Department of Agriculture, Trade and Consumer Protection

Division of Agricultural Development

Agricultural Development & Diversification Program (ADD)

Grant Project Final Report

Contract Number: 16104

Grant Project Title: Mechanically Modified Cellulose ("MMC") Produced from Agricultural Crop and Ag Processing Residues

Amount of Funding Awarded: $22,500

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Development of Fiberstar Fibrous Materials in Filtration, Absorbent, and Hydrocolloid Applications

FINAL REPORT

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February 7, 2004

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EXECUTIVE SUMMARY

In the proposal entitled *Proposals for development of Fiberstar fibrous materials in Filtration, Absorbent, and Hydrocolloid applications*, dated March 18, 2002, three projects were outlined that focused on the development of corn stover into a viable fiber product. Project one was to determine the viability of using Fiberstar's corn stover based product in filtration applications, project two was to evaluate the use of Fiberstar's product in absorbent applications, and project three was to evaluate the use of Fiberstar's product in hydrocolloid applications.

To summarize, the data suggests that there is potential for Fiberstar's product to be used in the filtration, absorbent, and hydrocolloid applications. However, the data appears to be the most positive in the hydrocolloid and absorbent applications. In the hydrocolloid application, the fiber appeared to have powerful thickening properties, similar to other high priced products currently used in the market place, e.g. xanthan gum.

In the absorbent applications, the fiber appears to hold the water as well as other diaper products currently on the market place but the issue to resolve is how to get the fiber to strongly bind the water so that would not give it up. For example, in is current state, if the fiber was used alone in a diaper, the diaper would feel more wet to the touch compared to other products currently being used.

In the filtration applications, although Fiberstar is able to produce a product with a high surface area, which is a unique but necessary property, the issue is still finding a way to get the product to drain well and maintain the surface area through the drying process, which was addressed but not successfully resolved in this project.

PROJECT 1: FILTRATION

Introduction

When fibrous materials are processed through Fiberstar's process, the products have a high surface area, which is an attractive physical property to the filtration industry. Filtration media needs to have a high surface area so that filter can catch a contaminate as it passes through the filtration media. According to studies done at the University of Minnesota, ball milled corn cobs and husks had a specific surface area of 0.9 square meters per gram (m^2/g), but after going through a similar cell rupturing process used by Fiberstar, the fibrous materials had a specific surface area of 37 m^2/g (Yi, 1997). In more recent processes used by Fiberstar with corn stalks as the raw material, we have attained specific surface areas of 8 – 15 m^2/g.

The filtration industry currently uses diatomite as filtration media, which is inorganic and is becoming increasingly unpopular because it cannot be incinerated and has to be landfilled. Due to the potential large savings in disposal costs, the filtration industry is seeking an organic filtration media to replace diatomite that can be incinerated after the filter pad is used.

In the manufacture of filter pads, there are three main components added--wood pulp, diatomite, and a resin. The pad is formed in a similar way paper is formed, where a slurry of the components are made and the slurry is dumped on a wire mesh that dewaters and eventually goes through drying processes to form a filter pad.

Resins are used to bind the diatomite and wood pulp together as well as act as a cationic charge in the filter pad, which results in the filter having a high zeta potential. The filtration media needs to be cationic because most particles that need to be removed are anionic and the opposite charges attract each other.

Due to its high surface, raw materials such as processed corn stover should, in theory, should be able to serve as filtration media. Studies have been done at the University of Minnesota showing that expanded cellulosic materials are effective at removing rancid particles in oil (Ruan et al, 1996). However, these experiments were not conducted by putting the cellulosic materials in a filter pad, which is how they would be commercially sold. Recently, Fiberstar has also done some preliminary work using our expanded products as filtration media to try and replace diatomite in filter pads. However, we have run into issues that need to be resolved because our products attract water so much that they swell and do not allow water to flow through pad. A high liquid flow through rate is an important characteristic of a good filter pad. To resolve low flow rate issues, our objective has been to try and make our fibers hydrophobic, which we should be able to do by co-homogenizing our fiber with a hydrophobic compound. We believe a hydrophobic coated fiber will maintain its desirable high surface area yet also allow water to pass through so that it does not slow down the filtration process. It would be advantageous if this additive could also be cationic so that in the manufacture of the filter pad, the expense of adding a cation resin would not be needed.

