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Fluid mechanics relevant to flow through pretreatment of cellulosic biomass

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HIGHLIGHTS

- Compaction of biomass, water absorption and fine particles increase pressure drop.
- Bagasse and switchgrass require more water than poplar to operate in a FT mode.
- Pretreatment pressure drop is unpredictable from measurements at room temperature.
- Water flow compressed switchgrass and bagasse above threshold initial loadings.
- Viscous compression was not observed with poplar.

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ABSTRACT

The present study investigates fluid mechanical properties of cellulosic feedstocks relevant to flow through (FT) pretreatment for biological conversion of cellulosic biomass. The results inform identifying conditions for which FT pretreatment can be implemented in a practical context. Measurements of pressure drop across packed beds, viscous compaction and water absorption are reported for milled and not milled sugarcane bagasse, switchgrass and poplar, and important factors impacting viscous flow are deduced. Using biomass knife-milled to pass through a 2 mm sieve, the observed pressure drop was highest for bagasse, intermediate for switchgrass and lowest for poplar. The highest pressure drop was associated with the presence of more fine particles, greater viscous compaction and the degree of water absorption. Using bagasse without particle size reduction, the instability of the reactor during pretreatment above 140 kg/m^3 sets an upper bound on the allowable concentration for continuous stable flow.

1 Introduction

Producing fuel from lignocellulosic biomass is of interest in the context of developing a sustainable global energy system (International Energy Agency, 2012). The main obstacle impeding production of cost-competitive cellulosic biofuels is the high cost of converting cellulosic feedstocks to reactive intermediates, termed biomass recalcitrance (Lynd et al., 1999; Himmel et al., 2007). In the case of biological conversion of cellulosic biomass to sugars, it has been widely observed that a pretreatment step is necessary in order to achieve high solubilization yields (Mosier et al., 2005; Wyman et al., 2005). There are a wide variety of pretreatment processes, generally involving elevated temperature and pressure and in some cases added chemicals (Mosier et al., 2005; Wyman et al., 2005; Yang and Wyman, 2008). Pretreatment has multiple objectives that are difficult to achieve at once including high recovery of sugars in concentrated form and high yields and rates upon subsequent hydrolysis (Dale and Ong, 2012). Pretreatment operated in a flow through (FT) mode typically achieves higher solids reactivity, higher xylan removal, less sugar degradation and substantially higher removal of lignin compared to pretreatment in non FT configurations at the same temperature and residence time (Mosier et al., 2005; Wyman et al., 2005; Yang and Wyman, 2008).

Operation of FT configurations in a practical context is challenging because the higher water usage compared to non-flow configuration dilutes the sugar streams and increases energy

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consumption (Bobleter, 1994). Several configurations have been proposed and investigated to address these concerns including "recirculation flow", "partial flow" and "counter-current flow"(Bobleter, 1994; Liu and Wyman, 2005; Shao and Lynd, 2013). Operation of FT is also challenging because of the mechanical complexities of arranging a reactor of biomass in a flow type configuration at elevated temperature (180–220 °C) and pressure (1000–2000 kPa). Although continuous counter-current flow operation is common in the wood pulp and paper industry (Marcoccia et al., 2000) and has been reported for wheat straw pretreatment at a pilot scale (Thomsen et al., 2008), operating continuous FT pretreatment at scale is challenging and few studies have reported related fluid mechanics.

Kim et al. (2001, 2002) used an inclined screw reactor designed by NREL intended to achieve counter current flow as a result of water draining to the bottom of the reactor. The reactor was found to be suitable for large particle size softwood residues, but it was unsuitable for severely pretreated softwood, poplar sawdust and chips. For these unsuitable substrates, the abundance of fine particles and smaller average particle size allowed lower drainage rates and caused problems such as compaction, lower void volume, increased pressure drop, blocking, channeling, packing and filter clogging. Sugarcane bagasse's mechanical properties are very prone to cause high resistance to flow (Plaza et al., 2002) making continuous FT pretreatment particularly challenging to implement.

