Physical methods for break down of cellulose Crystallinity

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Introduction:

Currently Coal, oil and gas are the main sources of where we get our energy and fuel. Liquid fuel is currently mostly obtained by fractioning crude oil. However fossil fuels have substantial problems such as being non renewable and also releasing previously trapped carbon into the atmosphere- increasing global warming. There are substantial deposits of shale oil but these carry an even greater environmental impact in extraction.

Growing crops for fuel is an alternative which is becoming increasingly popular as it is renewable and carbon neutral. However there is the problem that crops take up valuable farm space and there are ethical concerns with using farm land for fuel rather than food

A better alternative would be to extract the fuel from something like trees and current agricultural and timber waste. This has the benefit of not taking up valuable farm space and reduces waste.

To do this the cellulose structure of the woody biomass needs to be broken down to be attacked by enzymes to extract the sugars. A physical method such as ball milling may be a good way to break down the structure. Ball milling at cryogenic temperatures may be a more effective way to do this as it may make the cellulose structure more brittle and susceptible to being broken down. Physical methods also carry no chemical cost in purchasing, disposal etc.

Objectives:

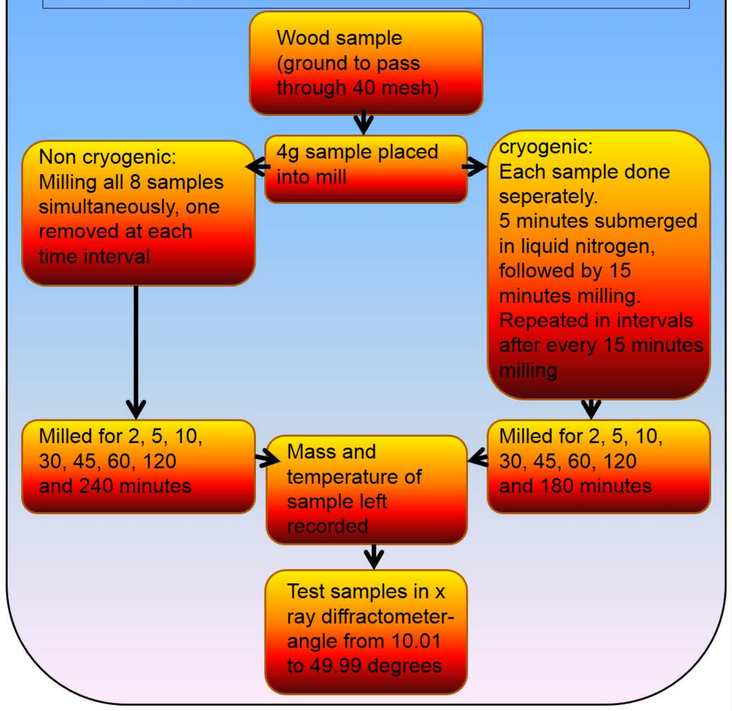
➔To test how physical methods such as ball milling affect the crystalline structure of cellulose in wood and at what time the cellulose structure starts to break down.
➔To see how milling at cryogenic temperatures effects the structure compared to milling at room temperature.
➔To see how much sample is lost after milling due to being released as fine dust etc.
➔To see how milling time affects temperature and whether there is the potential for an overheating problem.

Approach:

To mill the samples for a set amount of time each. Then to mill samples under cryogenic conditions- due to there being no way to keep the mill at a continuous cryogenic temperature I will submerge the mill container with sample into liquid nitrogen before milling, as well as re-submerging for 5 minutes after every 15 minutes to ensure it stays at a low enough temperature. The crystallinity will then be examined by x ray diffractometer.

Materials and methods:

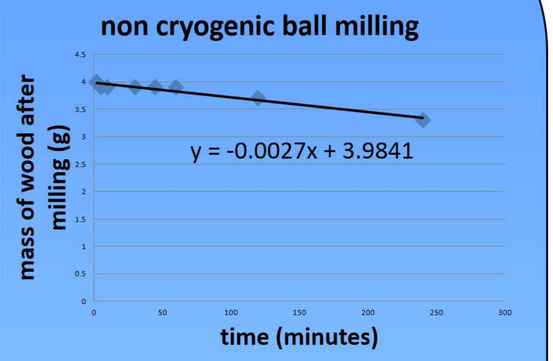
Douglass fir wood chips provided by Vaagen Brothers Lumber Inc. at Colville, WA, liquid nitrogen, Retsch Planetary Ball Mill PM 400 with 4 grinding stations, agate balls and 50 ml grinding jar; Phillip X'Pert x-ray diffractometer