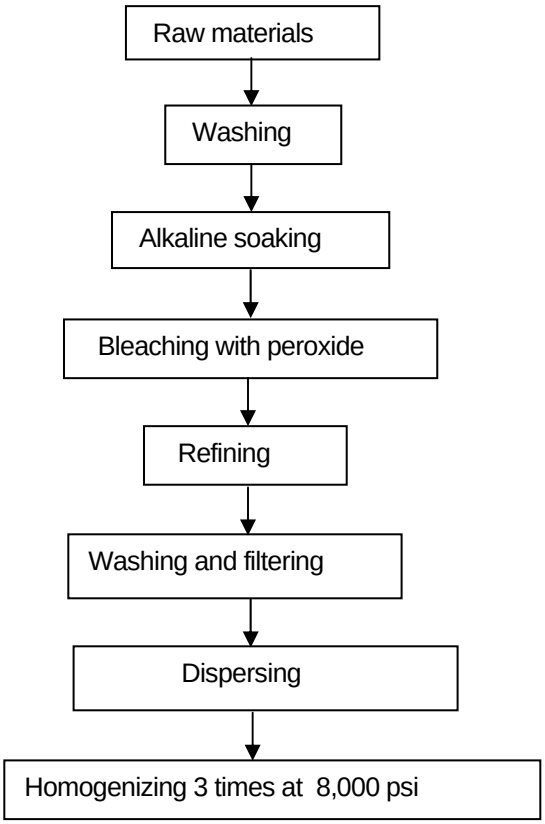
Another problem that needs to be resolved prior to us using our materials in the filtration industry is that we need to figure out a way to dry our products and maintain a high surface area. This problem may partially resolve itself by the addition of the hydrophobic additives mentioned above but even so,

a method for drying needs to be developed to maintain the high surface area of our materials. A process that goes along with nearly any drying operation is particle sizing, which also needs to be identified.

The objective of this project was to develop a cellulosic product that could be used for filtration media to replace diatomite and has a high flow through rate and ability to remove contaminants in water and/or oil.

Materials and Methods

The following general process was used to make the corn stover materials.



Materials were processed using two different methods to compare the results. In one method the fiber was processed with a surfactant and in another method the fiber was not processed with the surfactant. Water holding capacities and viscosities were measured to evaluate the functionality of the final products. A drainage water holding capacity was measured to evaluate the speed at which the fiber let go of water, which is a way to measure how well the fiber will work when being made into a filter pad.

Results and Discussion

The results of processing with the surfactant are as follows:

Description	dWHC*	AWRC**	Visc 10 r***	Visc 50 r (cP)	Separation****
Materials w/ surf before refiner and another add'n before homog	37	29.1	1260	428	N
Materials w/out a surfactant	39	30.6	1240	372	N

*This is a drainage water holding capacity, which is found by placing the hydrated product on a filter pad in a buchner funnel for 30 minutes and measuring the amount of water the fiber holds.
** Alkaline water retention capacity (AWRC) was the primary parameter measured to test the water holding capacity of the treated samples. The AWRC was measured using the test method from American Association of Cereal Chemists (AACC) Standard Method Number 246.
*** The viscosity of various samples was tested as another parameter to be used in this project. All viscosity tests were conducted using a Brookfield rotational viscometer and the results are recorded in centipose (cP). 10r means the viscosity at 10 rpm while 50 r means the viscosity at 50 rpm.

These results shows that using the surfactant really does not make that much of a difference in speeding up the draining ability of the fiber, which is shown by the drainage water holding capacity (dWHC) results. However, the using the surfactant still allowed the fiber to maintain its high water holding capacity, which is shown by the alkaline water retention capacity (AWRC), viscosity, and the fact that it did not separate in the solution.

Conclusion

At this point it does not appear the producing Fiberstar's product with a surfactant is an effective means increasing the drainage speed of the final product. Because of these results, it does not appear that this is the answer to solving the needs for what is needed in the filtration applications. The nest steps would be to look at using other types of products or processes to make Fiberstar's product hydrophobic to increase the drainage water holding capacity.

PROJECT 2: ABSORBENTS

Introduction

The use of Fiberstar materials as a highly absorbent material could potentially be a high volume use of corn stover. After going through Fiberstar's novel process, the water binding affinity or water holding capacity of corn stover is substantially increased. By using alkaline water retention capacity (AWRC) as a measure of water holding capacity, unprocessed corn stover had a AWRC of roughly 5-10 g water/g solids whereas after processing, corn stover has a AWRC of over 40 g water/g solids (Lundberg, MS Theses, 2000). Because of processed corn stover's high water absorbency and being an all-natural product, it could potentially be a desirable product in the absorbent industry.

Examples of products that use highly absorbent materials are diapers and other personal care products. Currently, two different materials are used in absorbent products—fluff pulp and superabsorbent polymers. Superabsorbent polymers typically used are chemically derived, quite expensive, and do not wick liquids well but have excellent water binding and holding characteristics. Fluff pulp on the other hand, does not have excellent water binding characteristics but does have wicking capabilities and provides bulk in the absorbent product.

Processed Fiberstar materials have desirable characteristics as an absorbent material because they have a high affinity for binding water, they are all-natural (not chemically derived or modified), and could be cost competitive. However, prior to being considered by a company that makes an absorbent product, appropriate test data must be developed to show how our products compare to current superabsorbent polymers. Additionally, because our products are much different than the fluff pulp and superabsorbent polymers currently on the marketplace, a method needs to be developed to show how the products could be incorporated into absorbent products.

The objective of this project is to develop the data needed to show how our products compare in functionality and cost to current superabsorbent polymers and/or fluff pulp on the marketplace and develop a method showing our processed materials could be incorporated into an absorbent product.

