

# Hot-Washing of Pretreated Corn Stover Using Integrated Sands Horizontal Screw and Jaygo Pretreatment Reactors with Pneumapress Automatic Pressure Filter

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## 1. Abstract

The "hot wash" concept developed at NREL (U.S. Pat. No. 6,228,177) uses hot water or hot dilute acid at temperatures above the lignin liquid/glass condensation temperature (~135°C) to wash out the solubilized lignin and hemicellulosic sugars. Cooling pretreated biomass below this condensation temperature allows solubilized lignin to condense and precipitate out on the cellulosic residue interfering with enzymatic hydrolysis of cellulose to glucose. Bench-scale observations have established that hot washing of pretreated biomass leads to a highly digestible lignocellulosic pulp with low enzyme loadings of cellulase enzyme.

An integrated pilot-scale system consisting of a 200 kg (dry) per day Sands (now Metso) horizontal screw pretreatment reactor, with a 130-L Jaygo high solids paddle type reactor and the Pneumapress® Model 3-C276 automatic pressure filter was used to "hot wash" pretreated yellow poplar sawdust (YP) with hot water and other catalysts at greater than 140°C. Pretreatment was carried out at 175°C, 0.7 wt-% H<sub>2</sub>SO<sub>4</sub> and 6 min residence times

A one ton (dry basis) per day Sands vertical reactor was used to pretreat corn stover at 165°C to 190°C, for 5-8 minutes at 1% acid. The pretreated biomass slurry was allowed to cool before "hot-washing". Enzymatic digestibility's of hot-washed filter cakes of YP and corn stover showed enhanced digestibility between 11% and 20% above controls that were not "hot washed".

## 2. Introduction

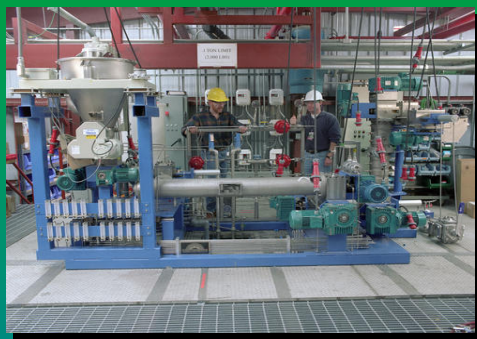
- "Hot wash" concept developed at NREL (U.S. Pat. No. 6,228,177)
- "Hot-wash" uses hot water or hot dilute acid at temperatures above lignin liquid/glass condensation temperature (~135°C) to wash out the solubilized lignin and hemicellulosic sugars.
- Cooling pretreated biomass below this condensation temperature allows solubilized lignin to condense and precipitate out on the cellulosic residue interfering with enzymatic hydrolysis of cellulose to glucose.
- Bench-scale observations have established that hot washing of pretreated biomass leads to a highly digestible lignocellulosic pulp using low enzyme loadings of cellulase enzyme.

## 3. Materials and Methods

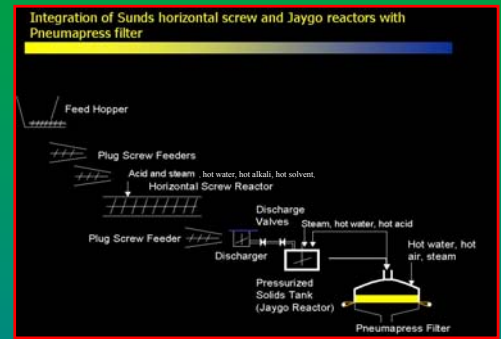
- Corn Stover was pretreated in a continuous pilot-scale specific Sands Hydrolyzer at 160°C to 190°C, 1% H<sub>2</sub>SO<sub>4</sub>, 5 to 8 min. Off-specification pretreated corn stover was allowed to cool after pretreatment.
- Yellow Poplar was pretreated in a pilot-scale continuous Sands Horizontal Screw reactor at 175°C, 0.7% H<sub>2</sub>SO<sub>4</sub>, for 6 minutes. Pretreated solids were not allowed to cool before "hot washing".
- Pretreated corn stover and yellow poplar were "hot washed" with hot water (~140°C), hot dilute alkali (0.1% and 0.2% NaOH), and hot dilute ethanol (0.2% and 0.5% EtOH).
- Digestibility of "hot washed" solids was performed in ~10% slurry using SSF assays at 37°C, with cellulose loading of 6g, with cellulase enzyme loadings of ~3-5 FPU/g cellulose, and the yeast *S. cerevisiae* D.A.