The relevant fluid mechanics, including the pressure drop, must be better understood to address the mechanical complexities of arranging a reactor of bagasse in a flow type configuration. It has been shown that laminar flow through a packed biomass reactor follows Darcy's law, described in Eq. (1) (Plaza et al., 2002):

$$\Delta P/Q = \mu L/KA = \mu R/A, \tag{1}$$

where ΔP (Pa) is the pressure drop across a porous media, Q (m^3/s) is the volumetric flow rate, μ (cP) is the fluid viscosity, L (m) is the porous media length, K (m^2) is the porous media permeability, R (m^{-1}) is the porous media resistance and A (m^2) is the cross-sectional area. The resistance of the media is a function of porosity, which is in turn a function of solids shape and size, compression, swelling or water absorption, temperature and pressure. Porosity, or void volume, is the fraction of free liquid. The remainder of the reactor is occupied by the solid particles. The solids contain a fraction of solid material and a fraction of pore volume containing bound water and air. The free liquid fraction decreases when the bed is compressed or when the solids swell or absorb water, resulting in a smaller void fraction available for flow and thus a higher resistance to flow. The specific resistance of a porous media can be determined from the graph of media resistance against the mass of solids per unit area.

In order to assess the feasibility of operating pretreatment in a FT mode at scale for sugarcane bagasse, the present study measures key fluid mechanical properties.

2. Methods

2.1. Material

Biomass description, analysis and handling were performed as described previously (Archambault-Leger et al., 2012). Switchgrass harvested in November was provided by the Great Lakes Bioenergy Research Center (BER DE-FC02-07ER64494). Sugarcane bagasse was harvested in the fall and kindly provided by Louisiana State University. The biomass glucan, xylan/mannan/galactan (XMG), arabinan and Klason lignin composition is shown in Table 1.

2.2. Pressure drop apparatus and experiments

An apparatus, illustrated in Fig. 1, was designed and built to study the fluid mechanics of water flow through biomass packed beds at reaction temperature (160–220 °C) and pressure (1000–2000 kPa). The apparatus was a 66 cm long stainless steel pipe with an internal diameter of 4.9 cm flanged on both ends to stainless steel manifold blocks, and featured a threaded water inlet and outlet, filters to retain the biomass within the pipe, pressure and temperature monitoring and pressure relief at the inlet allowing to maintain the pressure within 35 kPa of the set pressure. All pipes and fittings were stainless steel 316L, including the 0.1 mm pore size filter placed at the outlet of the reactor to contain the solids. The outlet piping diameter was kept constant (1/2″) and the hydrolyzate only flowed downward to the collection tank to minimize the likelihood of solubilized solids recondensing and clogging the pipes upon cooling. The equipment was mounted solidly on an aluminum extrusion frame and fully enclosed in lexan sheets. The lexan enclosure was vented and the back panel was latched for easy access to the apparatus.

111 ± 1 g of bagasse or switchgrass (95 kg/m^3) or 167 ± 1 g of poplar (140 kg/m^3) were soaked for 24 h and loaded in the 1.17 L reactor. The maximum solid concentration not requiring manual compaction during loading was chosen to ensure uniform solids distribution initially. When the pipe was filled with the desired amount of biomass, the top manifold block was flanged to the pipe and the inlet and outlet pipes were screwed to the manifold blocks. The lexan panel was latched to the extrusion frame, making sure the apparatus was fully enclosed and the venting duct was operational. Water was pumped through the reactor using a diaphragm pump (Wanner Engineering, MN) at 500 mL/min and room temperature. Once the outlet liquid was devoid of air bubbles, the system was pressurized by turning the back pressure regulator at the outlet stream to 2000 kPa. With the back pressure valve set to 2000 kPa, band heaters (Thermal Corporation, AL) installed on the reactor and a circulation heater (Durex Industries Inc.) were turned on and set at the desired temperature (170–200 °C). The start of the reaction time was set arbitrarily as the time when the heaters are turned on and the heating time was observed to be about 15 min by monitoring the temperature inside the bottom and top of the reactor with a thermocouple (Omega Engineering Inc.). The pressure drop was measured throughout the experiment with differential pressure gauges (Orange Research Inc.). After 20 min at reaction temperature, the heaters were turned off. The water flow was stopped and the reactor was depressurized when the temperature at the outlet of the reactor dropped below 60 °C. Experiments were performed in duplicates.