Results and Discussion

We compared Fiberstar's materials to currently used diaper absorbent materials. We found that the of the current diaper's bulk density was in the range of 0.073 g/cc, or around 4.5 lbs/ cu ft. For a test, the same volume of diaper materials and Fiberstar's materials were placed in a container and water was allowed to drip on the materials and after about 5 minutes all free water was drained off. In the volume, around 0.0785 g of the diaper fluff was used and around 0.22 g of Fiberstar's materials. The fluff diaper materials absorbed 3.04 g or water, or 38 g water/g solids and Fiberstar's materials absorbed 2.83 g or 12.8 g water/g solids. From a visual point of view, it did appear that Fiberstar's materials absorbed water a bit faster and had better wicking than the diaper materials. On a volumetric basis, Fiberstar's materials seem to have similar absorption abilities as the diaper materials. Both Fiberstar's materials and the diaper materials swelled. Fiberstar's materials also soaked up water and fell to the bottom of the container in solution. The alkaline water retention capacity (AWRC) of Fiberstar's materials at 1% solids was 21 and from using the drainage test, the

water holding capacity was 25. Also from a visual point of view, the absorbent materials in the diapers took up more water and held on to it real tightly from the superabsorbent swelling and permanently binding the water. Fiberstar's material will let go of the water more easily if they are squeezed where as the superabsorbent materials will not.

Conclusions

It does appear that there could be a potential application of Fiberstar's materials in an absorbent application. However, the are several issues that would need to be resolved, namely, economics, getting the water to bind the water tighter and not be as wet to the touch, and performance once put into an actual application. Some additional things to look for future studies include the differences with salt water or with artificial urine or trying making Fiberstar's materials into a mat to see how this affects the absorbency.

PROJECT 3: RHEOLOGY

Introduction

Corn stover, when processed through the Fiberstar process, forms a highly viscous solution, which makes it an attractive hydrocolloid for thickening and suspending applications.

A desirable hydrocolloid for a salad dressing company would be one that:

- Has thickening and suspending rheology so that it can suspends particles and create a desirable mouthfeel
- Is free flowing so that it passes through its bins and hoppers without bridging and causing difficulties in handling equipment
- Has particles small enough so that they will not be felt in the mouth
- Readily rehydrate without requiring a high amount of shear to go back into solution

There are also some problems with corn stover that need to be addressed prior to it being used in the marketplace. First, the current corn stover products we have been producing are extremely fibrous in nature. To be used as a product by a food company, our materials would need to be free flowing so that they readily could flow through bins and hoppers, which our current materials would not because of their fibrous nature. Additionally, the fibrous nature of the corn stover particle gives a mouthfeel problem because a good hydrocolloid should not be felt in the mouth. Because of the desired thickening and suspension properties, we believe our product would be advantageous over xanthan as a hydrocolloid in salad dressings and sauces applications if we can resolve the fibrous and mouthfeel problems, which we believe we can resolve with right particle sizing processes.

An additional problem with corn stover relates to how it is dried and rehydrated. We currently have not identified a practical method for drying corn stover. Although several technologies exist for drying, we have not spent the time and money needed to resolve the drying process because of the need to resolve the particle size and shape first. An issue with the post-dried corn stover that needs to be addressed is making a dry product that readily rehydrates. To be customer friendly, a hydrocolloid should go back into solution by using moderate levels of mixing and not needing a high shear mixer for them to develop their full thickening, suspending, and water holding potential.

The first objective of this study was to compare the rheology of corn stover to other currently available products on the marketplace as well as other Fiberstar products, namely, processed beet pulp. The second objective of this project is to make corn stover attractive as a hydrocolloid in salad dressing and sauce applications by making it a free flowing powder and eliminating the mouthfeel problems while maintaining its desirable rheology.

Results and Discussion

We found that corn stover has similar rheology curves as xanthan, which is commonly used in salad dressings and many other food products. Because of xanthan's high costs (commonly $5-6 / lb) there is plenty of room for a Fiberstar product to be cost competitive. Corn stover also has a lot thickening power than a lot of other rheology modifying products, which is compared in the results below and is compared to processed beet pulp, which is marked by the letter Bp. Corn stover is shown in the following graphs and is marked by the letters Sb.

