# Mechanical Pretreatment to Produce Cellulosic Sugars on a Pilot Scale

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# Mechanical Pretreatment for Alternative Jet Fuels (AJF)

### Problem

The conversion of alcohols to alternative jet fuels is currently under consideration by ASTM D7566 and expected to be balloted this spring. This method requires biomass to be reduced to sugars, which are subsequently fermented to alcohols and catalytically converted to iso-paraffinic kerosene as the alternative jet fuel. Sustainable, non-food grade sugars are needed to enable this process. A simplified process of producing these sugars from biomass will enable development of a distributive biofuel production system. Unfortunately, cellulosic sugar costs can represent as much as 65 percent of the total product costs of AJF. Moreover, the degradation byproducts of producing cellulosic sugars with thermochemical pretreatments can be the limiting factor.

#### Solution

This project seeks to develop a cost competitive mechanical pretreatment for disrupting the recalcitrance of wood biomass for producing clean, fermentable, cellulosic sugars. The barrier to commercializing the mechanical pretreatment is the perceived high energy requirements. Based on significant research, we have identified a three-stage milling process consisting of coarse, fine, and amorphization stages as a potential cost-effective route on an industrial scale. The **innovation** of the three-stage milling pretreatment is in separating coarse and fine size reduction steps from the energy intensive amorphization (cellulose crystalline structure disruption) process. This process utilizes different mill architectures to apply different modes of mechanical force corresponding to the primary comminution mechanisms at different size ranges.

### Goal

The overall goal is to produce highly digestible substrate at a modeled cost of 1.5 kwh/kg to enable the overall manufacturing of cellulosic sugars for biological processing at a full cost less than \$0.50 per kilogram of substrate. Specifically, this one -year project is to achieve 2.5 kwh/kg utilizing the existing milling technology on a pilot scale.

### Vision

Figure 1 is a process flow diagram as we have conceived to produce AJF from cellulose sugars through the mechanical pretreatment. It includes four process modules: input (feedstock preprocessing), mechanical pretreatment, hydrolysis (cellulosic sugars) and fermentation (alcohols), and products (refineries). It is envisioned that the preprocessing, mechanical pretreatment and hydrolysis can be implemented in a distributed depot system to produce cellulosic sugars. The depots can be deployed in the rural areas near the biomass resources, or co-located with existing sawmills or pulp mills. Shipping sugars to the central located alcohol or refinery plants will greatly reduce the transportation cost. Cellulosic sugar production through the dry milling pretreatment is ideally positioned to meet technological need for a small scale depot style production.

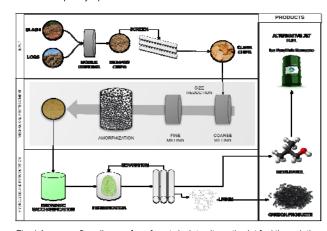
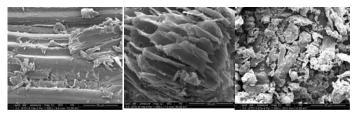


Fig. 1 A process flow diagram from forest slash to alternative jet fuel through the mechanical pretreatment

# **Energy Consumption and Particle Morphology**



Fig. 2 Total actual milling energy of 2.27 kwh/kg is based on a pilot scale system. The result is strong and sets us up for success at scale up. The larger machine is expected to be more energy efficient. So does that justify the 2015 funding because we are well in reach to proceed to a larger scale processing to achieve overall goal of 1.5 kwh/kg.



Wiley Knife Milled, Longitudinal Wiley Knife Milled, Cross Ball Milled 120-min

Fig. 3 Morphology changes from the coarse (Left and Middle: Wiley milled -40 mesh) to ball milling (Right: ball milled for 120-min under room temperature) stages. Coarse milling reduces the particle size but maintains the integrity of the wood structure and ultrastructure. Ball milling for 120-min under ambient temperature leads to the almost complete disappearance of discernible tissue structures (Right image).

100

80

20

e c (%)

# **Crystallinity and Sugar Yields**

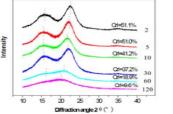


Fig. 4 Cellulose crystallinity index decreased (amorphization) with ball milling time under room temperature milling.

Fig. 5 Glucan yields of 72-hour enzymatic saccharification increased with the milling time under different milling conditions (Wiley knife milled, ball milled without controlling chamber temperature, and cryogenic milling)

Milling Time (min)

5 10 30 45 60 120 240

Room Temperature

Cryogenic

Wiley Milled 40 mesh

# Decreased Fermentation Inhibitors in the Sugar Stream



 Mechanical
 Hot Water
 Dilute Acid
 Dilute Alkalin

 Pretreated
 Pretreated
 Pretreated
 Pretreated

Fig. 6 Color difference of clarified enzymatic hydrolyzates. The light color of the hydrolyzate from the mechanical pretreatment indicated its less inhibitors, confirmed by smaller intensity at 280 nm (furfurals) and by the HPLC analysis.

# Improvement of Fermentation Efficiency

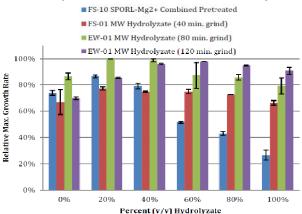


Fig. 7 Maximum growth rate of isobutanol producing yeast strain in FS-10 SPORL-Mg<sup>2+</sup> pretreated hydrolyzate, FS-01 MW (40 minute grind), EW-01 MW (80 minute grind) and EW-01 MW (120 minute grind) supplemented with a nutrition package. Mock media made for each hydrolyzate is a combination of hexose sugars, pentose sugars, and acetate at the same concentrations as the hydrolyzates supplemented with nutrition. Dilutions (volume/volume) of hydrolyzate were created using mock media. Growth carried out at 33°C and maximum growth rate measured for each percent (v/v) hydrolysate.

# **Clean Reactive Lignin**

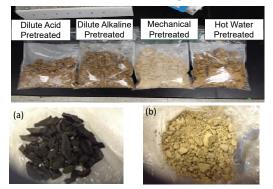


Fig. 8 Wet lignin residues after vacuum filtration. The color of the lignin residue from the mechanical treatment is lighter. After dried at 103 °C overnight, it hardened into chunks indicating its high reactive (a) while other thermochemical treated residues looked like loose sandy materials (b).

### Forward Looking

An industrial scale dry milling pretreatment will: (1) result in a clean sugar hydrolyzate that is less toxic and inhibitory to microorganisms for fermentation and more suitable to catalytic conversion; (2) produce a clean reactive lignin residue amenable to conversion for value-added lignin-derived chemicals and products; (3) simplify biomass pretreatment by eliminating hazardous chemicals and expensive process conditions of high temperature and high pressure; and (4) enhance development of depot style facilities near biomass resources or co-locate with existing rural facilities such as sawmills, composite plants, or pulp mills.