When the pipe was at atmospheric pressure and its temperature was below 60 °C, the inlet and outlet pipes were disconnected from the manifold blocks and the top manifold block was removed. Compressed air at about 140–200 kPa was fed to the bottom manifold block to push the biomass out and to collect it. The biomass samples and collected hydrolysate were refrigerated for later analysis.

Table 1
Feedstock sugar composition before pretreatment with the standard deviation on duplicates.

	% Glucan	% XMG	% Arabinan	% Lignin
Switchgrass	37.9 ± 0.5	24.4 ± 0.3	3.0 ± 0.2	18.0 ± 0.8
Sugarcane bagasse	38.6 ± 1.1	22.1 ± 0.7	1.9 ± 0.1	20.9 ± 1.9
Poplar	37.8 ± 0.5	16.1 ± 1.3	0.9 ± 0.2	21.9 ± 0.5

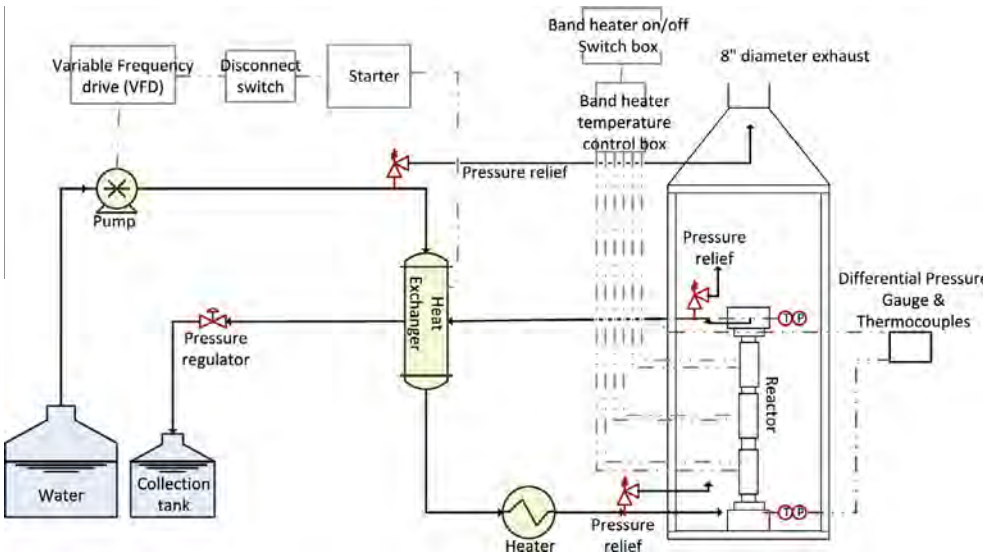

Fig. 1. Schematic diagram of the pressure drop apparatus.

2.3. Specific resistance calculation

Darcy's law (Eq. (1)) can be manipulated as follows to estimate pressure losses from design parameters and empirical specific resistance:

$$\Delta P/L = \alpha \mu c Q/A \tag{2}$$

where ΔP (Pa) is the pressure drop across a porous media, Q (m^3/s) is the volumetric flow rate, μ (cP) is the fluid viscosity, L (m) is the porous media length, K (m^2) is the porous media permeability, A (m^2) is the cross-sectional area, c (kg/m^3) is the solids concentration and α (m^{-1}) is specific resistance of the porous media. In order to measure the specific resistance of a sugarcane bagasse reactor, pressure drop was plotted against flow rate for various bed lengths. An example of this graph is provided in Appendix A, Fig. A1. The slope of each line on this graph is the porous media resistance for a particular bed length. The porous media resistance is then plotted against the dry mass per unit area (Fig. A2), which slope indicates the measured specific resistance for a particular solid concentration.

