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Integration of extrusion and clean fractionation processes as a pre-treatment technology for prairie cordgrass

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HIGHLIGHTS

- Prairie cordgrass (PCG) was extruded at predetermined optimized conditions.
- Clean fractionation (CF) processing was performed on extruded prairie cordgrass.
- Pulps were enzymatically hydrolyzed and glucose yields were measured.
- Optimal conditions resulted in 92% glucose yield, 87% lignin and 95% xylan removal.
- Pre-extrusion improved fractionation effectiveness and glucose yield.

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ABSTRACT

Prairie cordgrass (PCG) was pretreated by sequential extrusion and clean fractionation (CF) processing. Following CF, PCG was fractionated into cellulose, hemicellulose and lignin-rich fractions. Cellulose pulp was then enzymatically hydrolyzed, producing glucose. The main purpose of this study was to produce the highest glucose yield as possible. The effects of time, temperature, catalyst concentration and solvent mixture composition on the fractionation were tested. Different proportions of methyl isobutyl ketone (MIBK), ethanol and water with sulfuric acid as a catalyst were evaluated. Optimal conditions for sequential extrusion and clean fractionation (39 min, 129 °C, 0.69% catalyst, and 28% MIBK) resulted in higher glucose yield (92%), and more lignin (87%) and xylan (95%) removal than for clean fractionation alone. Pairwise comparison of raw PCG with extruded PCG clean fractionation revealed no difference in glucose yields, but xylan and AIL removal were higher in the case of clean fractionation of the pre-extruded PCG.

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1. Introduction

Lignocellulose has been gaining interest of researchers due to its tremendous potential for replacing petroleum in production of energy, chemicals, polymers and pharmaceuticals. Lignocellulosic materials which attract the most attention include softwoods, herbaceous energy crops, and agricultural residues (González-García et al., 2010). Prairie cordgrass is a warm-season prairie grass, widely used in ethanol production research (Cybulska et al., 2009; Karunanithy and Muthukumarappan, 2011b), mainly because it does not compete with food or feed production (due to its coarseness), is relatively high in cellulose, is tolerant to difficult growing conditions, and it is easily available since its growing rate is high (up to 9696 kg DM (dry matter) ha^{-1} can be produced in eastern South Dakota) (Boe and Lee, 2007; Lee et al., 2007). High bulk density of prairie cordgrass (195 kg/m^3) (Karunanithy and Muthukumarappan, 2011a) can improve storability, reduce transportation costs, and ease the handling.

The high potential of lignocellulosic biomass for value added processing is associated with its complex structure, which allows generation of various bio-based products. However, due to its complex nature, lignocellulosic biomass is resistant to processing and requires an efficient pretreatment method.

Extrusion is a physical pretreatment method which utilizes elevated temperature as well as shear stresses. Providing continuous heating, mixing and shearing of the biomass, extrusion is a complex pretreatment method that results in physical and chemical changes in the lignocellulose structure (Karunanithy and Muthukumarappan, 2010). This treatment has an advantage over other physical treatments that utilize high temperatures and pressures (e.g. hydrothermal treatment, steam explosion) by using relatively low temperatures, and therefore preventing by-product

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formation. The most common and bothersome by-products (that can lead to inhibition of the fermentation process) are furfural, 5-hydroxymethyl-2-furfuraldehyde (HMF) and acetic acid (Klinke et al., 2004), which are all formed at temperatures over 180 °C. Cellulose digestibility has been found to increase after extrusion when herbaceous materials were used as feedstock. Pretreatment optimization study performed on prairie cordgrass using a single screw extruder revealed maximum glucose, xylose, and combined sugar yields of 48.3%, 77.8%, and 56.9%, respectively, at the optimized conditions: barrel temperature 90 °C, screw speed 65 rpm, moisture content of 20%, and particle size of 8 mm (Karunanithy and Muthukumarappan, 2011b). Sugar recovery could be improved when combining with other pretreatments. Integration of extrusion with other processes was found to produce more digestible cellulose (Karunanithy and Muthukumarappan, 2011b; Lee et al., 2010). In this study extrusion was integrated with clean fractionation organosolv pretreatment.

Organosolv treatment is a promising biomass processing method that creates an opportunity for implementing the biorefinery concept (Wyman, 1996; Zhao et al., 2009) through biomass fractionation. The principle of this process is based on different affinities of the lignocellulosic components towards different solvents. Generally, organic solvents (alcohol, organic acid, ketone or ester) are used to dissolve the lignin fraction, while cellulose remains in the solid (Zhao et al., 2009). Hemicellulose can be extracted to aqueous phase if water is added to the process. There are many alternatives of the organosolv treatment, using different solvents or mixture of the solvents. These include ALCELL®, Lignol (ethanol as lignin solvent), Acetosolv (acetic acid as lignin solvent), Organocell (methanol as lignin solvent) (Young, 1998), or clean fractionation (MIBK and ethanol as lignin solvents) (Black et al., 1998). Clean fractionation was developed at the National Renewable Energy Laboratory (NREL) and has been found to be an efficient fractionation method when applied to prairie cordgrass, producing high lignin recoveries (87%) and glucose recoveries (84%), at 39 min, 154 °C, 0.69% catalyst, and 9% MIBK conditions (Black et al., 1998; Brudecki et al., 2012).

