

Multi-scale Visualization and Characterization of Wood Cell Wall Deconstruction during Mechanical Milling

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Background

Despite of decades of research and development, commercialization of a profitable woody biomass conversion process to fuels and chemicals still remains as a significant challenge due to the robust biomass recalcitrance. Pretreatment, therefore, is recognized as a necessary to produce substrate highly susceptible to enzyme. Most of the current biomass pretreatment methods rely on severe chemical treatments and are typically associated with high capital cost, low products yield and the presence of inhibitory compounds to subsequent process.

Mechanical deconstruction of woody biomass by pulverization offers an attractive alternative to address these deficiencies and has a promising potential for implementation as a small scale facility, distributed sugar depot, to produce fermentable sugars.

However, a comprehensive understanding of the mechanism of mechanical pulverization toward producing substrate highly susceptible to enzyme is still lacking.

Objectives

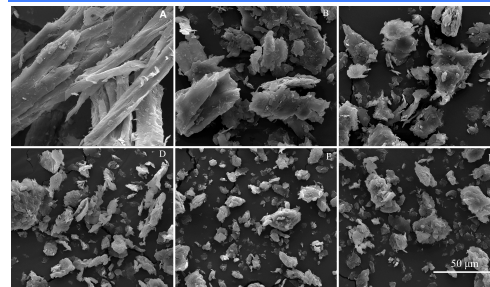
- To delineate the effect of mechanical pulverization on the morphological and physicochemical changes of wood cell wall
- To understand the relationship between properties of milled wood and sugar yield

Research approaches

- Mechanical pulverization with Ring & Puck mill
- Confocal laser scanning microscopy (CLSM)
- Atomic force microscopy
- Electronic microscopy
- X-ray diffraction
- Enzyme assay

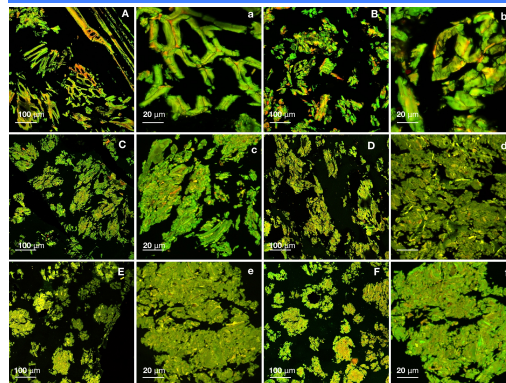


SEM reveals viability of particle size



SEM micrographs reveal fiber delamination and cellular fragmentation of milled wood, 2 min (A), 4 min (B), 6 min (C), 8 min (D), 10 min (E), 12 min (F)

CLSM reveals micro-scale chemical redistribution

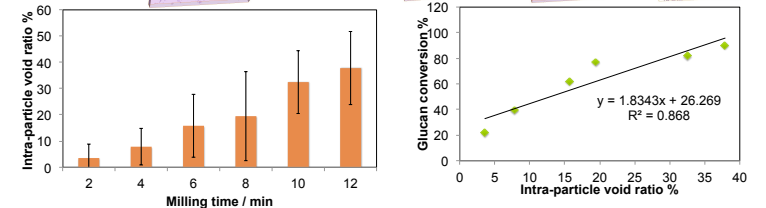
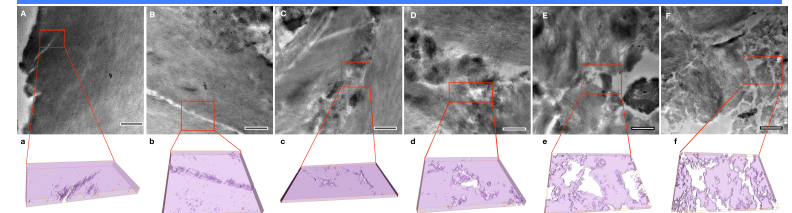


CLSM micrographs of milled wood with chemical composition redistribution; 2 min (A, a), 4 min (B, b), 6 min (C, c), 8 min (D, d), 10 min (E, e), 12 min (F, f)

Correlation between structural features and digestibility

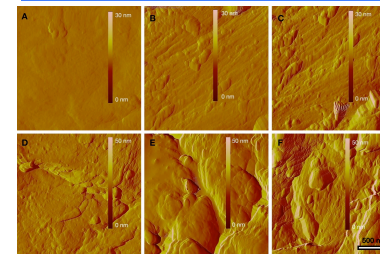
Sample	Glucan release %	Particle size / μ m	Aspect ratio	Inter-particle void %	Surface roughness /mm	Crystallinity index /%	Main peak θ /°	FWHM of main peak /°
RM 2 min	23.73	115	2.05	3.6	0.67	47.9	22.32	3.67
RM 4 min	41.35	26.8	1.95	7.8	1.38	30.82	22.17	4.644
RM 6 min	62.06	21.7	1.83	15.7	2.76	22.64	21.94	5.54
RM 8 min	77.32	17.9	1.77	19.4	3.46	17.32	21.74	6.08
RM 10 min	81.77	15.5	1.73	32.5	4.34	11.92	21.46	6.57
RM 12 min	89.81	15.5	1.63	37.9	6.62	8.87	21.36	7.41
Correlation factor (R^2)	-	0.66	0.98	0.87	0.87	0.97	0.94	0.97

3D-TEM reveals nanofibrillation of cell wall



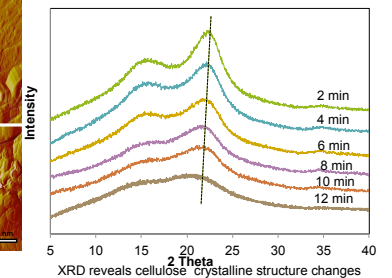
3D-TEM tomography of milled wood cell walls and surface renditions of subvolumes 2 min (A, a), 4 min (B, b), 6 min (C, c), 8 min (D, d), 10 min (E, e), 12 min (F, f), bars 500 nm

AFM reveals surface roughness



AFM micrographs reveal surface changes, 2 min (A), 4 min (B), 6 min (C), 8 min (D), 10 min (E), 12 min (F)

Crystalline structure changes



Conclusion

- Coincident with the particle size reduction of wood material during mechanical milling process includes tissue disintegration, cell wall fragmentation, disordering of layered cell wall fragments, aggregation of disordered cell wall fragments and composition redistribution.
- Macro/micro/nano morphological and physicochemical alteration of wood cell wall contribute to increased accessibility and susceptibility of cellulose.

Acknowledgement

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