2.4. Viscous compaction

When the solids were pushed out of the reactor with compressed air, they retained their shape. They were separated in multiple sections and the length of each section was measured. They were then dried in a 102 °C oven and weighed. The concentration, c (kg/m^3), of each section of the pipe as a function of initial bed length was calculated using Eq. (3):

$$C = W/AL, \tag{3}$$

where L was the section's length (m), A was its cross-sectional area (m^2) and W was its dry solids weight (kg).

2.5. Water absorption

A well mixed sample with known moisture content was weighed in an aluminum weigh boat with known weight. Water was added to the weigh boat to completely submerge the sample. The solids soaked in water for 48 h. Two procedures were used to measure the solids moisture content at saturation. In the first procedure, termed saturation moisture after draining water, the excess water was poured through a 0.1 mm filter and the sample as weighed. In the second procedure, termed saturation moisture after vacuum filtration, the sample was transferred to a Büchner funnel equipped with a 40 μm filter (material and distributor) and vacuum filtrated. The sample was transferred back to its weigh boat and weighed. The saturation moisture, m, was calculated using Eq. (4):

$$M = 1 - (M_{\text{dry solids}} - M_{\text{weight boat}})/(M_{\text{saturated solids}} - M_{\text{weight boat}}) \tag{4}$$

3. Results and discussion

3.1. Pressure drop correlations and scale up prediction

Specific resistances, corresponding to the derivative of the cake resistances per area density, were measured in a 2″ pipe at room temperature and pressure for milled bagasse concentrations of 100, 150 and 200 kg/m^3. The pressure drop per unit length was calculated from the measured specific resistances for a 30 m long, 2 m diameter bagasse reactor with a superficial velocity of 0.033 m/s. The calculated pressure drops are about 12,300 kPa/m at 200 kg/m^3, 2580 kPa/m at 150 kg/m^3 and about 197 kPa/m at 100 kg/m^3. The pressure drop, proportional to water viscosity, was expected to be 5.6 times lower at 220 °C than at 25 °C because of the lower water viscosity (Fig. A3). Nevertheless, the instability of the reactor due to the pretreatment reaction impeded predicting pressure drop at reaction temperature from measurements at room temperature. Thus, pressure drop data must be collected at pretreatment reaction condition.

3.2. Time profile of pressure drop across the reactor for various feedstocks

Pressure drop across packed beds was monitored during the hot water FT pretreatment of bagasse, switchgrass and poplar milled to pass through a 2 mm sieve. Sugar cane bagasse and switchgrass were initially loaded with 95 kg/m^3 and poplar with 145 kg/m^3, the water flow rate was 500 mL/min. The observed pressure drop due to fluid flow was highest through bagasse, intermediate for switchgrass, and lowest for poplar as shown in Fig. 2. The pressure drop before the temperature was brought down and the reactor was depressurized was about 248–296 kPa/m in the bagasse

