is to find a way to isolate hemicellulose from cheap and widely-available farm byproduct (corn cobs), and to find a way to reproducibly and dramatically increase the gas barrier and strength properties of previously uncoated linerboard.

Experimental

Laboratory Equipment

- 4000mL Beaker
- Arrow 850 Laboratory Mixer
- Hot Plate with Stir Bar
- Draw down bench
- #8 Meyer Rod
- Heat gun
- Laboratory Oven

Reagents

- Citric Acid
- Sodium Hypophosphite
- Sorbitol
- Konjac Glucomannan
- Sodium Hydroxide pellets
- Deionized water
- Spartan™ frozen Corn on the Cob
- 300g/L Acetic Acid
- Denatured Ethanol
- Uncoated linerboard courtesy of Green Bay Packaging Co., and Westrock Co.

Methods

Pure Water-Soluble Xylan Isolation:

Dried corn cobs were blended in a grinder typically used for wood chipping to obtain a coarse powder (particle size average around $\frac{1}{4}$ "). The powder was then treated with caustic and heat, as treating with acid to remove the hemicelluloses may result in smaller molecular weight (Benko, et al., 2007). The corn cob powder was placed into 5% NaOH, 95% deionized water (10% w/w powder, dry), and kept at 70°C for 1 hour in a beaker on a hot plate under continuous agitation, with an immersed sonicator set to 30W (the maximum available on this device). The emulsion was then pH adjusted with 30% acetic acid to pH 5.5 to precipitate the insoluble Hemicellulose-A (Gáspár, Kálmán, & Réczey, 2007). The resulting liquid was centrifuged to separate the remaining solids (mainly cellulose and lipids, now with water insoluble xylan) from the liquid portion (concentrated hemicellulose) for ten minutes at 500*g. The centrifuge tubes were carefully decanted into a bucket and twice the volume of this liquid was added of pure ethanol. The bucket was sealed and allowed to settle over a weekend. The resulting precipitate was then gathered and washed with a Büchner funnel using copious amounts of additional ethyl alcohol and a vacuum flask beneath the filter-lined funnel. Once the powder was dry, it was removed from the filter paper and stored in a refrigerator until it was prepared as a coating. This method of separation has been shown to extract the xylan from various corn materials in previous research which is why it was selected (Benko, et al., 2007), (Gáspár, Kálmán, &

Réczey, 2007). For the purposes of this paper, this powder will be referred to as solid xylan or soluble xylan.

Insoluble and Soluble Xylan in Liquid Phase Extraction

To preserve the insoluble portion of the xylan to be used in the coatings along with the water soluble portion, the cook of corn cob powder occurred in the exact same conditions as before (10% w/w corn cob in 5% NaOH for 1 hour at 70°C with 30W of sonication and continuous agitation). However, after the cook was complete, instead of neutralizing with acetic acid, the entire mixture was placed into a 150μ nylon mesh while still at alkaline pH. The mesh was placed into a Büchner funnel and sealed from the top. Then, a 5kg brass weight was placed onto it carefully, and pressed down to liberate as much liquid as possible to the vacuum chamber below the funnel. The solids inside the mesh were neutralized with acetic acid and discarded, and the liquid that was separated into the vacuum flask was bottled and refrigerated. The purpose of this extraction was to investigate the efficacy of a more economically viable and safe method than the pure powder extraction, as ethanol is both dangerous and expensive. For the purposes of this paper, this extracted xylan will be referred to as liquid xylan, xylan emulsion, or combined xylan.

Laboratory Digester Xylan Extraction

In an effort to further increase the economic viability of the use of corn cob xylan, a trial was conducted using a standard laboratory digester with an integrated process control system to maintain constant temperature and a recirculation pump that delivers cooking liquor from the bottom of the cooking vessel (a cylinder approximately 4" in diameter and 2' tall) to the top in a laminar flow pattern. Without grinding or drying the cobs, they were placed into the digester

column atop a thin bed of woodchips. The chips were to act as a filter to prevent lipids from the corn cobs from entering the pump too rapidly and causing damage. The chips were made of poplar and no more than 50g were used. Comparatively, 1340g of corn cobs were loaded into the digester, at 31% oven-dried solids content. The digester was run at 90°C for two hours in 5% NaOH, at which point the NaOH was recharged (the exact same amount was added again), and then digestion continued for another two hours. The liquid phase was extracted from the apparatus and the solid phase examined, then discarded. The liquid phase will ideally be rich in xylan and can be made directly into a coating.