Sands vertical reactor for pretreatment of corn stover.



Sands Horizontal Screw reactor for pretreatment of yellow poplar sawdust.



Integrated "hot wash" system.



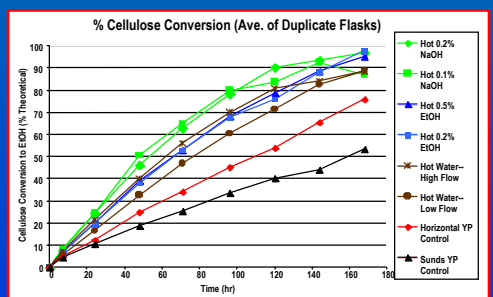
Jaygo high solids, paddle type batch reactor.



Pneumapress automatic pressure belt filter for "hot washing" pretreated corn stover and yellow poplar sawdust.

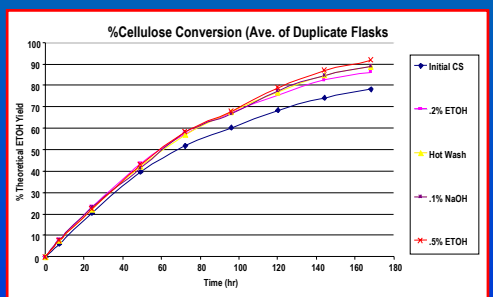
## 4. Results and Discussion

- Pneumapress filter system effectively washed and separated both the pretreated corn stover and yellow poplar.
- Hot washing with 0.5% EtOH and 0.1% NaOH was more effective in increasing substrate digestibility than hot washing alone.
- "Hot washing" pretreated yellow poplar sawdust increased the digestibility of residual cellulose from ~75% to ~97%.
- "Hot washing" pretreated off-specification corn stover increased digestibility of residual cellulose from ~78% to ~90%, even though pretreated slurry was allowed to cool.
- "Hot washing" allows high enzymatic digestibility (~90%) even though pretreatment was not optimal, suggesting pretreatment severity may be lowered.
- The "Hot washing" effect was greater in the pretreated yellow poplar than the pretreated corn stover.



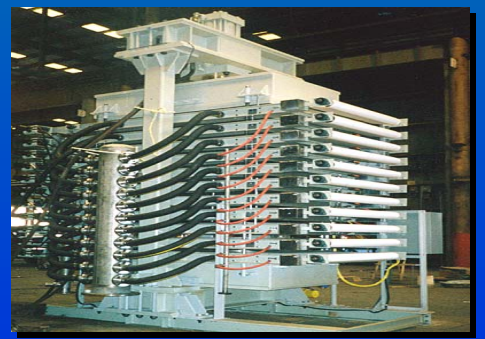
Pilot-Scale "Hot Wash" of Yellow Poplar

- SSF of Pretreated Yellow Poplar (175°C, 0.7% H<sub>2</sub>SO<sub>4</sub>, ~8 min)
- Followed immediately by hot separation/wash at ~140°C
- Cellulase loading: 20 mg protein/g cellulose (Genencor Spezyme) (~5 FPU/g)



Pilot-Scale "Hot Wash" of Corn Stover

- SSF of Off-specification Pretreated Corn Stover (165°C-190°C, 1% H<sub>2</sub>SO<sub>4</sub>, ~5-8 min)
- Hot washed at ~140°C
- Cellulase loading: 12 mg protein/g cellulose (Genencor Spezyme) (~3 FPU/g)



Possible commercial-scale filter for scaling up "hot-wash" process.

## 5. Conclusions

- Pilot-scale integrated "hot washing" system was installed at NREL
- Selective washing processes can produce lignin-based co-products.
- Optimized conditions for Pneumapress solid/liquid separation could reduce time required for substrate bioconversion.
- Flexibility of the Pneumapress automatic pressure belt filter could be used for other pilot-scale solid/liquid separations.

## 15. Acknowledgements

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