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Figure 1: Viscosity vs Shear rates for various products at 1% concentration

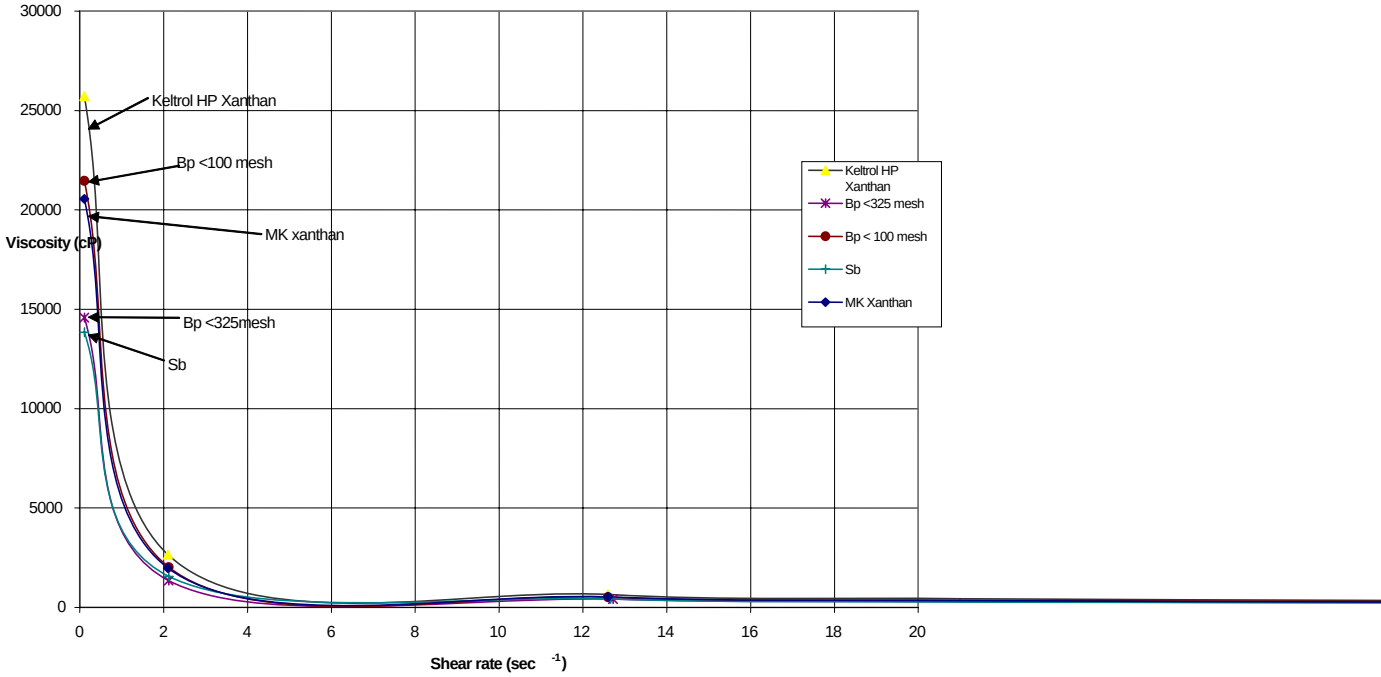

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Figure 2: Viscosity vs Shear rates for various products at 0.5% concentration

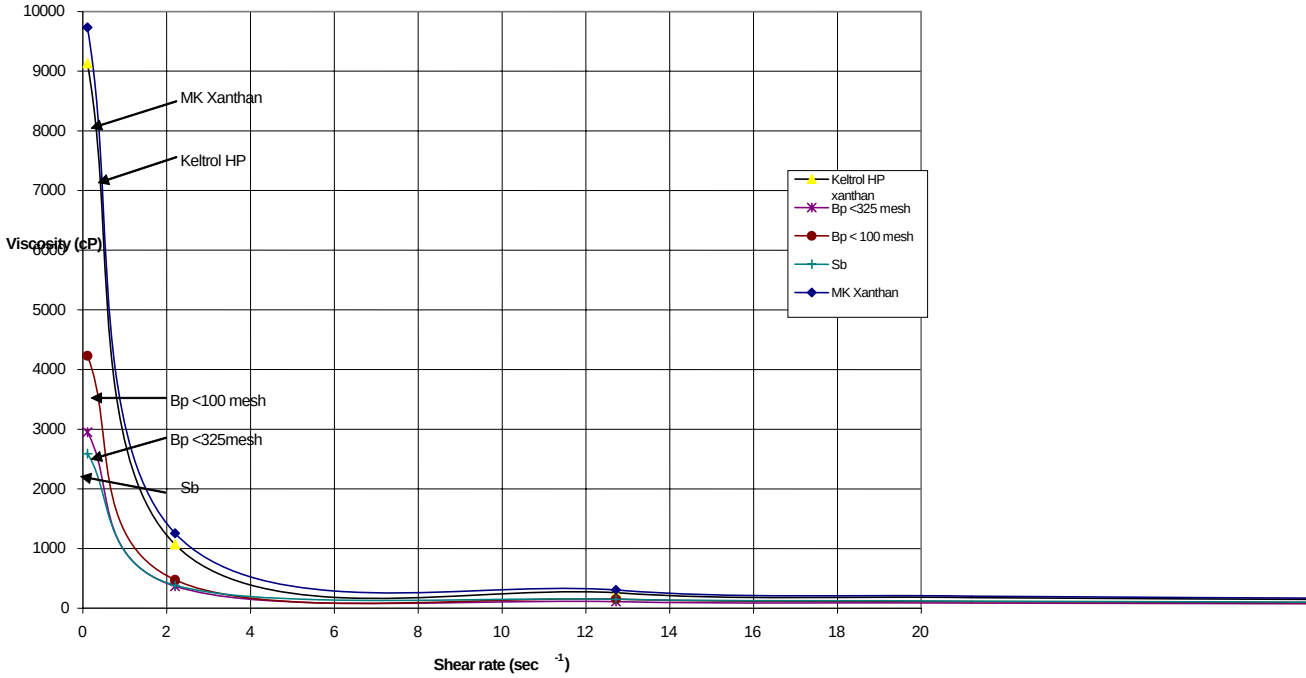

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Figure 3: Viscosity at 0.5% concentrations and at 0.1 sec $^{-1}$ shear rate[1,2,3]