Coating Preparation

Each coating in this experiment was prepared similarly, with some key differences. For the solid xylan powder and the glucomannan coating, the target substance was very slowly added into DI water under continuous agitation with the Arrow 850 stand mixer, and allowed to mix for five minutes. Then, the sorbitol (as a plasticizer) was slowly added and then allowed to mix for another 5 minutes. Sorbitol as a plasticizer is being added because hemicellulose alone in coatings is known to be brittle (Aulin & Lindström, 2011). Currently sorbitol is the target plasticizer as it had a better effect on tensile elongation than other plasticizers when used with xylan (Hansen & Plackett, 2008). After the plasticizer and target substance are thoroughly mixed, the citric acid (crosslinker) and sodium hypophosphite (catalyst) were slowly mixed. The whole coating was allowed to stir for yet another five minutes. The coating was applied from a stir-bar mixer to the substrate using a laboratory pipette, approximately 4-5mL at a time. The pipette was used to flood the nip of the #8 Meyer rod and the paper, and a drawdown was made for 1-4 coats, drying with a heat gun between each. All samples were then cured in a 105°C

laboratory oven for two hours, then transferred to a room at TAPPI standard atmospheric conditions (23°C, 50% RH).

For the liquid xylan extract coating, the extracted cooking liquor was the base liquid instead of deionized water. The liquor was acidified using 300g/L acetic acid until it reached a pH of approximately 6.7, as citric acid acts as a stronger crosslinker in acidic pH (Widsten, Dooley, Parr, Jaworski, & Suckling, 2014). This precipitated out the insoluble xylan, but under continuous agitation the insoluble portion remained at a constant enough concentration in emulsion, and with small enough particle size, to make reproducible coatings. Then, the sorbitol, citric acid, and sodium hypophosphite were added in under agitation and allowed the same time to mix, at room temperature. Again, the coating was transferred, 4-5mL at a time using a laboratory pipette, from a beaker on a stir-bar mixer to the substrate to flood the nip of a #8 Meyer rod before the drawdown commenced. This was repeated for 1-5 coatweights, drying with a heat gun between each set. The samples were then cured at 105°C for 2 hours, and transferred to TAPPI standard atmospheric conditions.

A comparison coating with glucomannan instead of xylan was made at 1% glucomannan concentration and all other ingredients were added at the exact same concentration and allowed to mix for the same amount of time. Drawdowns were completed using the same method as before, with heat gun use between coats and the same curing conditions.

Formulations

The coating formulations are as follows:

4% Solid Water-Soluble Xylan Coating:

- 4% isolated bone-dry water-soluble xylan
- \bullet 0.25% citric acid
- 0.125% sodium hypophosphite
- 2% Sorbitol
- 93.625% water

4% Liquid Xylan Extract Coating:

- 4% water soluble and insoluble xylan (in emulsion)
- \bullet 0.25% citric acid
- 0.125% sodium hypophosphite
- 2% Sorbitol
- 93.625% cooking liquor from extraction

Glucomannan Comparison Coating:

- 1% Glucomannan
- \bullet 0.25% citric acid
- 0.125% sodium hypophosphite
- 2% Sorbitol
- 96.625% water

Results & Discussion

Each coating had a different appearance and physical properties of the others. To quantify this difference, the viscosities of each coating were tested using a Brookfield viscometer and are listed in Table 1.

Table 1. Brookfield viscosities of the three coatings [cP].

As evidenced by the extremely high viscosity, the glucomannan coating essentially gelatinized each time it was made. Furthermore, the solution contained globules of glucomannan that would not dissolve no matter how long, calmly or aggressively the mixing occurred. This in effect created "fish eye" defects in the coating. Even halving the glucomannan content still resulted in a gelatinized coating containing globules. Accordingly, after ten attempts to make a glucomannan coating, it was eliminated from further experimental work.