Since the ideal pretreatment method (applicable to all biomass types, environmentally friendly, having low cost of operation) is yet to be found, many researchers have attempted to integrate two or more pretreatment methods to lower the severity, improve efficiency and selectivity of the processes (Chandra et al., 2007; Wyman, 1996).

Organosolv lignin obtained from the CF process has lower molecular weight and no sulfur in compared with other industrial lignin production processes (Cybulska et al., 2012b). Therefore, organosolv lignin could be an attractive source of low-molecular weight phenols or aromatics. Screening for promising candidates from biorefinery lignin resulted in the following potential utilizations: power, green fuels, syngas (combustion, gasification, pyrolysis, and hydro-liquefaction), macromolecules (carbon fibers, polymer modifiers resins, and adhesives binders) and aromatic chemicals (benzene-toluene-xylene (BTX) chemicals, monomeric lignin molecules, low molecular weight byproducts, fermentation products) (Bozell et al., 2007). In recent study performed using lignin extracted by modified clean fractionation (using ethyl acetate, ethanol and water solvent mixture) from prairie cordgrass, switchgrass and corn stover, revealed a high potential for vanillin production by nitrobenzene oxidation. High yields of the vanillin were observed in all lignin types (approx. 40–60% w/w, calculated per initial lignin input weight), 48% w/w was obtained in case of PCG lignin (Cybulska et al., 2012b).

In this research, a possibility of an integrated process composed of extrusion and clean fractionation was explored. Other study focused on extrusion pretreatment optimization did not produce maximum sugar yields (Karunanithy and Muthukumarappan, 2011b). Extruded PCG was more compacted than raw material; therefore extrusion can reduce feedstock transportation cost. The integration of extrusion and clean fractionation processes was hypothesized to result in more efficient biomass component fractionation, produce higher enzymatic hydrolysis glucose yield or reduce the severity of clean fractionation process conditions, while still obtaining a high fractionation and enzymatic hydrolysis glucose yield.

The objective of this study was to optimize processing parameters of pre-extruded PCG using clean fractionation, including time, temperature, catalyst concentration, and organic solvent mixture composition. The fractionation process effectiveness was evaluated based on the glucose yield, optimization of which was the main goal of this study, while the secondary priority was maximizing the amount of extracted lignin and hemicellulose.

2. Methods

2.1. Sample preparation and extrusion

PCG biomass, with a composition of 37.43 ± 0.59% glucose, 15.48 ± 0.25% xylose, 2.83 ± 0.13% arabinose, 1.40 ± 0.32% galactose, 0.50 ± 0.11% mannose, 16.40 ± 0.67% acid insoluble lignin, and 4.84 ± 0.08% ash, calculated per dry matter (Brudecki et al., 2012), was obtained from a local farm near Brookings, South Dakota. PCG was air dried and ground in a hammer mill (Speedy King, Winona Attrition Mill Co, MN, USA) using an 8 mm sieve. The moisture content of the ground PCG samples was determined according to NREL/TP-510-42621 (Sluiter et al., 2008a), and then was adjusted to 20% wet basis (wb) with deionized water and equilibrated overnight. Next, the moisture–adjusted PCG was subjected to extrusion in a single screw extruder (Brabender Plasticorder Extruder, Model PL2000, Hackensack, NJ). The extruder's screw compression ratio was 3:1 and a barrel length-to-screw diameter (L/D) was 20:1. Predetermined optimized conditions were used for extrusion as follows: 90 °C barrel temperature, 65 rpm screw speed, 20% (wb) moisture content, and 8 mm particle size (Karunanithy and Muthukumarappan, 2011b). Extruded material

Table 1
Experimental design factors and corresponding values.

Coded factors	Factor 1	Factor 2	Factor 3	Solvent composition Factor 4		
	Time (min)	Temperature (°C)	Catalyst - H_2SO_4 (% w/w of solvent)	MIBK (% w/w)	Water (% w/w)	Ethanol (% w/w)
$-\alpha$	16	104	0.12	3	71	26
−1	20	110	0.20	7	63	30
0	30	125	0.39	16	50	34
+1	40	140	0.57	25	40	35
$+\alpha$	44	146	0.65	29	36	35
Additional runs	30	160	0.80	44	24	32

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