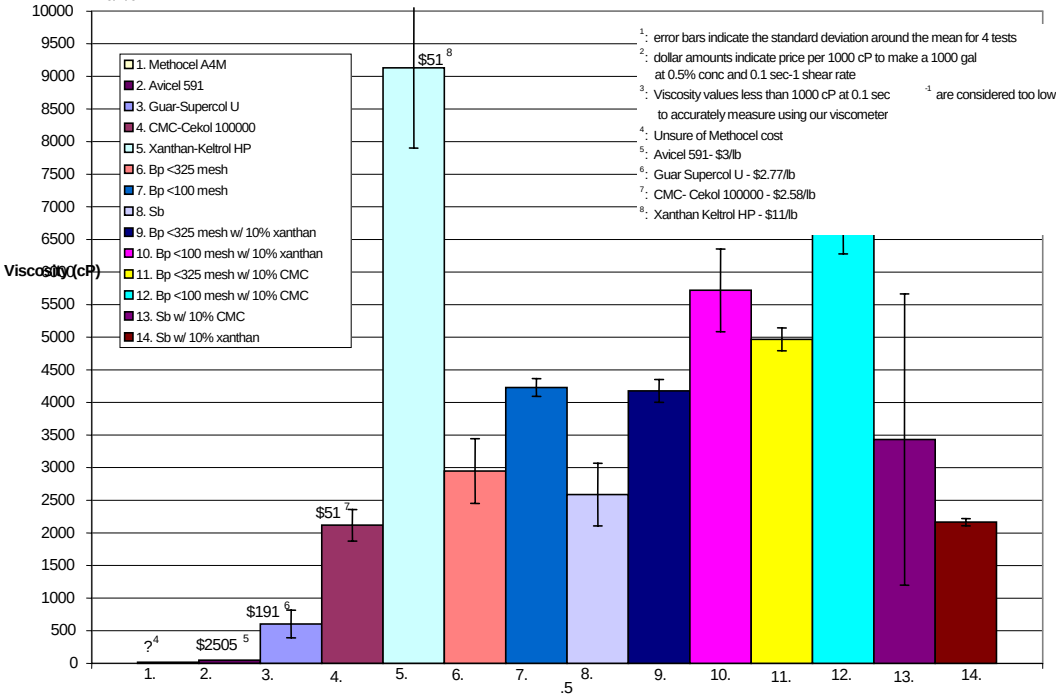

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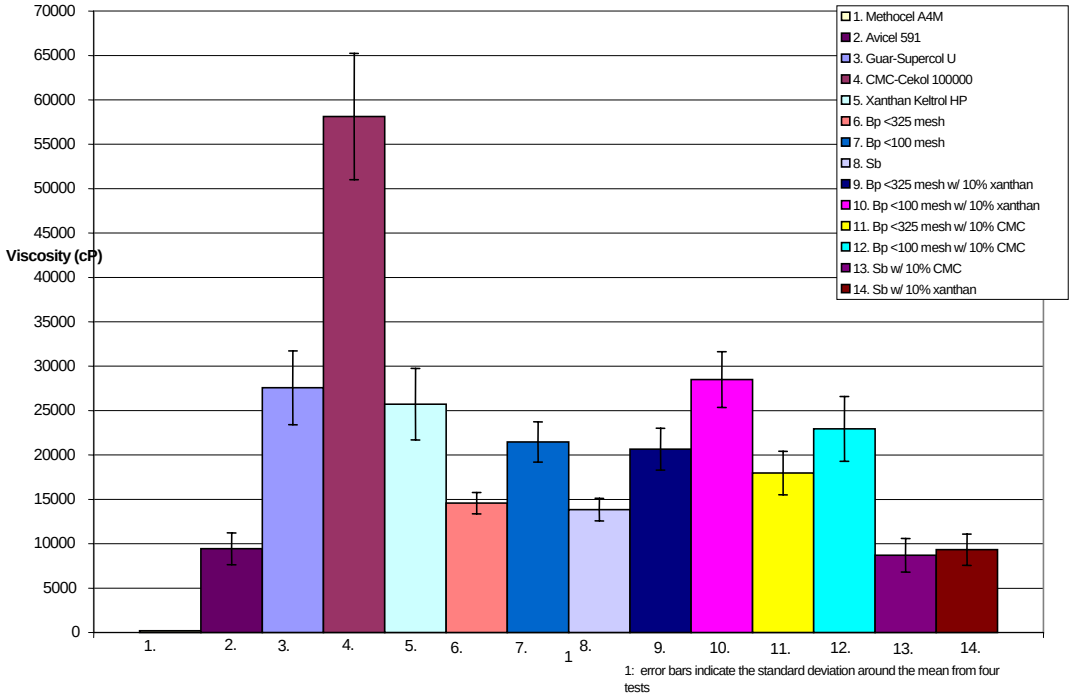
Figure 4: Viscosity at 1% concentrations and at 0.1 sec $^{-1}$ shear rate[1]