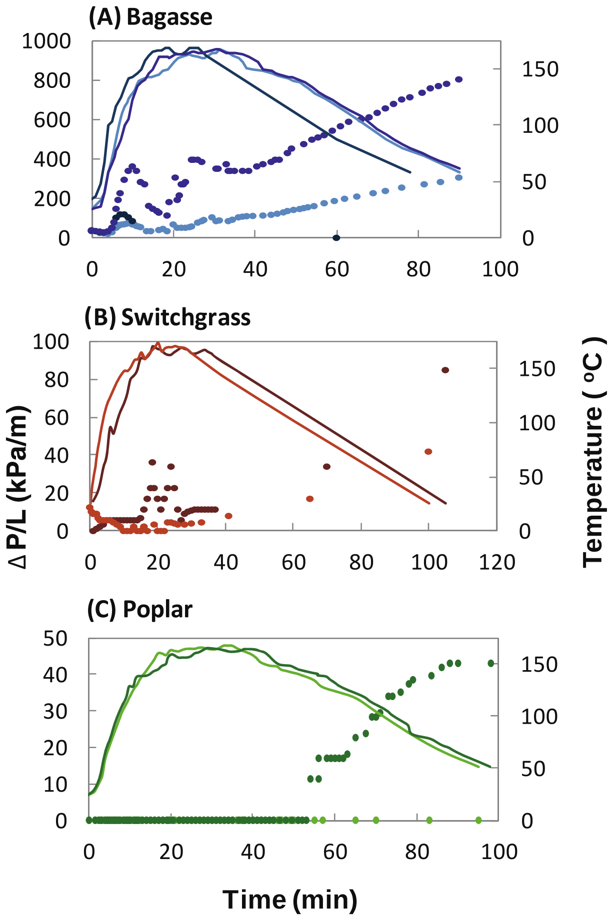

Fig. 2. Pressure drop time profile across the reactor in the FT reactor for milled (A) bagasse, (B) switchgrass and (C) poplar. Distinct colors refer to separate experiments. Dots are pressure drop per unit length (kPa/m) data and lines show each experiment's corresponding temperatures (°C).

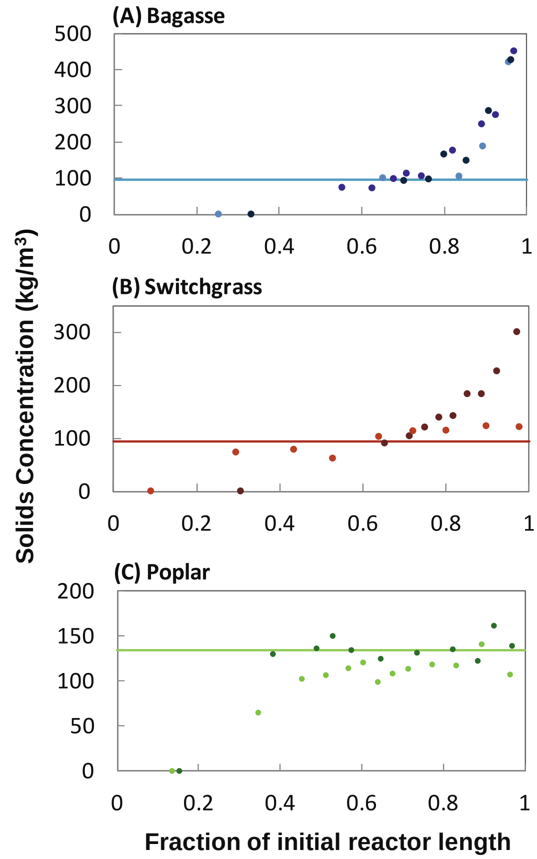

Fig. 3. Solid concentration distribution in the FT reactor for milled (A) bagasse, (B) switchgrass and (C) poplar. Distinct colors refer to separate experiments. Horizontal lines show the initial solids concentration in the reactor.

reactor, 21 kPa/m in the switchgrass reactor and smaller than 6 kPa/m in the poplar reactor.

During heat up and at the beginning of the reaction, pressure drop oscillations were observed as shown in Fig. 2. The oscillations stopped after the reaction was complete. This unstable behavior suggests that close attention should be paid to the design of the reactor during large scale FT pretreatment implementation, for example by providing mixing to minimize compaction and rearrangement of the biomass bed. As the temperature was brought down, the pressure drop increased linearly with increasing water viscosity, which increases with decreasing temperature according to the Arrhenius equation (Bird et al., 2002). An Arrhenius model for the temperature dependance of viscosity predicted the increase in pressure drop observed as the reactor was cooled, as shown in Appendix B. However, the temperature dependance of viscosity did not predict the pressure drop during the reaction due to the instability of the biomass bed. The compaction of the biomass, its water absorption and its particle size influenced pressure drop.

