

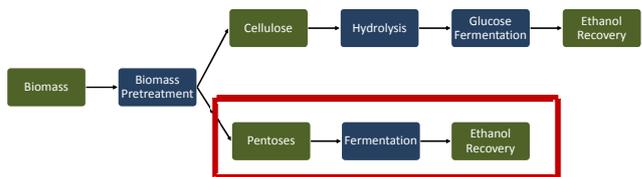
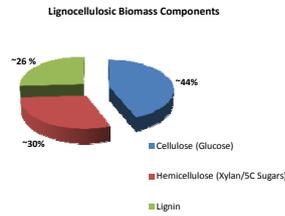
Determination of an Efficient Extraction Method of Xylan From Biomass

John Rowley, Stuart Black

Background

• Biofuels are becoming more widespread throughout the United States as more advanced conversion methods become available. The most advanced process at this point is the conversion of biomass into ethanol (5). However, research into biofuels other than ethanol is an important aspect of the DOE's mission to promote the spread of renewable energies. Before progress can be made in these areas it is necessary to understand in detail the structural components that make up biomass.

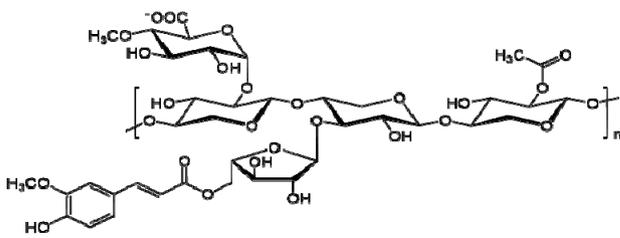
• This study focused on improving the efficiency of xylan isolation from biomass which can assist in further structural studies on this important source of 5C sugars.



— Starting Material or Product
— Process

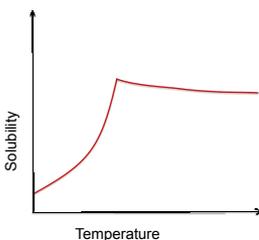
Biomass is converted to biofuels such as ethanol through a process outlined here. This experiment focuses specifically on the pretreatment phase of production and the structural components of the starting material.

Experiment



Many rearrangements of the xylan structure are found after isolation. One example of the xylan structure is found above (3).

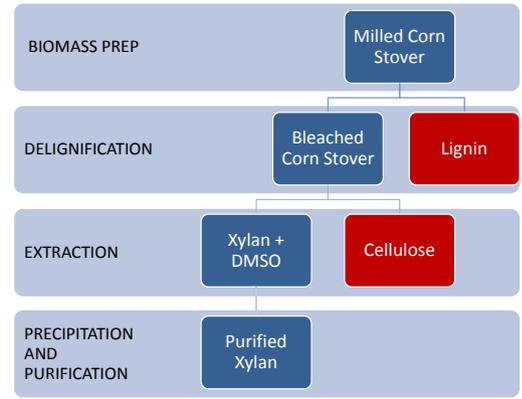
Many biomass pretreatment methods result in degradation of the xylan or de-acetylation of the xylan resulting in a water-insoluble product which is unusable for further analysis. Dimethyl sulfoxide (DMSO) extractions have been shown to avoid this structural change (3). In this study, a DMSO extraction of xylan in corn stover was studied at varying temperatures of extraction.



Why would heat improve the extraction of xylan in DMSO? The reasoning for this hypothesis is shown in this general solubility curve.

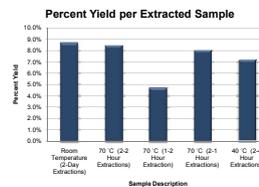
Materials and Methods

1. Delignification: Lignin was removed from the milled corn stover using acid chlorite.
2. Extraction: Delignified corn stover was extracted with DMSO. The variables analyzed were: Temperature, time of extraction and number of extractions (volume of DMSO used).
3. Precipitation and Purification: Ethanol (1 gallon/L solution) and a small amount of hydrochloric acid was added to the xylan/DMSO solution. This was allowed to cool to ~4 degrees C to precipitate the product. The xylan was purified with ethanol and ether and dissolved in water.

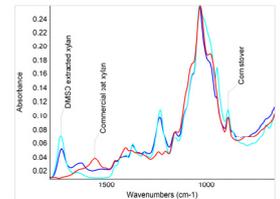


Results and Conclusion

$$\% \text{ Yield} = \frac{\text{Mass of Recovered Xylan}}{\text{Initial Mass of Corn Stover}}$$



Xylan yield from several 5 different extraction methods



IR Spectra of three extracted xylan samples. The commercial xylan is obtained through NaOH extraction.

It was concluded that heating to 70 degrees C while extracting can greatly increase the efficiency of the extraction. Between the 2-extraction heated samples and the non-heated sample, no significant loss in yield was discovered and the extraction time was greatly reduced. No significant structural differences were found upon heating.

Future Work: It should be determined if increasing the volume of DMSO used in one extraction can limit the number of extractions needed.

Sources

1. Ebringerova A, Hromadkova Z, Heinze T (2005) Hemicellulose. Adv Polym Sci 186:1-67. doi:10.1007/b136816
2. Hagglund E, Lindberg B, McPherson J (1956) Dimethylsulphoxide, a Solvent for Hemicelluloses. Acta Chem Scand 10:1160-1164. doi:10.3891/acta.chem.scand.10-1160
3. Naran R, Black S, Decker S, Azadi P (2009) Extraction and Characterization of Native Heteroxylans From Delignified Corn Stover. Cellulose 16:661-675
4. Kim T, Nghiem N, Hicks K (2009) Pretreatment and Fractionation of Corn Stover by Soaking In Ethanol and Aqueous Ammonia. Appl Biochem Biotechnol (2009)
5. Kazi FK et al. Techno-economic comparison of process technologies for biochemical ethanol production from corn stover. Fuel (2010), doi: 10.1016/j.fuel.2010.01.001