1: error bars indicate the standard deviation around the mean from four tests

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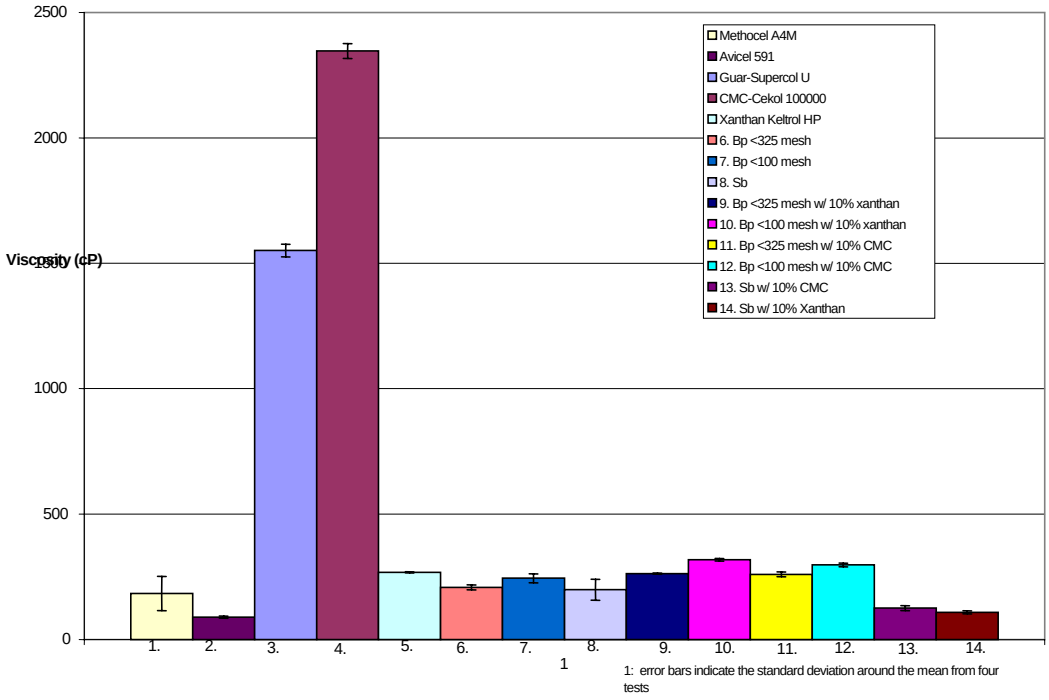
Figure 5: Viscosity at 1% concentrations and at 42 sec $^{-1}$ shear rate[1]



1: error bars indicate the standard deviation around the mean from four tests

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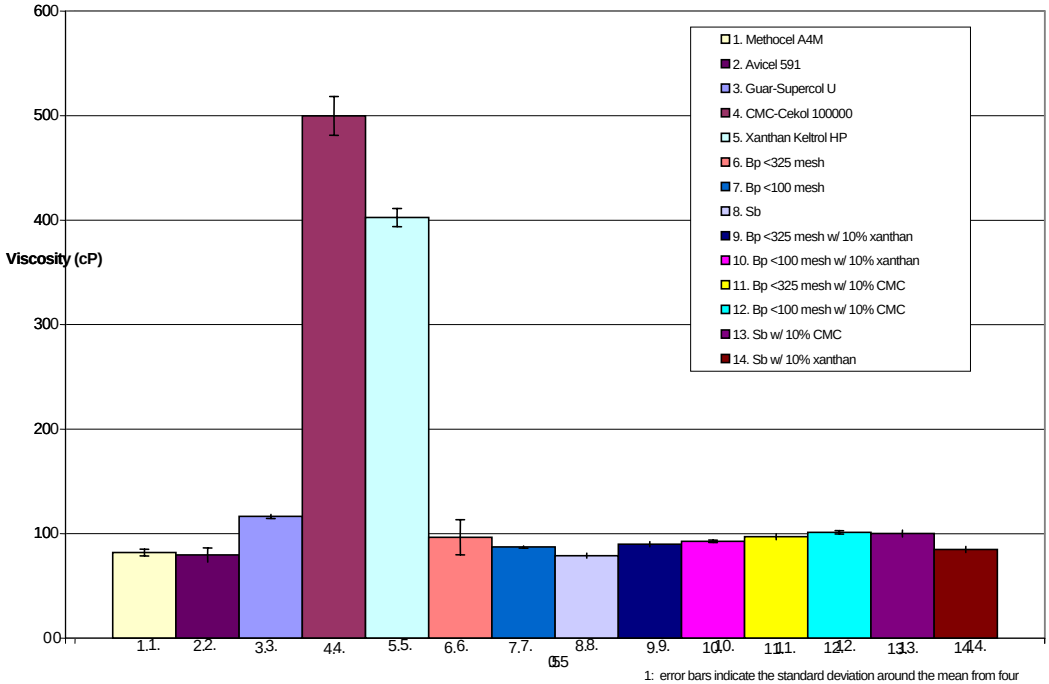
Figure 6: Viscosity at 0.5% concentrations and at 42 sec^{-1} shear rate[1]