3.3. Viscous compaction

Water flow exerts a viscous force on biomass, causing it to compact as illustrated for milled substrates in Fig. 3. The horizontal lines in Fig. 3 indicate the initial concentration of the reactor and the data points indicate the concentration after the reaction along the length of the reactor. The viscous force is cumulative through the bed, as the water flow pushes on the first layer of biomass, which then pushes on the second layer along with the water flow and so on. Of the milled feedstocks studied, bagasse was the most susceptible to compaction. It compacted above a threshold initial concentration of 95 kg/m^3 and formed a plug containing about 450 kg/m^3 at the water outlet. Switchgrass also compressed to a lesser extent compacting above a threshold initial concentration of 95 kg/m^3 and forming a plug containing about 300 kg/m^3 at the water outlet. Poplar did not compress significantly. Understanding the viscous compaction during FT pretreatment for several feedstocks is important for designing a reactor with a solids concentration that will allow flow. Preventing compaction with mixing or other physical means would greatly reduce the observed pressure drop.

3.4. Water absorption

When biomass absorbs water, free water becomes bound water, reducing the volume available for water flow. Biomass behaves similarly to a sponge, absorbing and holding many times its weight in water. Fig. 4 illustrates the sponge like behavior of bagasse, poplar and switchgrass with and without particle size reduction as well as after pretreatment. Bagasse with no particle size reduction absorbed 10.7 times its weight in water, the most of all substrates studied. Poplar chips absorbed 2.5 times its weight in water, the least of all substrate studied. Keeping in mind that free water in the reactor is necessary for flow, the absorption behavior determines how much biomass can be loaded in the FT reactor. Therefore, a bagasse FT reactor requires a much lower solids

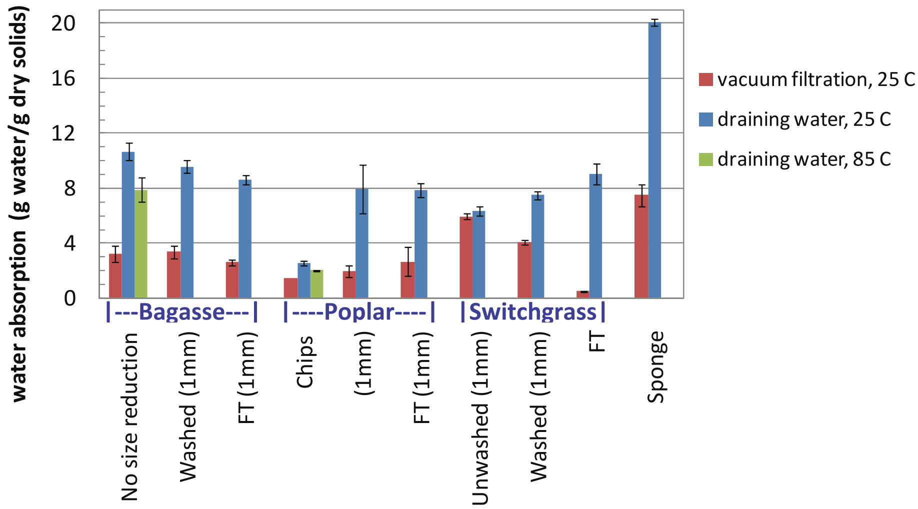

Fig. 4. Water retention for various substrates.

concentration compared to a poplar FT reactor. The higher required water content makes bagasse FT pretreatment more challenging to implement than poplar FT pretreatment because more water usage causes more sugar dilution and higher energy consumption. Thus, further work is required to determine whether the extra water needed is acceptable for sugar dilution and energy consumption.

The water retained by the biomass after draining water but removed after vacuum filtration is water that is lightly bound or held by surface tension, but that can relatively easily be moved out of the biomass. For example, while bagasse with no particle size reduction absorbs 10.7 times its weight in water, vacuum can pull 70% of this bound water out of the biomass indicating that applying a force can move the lightly bound water. This suggests that a pressure differential in a biomass reactor can force water movement even if the solids concentration is above its saturation level. Interestingly, vacuum does not pull a significant of water out the saturated unwashed switchgrass whereas it pulls out 45% and 94% of the bound water for washed and FT pretreated switchgrass, respectively. The very fine particles give the saturated unwashed switchgrass the consistency of mud and create a plug where no water can move. As the fines are removed during washing and the fibers structure is opened during pretreatment, there is more volume available for the water to move.