The coatings made from the solid isolated xylan were very runny at only 5cP, but still had good uptake into the sheet and did not wick into the sheet before the drawdown could be made. However, since the coating was so thin, the substrate experienced severe curl. Accordingly, the study was stopped at four drawdowns as a maximum because after that point the curled substrate was difficult to apply further coatings onto with an evenness needed for this experiment.

The coating that left the best initial impression was the liquid xylan extract coating, as it had just the right viscosity and uptake into the sheet. There was some minor curl but that can be expected when not coating a substrate under tension. Overall, this coating provided the best appearance and feel. However, after curing in the oven and placing into TAPPI standard conditions, this coating was hygroscopic enough feel damp to the touch after 24 hours in 50% relative humidity. The specific moisture data is listed in Table 2 for the liquid xylan coating and displayed graphically in Figure 2. Specific moisture data for the solid xylan coating can be found in the Appendix.

During the laboratory cook that used the digester, almost zero hemicellulose was present in the cooking liquor when tested with acid and ethanol. Furthermore, the cobs that were loaded into the digester chamber were essentially the exact same shape and size after digestion. Therefore, the liquor from this step was not used in coating formulations.

Liquid Xylan Coating Moisture Content at TAPPI Conditioning							
Liquid Xylan	Basis Weight Samples						
# of Coatings	Conditioned Coatweight (g/m^{2})	Coated, Conditioned Moisture Content (%)	Bone Dry Coatweight (g/m^{2})	Water Absorption (g/m^{2})			
	6.125	8.16	5.00	1.125			
ำ	7.575	8.61	5.60	1.975			
3	14.625	9.42	10.70	3.925			
	20.125	10.16	14.40	5.725			
5	27.025	10.58	19.85	7.175			
Bone Dry			Control Moisture Content:				
Control Basis Wt.		140.8 g/m^{2}	7.75%				
Conditioned Control		152.6 g/m^2		11.825 g/m^{2}			
Basis Wt.							

Table 2. Moisture content data for the liquid xylan extract coating

Figure 2. Moisture vs. Number of Drawdowns for both xylan coatings.

The solid xylan coating also exhibited significantly less bone cry coatweight than the liquid xylan coating at the same number of drawdowns. This is exhibited in Figure 3. All relevant data is included in the appendix.

average where N=2.

The main purpose of this experiment was to create a gas barrier coating using otherwise discarded hemicellulose. Accordingly, the most appropriate experiments to verify this gas barrier property are either Gurley or Parker Print Surf porosity, which are inversely related. For this study the Parker Print Surf was used. The results of the porosity study are displayed graphically in Figure 4. The raw data table can be found in the Appendix.

Figure 4. Porosity vs. number of coatings for both coatings. The error bars on the control line represent plus or minus one standard deviation of the control samples. Each point is an average

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where N=3.
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When dealing with linerboard, burst strength is also of particular interest to manufacturers and their customers. For this experiment, Mullen burst was used. The results of the testing for both xylan coatings at different coat weights are shown in Figure 5.

Figure 5. Mullen burst vs. number of coatings for both coatings. The error bars on the control line represent plus or minus one standard deviation of control samples. Each point is an average where N=3.

All data from Figure 5 is included in the Appendix.

Lastly, tensile strength can be a good indicator of overall linerboard strength, and the results of tensile testing all of the samples are shown in Figures 6 and 7. The relevant data is included in the Appendix.

Figure 6. MD tensile strength vs. number of coatings. The error bars on the control line represent plus or minus one standard deviation of control samples. Each point is an average where N=5.

Figure 7. CD tensile strength vs. number of coatings. The error bars on the control line represent plus or minus one standard deviation of control samples. Each point is an average where N=5.

Conclusion

Biodegradable coatings were formulated based on xylan, isolated from corn cobs by various methods. Xylan in the coating was crosslinked via citric acid which was catalyzed by sodium hypophosphite. The prepared coatings were applied onto unbleached board and various properties of these boards were compared to uncoated board. Due to time constraints, no analysis of citric acid alone on board properties was studied.

As is evident in Figure 4, the porosity of the samples coated with the liquid xylan combination based coating drastically decreased with each additional coat. However, the isolated solid xylan coating had almost no significant effect on porosity.