1: error bars indicate the standard deviation around the mean from four tests

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Figure 7: Viscosity at 1% concentrations and at 12 sec^{-1} shear rate

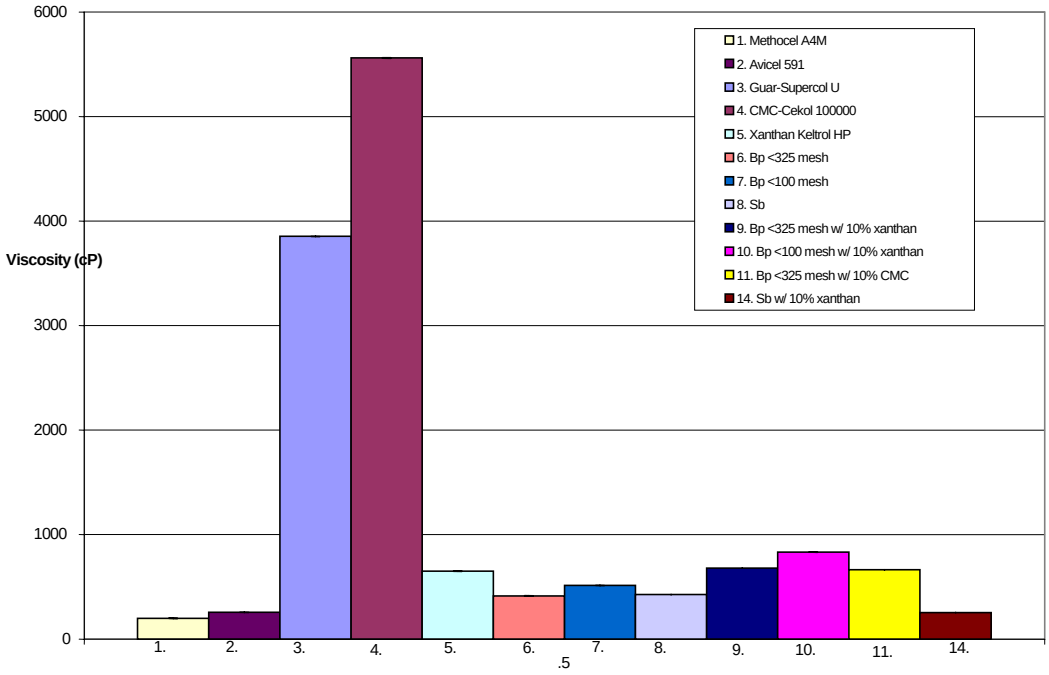

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Figure 8: Viscosity at 0.5% concentrations and at 12 sec $^{-1}$ shear rate