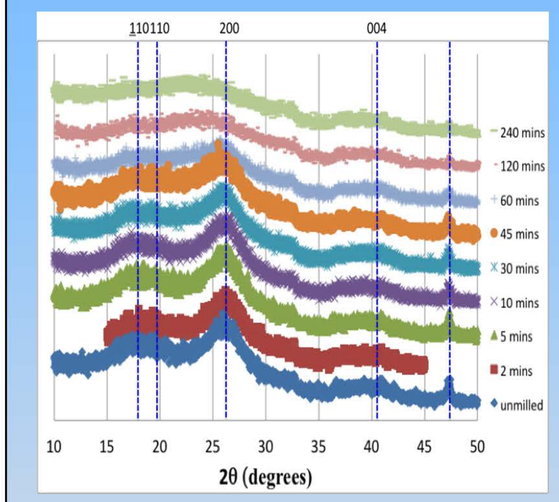


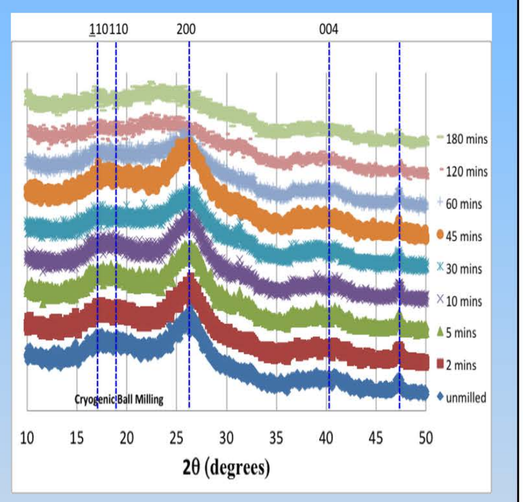
Results:

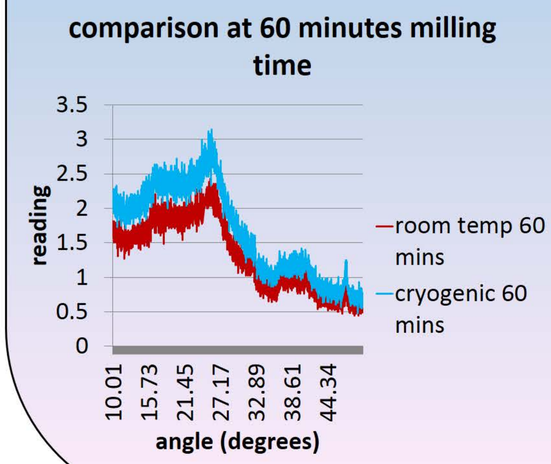
non cryogenic

time in mill (minutes)	mass of wood before milling (g)	mass of wood after milling (g)	temperature of wood before milling (°C)	temperature of wood after milling (°C)
2	4	4	25.8	25.8
5	4	3.9	25.8	26.4
10	4	3.9	25.8	29.8
30	4	3.9	25.8	31.2
45	4	3.9	25.8	31.4
60	4	3.9	25.8	33
120	4	3.7	25.8	34.2
240	4	3.3	25.8	35.8





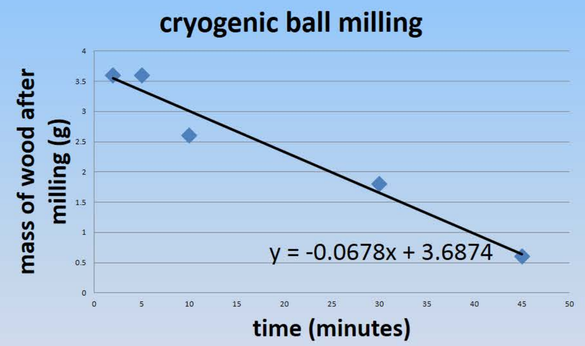




Results:

cryogenic ball milling

time in mill (minutes)	mass of wood before milling (g)	mass of wood after milling (g)	temperature of wood before milling (°C)	temperature of wood after milling (°C)
2	4	4	Too low	Too low
5	4	3.9	Too low	Too low
10	4	4	Too low	-30.8
30	4	3.6	Too low	7.4
45	4	3.6	Too low	-2.8
60	4	2.6	Too low	5
120	4	1.8	Too low	3.2
180	4	0.6	Too low	4.5



Conclusion:

The samples appear to retain their structures during ball milling until about the 60 minute mark. The crystallinity of Douglas-fir wood flour decreased slightly with the increasing amount of ball milling time before 60 mins. Around 60 mins of ball milling, the crystalline structure disappeared and wood flour became fully amorphous. Milling for over this time gives little more effect so will not be cost effective. The cryogenic milling actually seems to break down the sample fast in general but decrease crystalline structure slowly. Particle size analysis will also most likely be needed to draw a better conclusion. The additional cooling during the milling process is of no benefit and is certainly not worth the additional cost. Cryogenic grinding also leads to sample stuck on the inner walls of the grinding jar which makes the entire sample difficult to recover.

Potential further work:

Testing different milling techniques e.g. Ring milling, testing more milling times around the 60 minute mark e.g. 50 minutes, 75 minutes. Drying the samples and containers to see if reduced moisture content affects the crystallinity and ease of break down. The effects of crystallinity on the enzymatic hydrolysis is worthwhile to further investigation.

This work was supported by the National Science Foundation's REU program under grant number