Conversely, the Mullen burst of the samples slightly improved when adding the solid xylan coating, with an apparent diminishing return after the second coating (Figure 5). However, this result could be based on the fact that more citric acid is going into the substrate, rather than the xylan, as citric acid is shown to improve strength properties even when used alone (Widsten, Dooley, Parr, Jaworski, & Suckling, 2014).

The liquid xylan coating seemed to have the best effect on Mullen burst at one coat, and diminished back to the control levels with increasing coatings (Figure 5). This is potentially due to the hygroscopic nature of the liquid-xylan coating, as additional moisture in the substrate is known to weaken burst strength.

Neither coating seemed to significantly affect tensile strength (or elongation, shown in the Appendix) either in the machine or cross direction (Figures 6 and 7).

Recommendations

To effectively create a suitable coating base liquor from a laboratory digester, one must grind the corn cobs to a coarse grain first to expose enough surface area to the cooking liquor to effectively dissolve the xylan. This was noted by the ineffectiveness of the digester to yield any xylan or effectively break down the intact corn cobs.

The liquid xylan extract was by far the best coating to reduce porosity, but it had a diminished effect on Mullen burst with increasing coatweight. This was likely do to its hygroscopic nature, as increased moisture in the substrate will reduce strength properties. Thus, if research on this substance is to go forward, this property should be targeted and reduced, perhaps by adding more crosslinker.

The solid xylan coating failed to produce a gas barrier, but did noticeably improve the burst strength of the substrate. However, it is possible that this is only due to the citric acid content, as discussed by Widsten, et al. (Widsten, Dooley, Parr, Jaworski, & Suckling, 2014).

Going forward, an investigation to see the effects of only citric acid and SHP on linerboard should be conducted, in an attempt to refute the result produced by the xylan coatings on Mullen burst strength.

Furthermore, to verify the results of this experiment, all steps should be replicated and the results compared to those found during this study before concluding that any effect seen here is not due to chance alone.

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Appendix

Table 3. The moisture content of the solid xylan coating compared to number of coats. It was excluded from the full report as it does not deviate from the control by enough to cause concern.

Solid Xylan Coating Moisture Content at TAPPI Conditioning							
Basis Weight Samples Solid Isolated Xylan							
# of Coatings	Coated, Coat Weight Conditioned (Conditioned) Moisture Content		Coat Weight (Bone Dry)	Water Absorption			
	1.575	7.7%	1.5	0.075			
	0.775	7.4%	1.2	-0.425			
3	3.075	8.0%	2.4	0.675			
4	4.975	8.2%	3.8	1.175			
Units	lg/m^2		g/m^{2}	g/m^{2}			

Table 4. Porosity data for both coatings where N=3 on all samples.

Table 5. Mullen Burst data for both coatings where N=3 on all samples.

Mullen Burst (psi)							
Coats	Solid Xylan			Liq. Xylan			
			Average Std Dev Std Dev % Average Std Dev Std Dev %				
	44.0	3.4	7.7%	44.0	3.4	7.7%	
	45.8	8.3	18.0%	55.0	4.6	8.3%	
$\overline{2}$	54.8	4.5	8.3%	47.8	5.6	11.7%	
3	57.5	2.6	4.6%	46.8	4.3	9.1%	
4	58.5	2.8	4.8%	43.3	2.5	5.8%	
5				44.0	5.6	13%	

Table 6. CD Tensile data for both coatings, where N=4 for all samples.

CD Tensile								
	Solid Xylan				Liq. Xylan			
Coats	Tensile Strength (kgf)	Elongation (mm)	Tensile Std Dev	Elongation Std Dev	Tensile Strength (kgf)	(mm)	Elongation Tensile Std Dev	Elongation Std Dev
	4.91	3.42	5.3%	20.0%	4.91	3.42	5.3%	20.0%
	6.05	4.67	4.6%	6.3%	5.05	3.89	7.1%	19.6%
	5.44	3.82	4.5%	14.3%	5.12	4.85	5.5%	14.2%
	5.49	4.24	9.7%	21.5%	4.30	3.89	6.2%	14.3%
	5.66	4.38	3.1%	10.4%	4.55	4.22	9.4%	22.9%
					4.69	3.60	9.6%	88.9%

Table 7. MD Tensile data for both coatings, where N=4 for all samples.