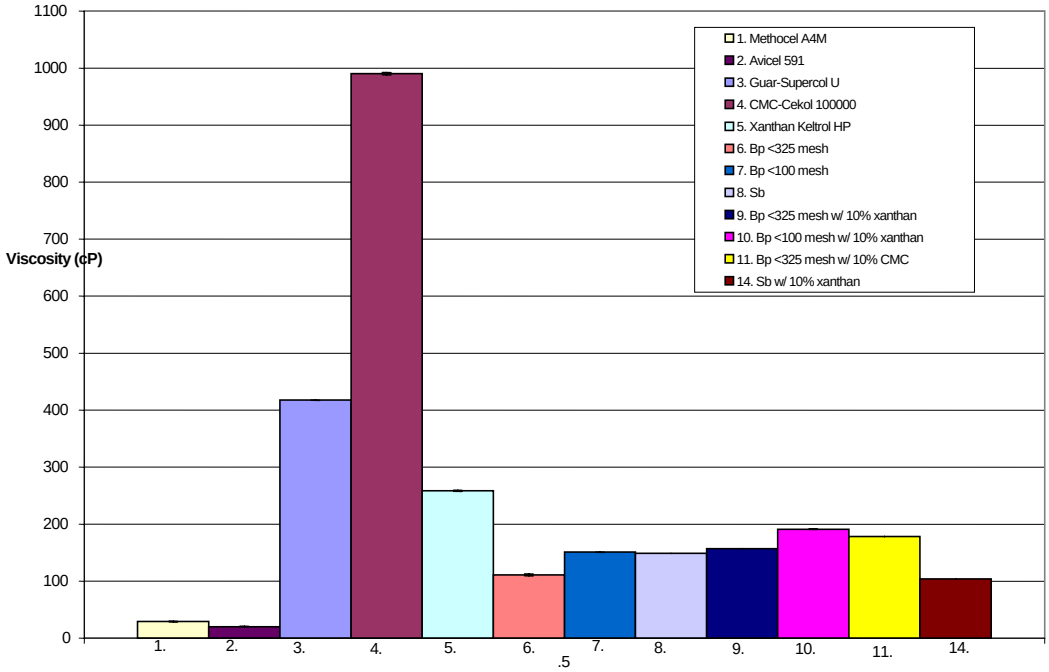

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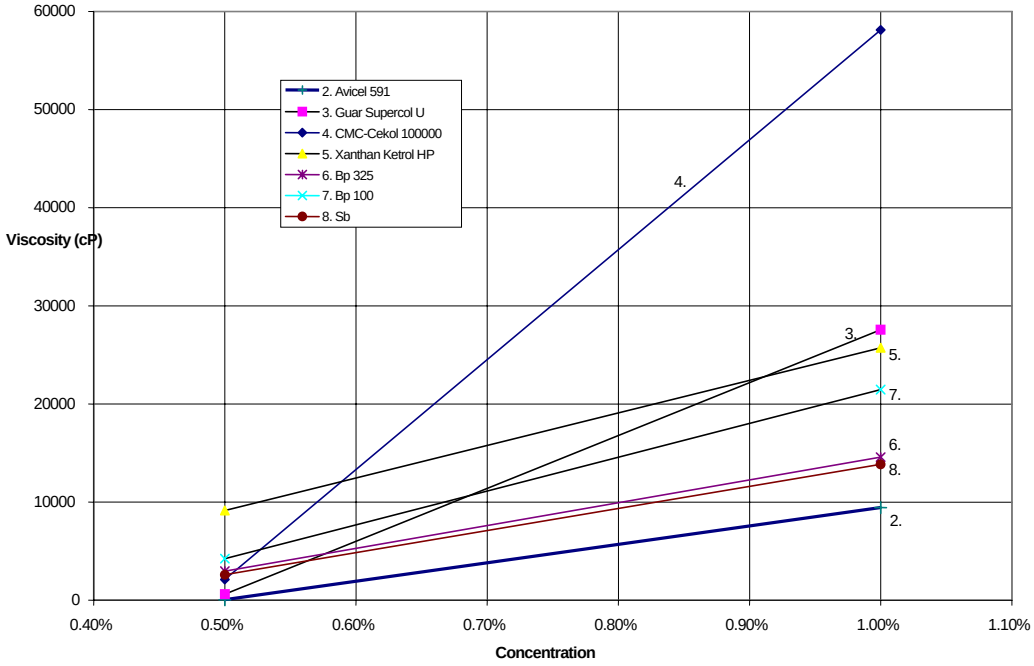
Figure 9: Visc vs concentration at 0.1 sec $^{-1}$ shear rate



Particle sizing results

With the equipment that we recently installed in Ellsworth, we have made significant improvements in making our materials into a free flowing powder. The improvements in particle sizing are mainly due to the advances that we have made in drying and milling our materials. Because we are able to mill the corn stover to relatively small particle sizes, this not only means the particles are free flowing, but they also have significantly less mouthfeel compared to what we have been able to do in the past.

Also, the more times we sent the products through the mill, the smaller they became. However, the functionality was also influenced by the milling, as shown in the data below.

Table showing functionality results of materials after being passed through a mill zero to three times.

Description	Solids %	Drain WHC*	AWRC**	Visc10 rpms***	Visc50 rpms	Separation****
No times through	--	35	30	1200	710	N
1st time through	89	34.4	29.8	1180	690	N
2 times through	93.7	28.2	25.0	790	480	N
3 times through	93.5	32.3	27.9	720	435	N

*This is a drainage water holding capacity, which is found by placing the hydrated product on a filter pad in a buchner funnel for 30 minutes and measuring the amount of water the fiber holds.
** Alkaline water retention capacity (AWRC) was the primary parameter measured to test the water holding capacity of the treated samples. The AWRC was measured using the test method from American Association of Cereal Chemists (AACC) Standard Method Number 246.
*** The viscosity of various samples was tested as another parameter to be used in this project. All viscosity tests were conducted using a Brookfield rotational viscometer and the results are recorded in centipose (cP). 10r means the viscosity at 10 rpm while 50 r means the viscosity at 50 rpm.

Conclusions

The data does suggest that Fiberstar's processed corn stover product does have potential as a thickener. The rheology curves compare well to other products currently on the marketplace. However, much needed data would be required to study how the materials function in an actual application, such as salad dressing, sauces, etc, followed by sensory evaluations and customer interaction, which should be considered the next steps for this project.