Biomass water absorption tends to be lower at higher temperature. Bagasse as received and poplar chips absorb 27% and 20% less water at 85 °C than at 25 °C, respectively. Therefore, the water absorption should have a smaller effect on pressure drop at reaction temperature than at room temperature.

3.5. Particle size

Removing the particles smaller than 85 μm from the milled bagasse and maintaining the same initial solids concentration (95 kg/m^3) reduced the pressure drop during the reaction from 248–296 kPa/m to 33 kPa/m, only slightly higher than in the switchgrass reactor (21 kPa/m). Consistent with previous studies showing that smaller particle size result in lower drainage rates (Lee and Bennington, 2005), the presence of more fine particles in the milled bagasse compared to switchgrass and poplar mostly explain the higher pressure drop across the packed bed shown in

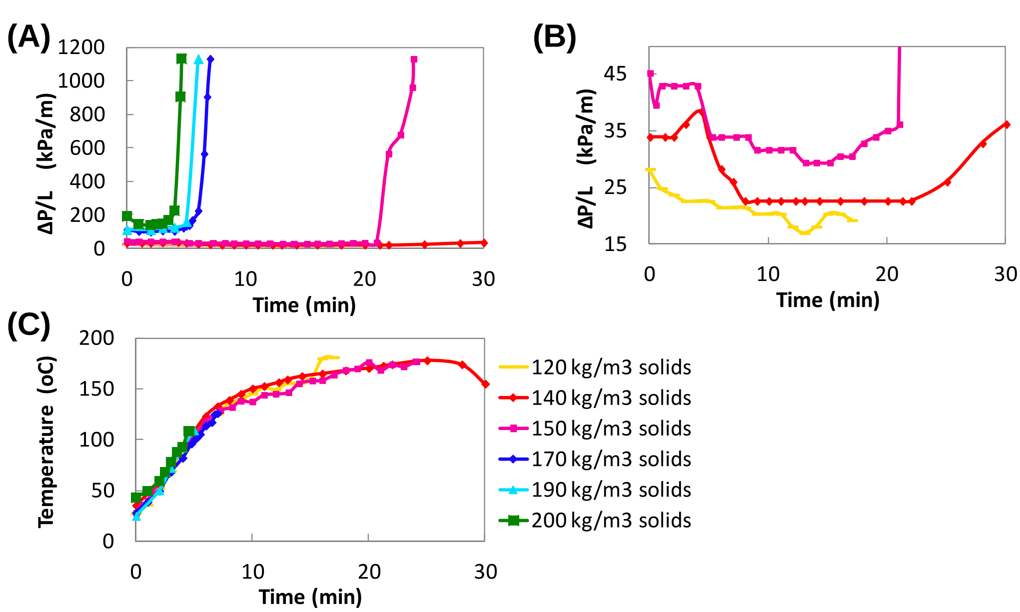

Fig. 5. (A) Pressure drop per unit length for several bagasse concentration as reaction proceeds, no size reduction. (B) Figure A zoomed in on 120–150 kg/m^3 solids. (C) Temperature profile corresponding to figures A and B.

Fig. 2. No pressure drop was detected for reactors packed with the same initial concentration of bagasse with no particle size reduction. Hence, smaller average particle size causes larger pressure drop.

Flow through milled bagasse is neither practical from a fluid mechanical perspective because the pressure drop per unit length is too high nor from a logistical perspective because an additional size reduction processing step is undesirable. The pressure drop across a reactor of bagasse as received from the sugar mill diffusers is of greater practical interest. At room temperature, the pressure drop across the reactor packed with 100 kg/m^3 of bagasse as received was below 6 kPa/m. At 120 kg/m^3, the pressure drop was 34 kPa/m and it increased exponentially to 192 kPa/m at 200 kg/m^3. No flow was possible at 400 kg/m^3.

The pressure drop per unit length at different bagasse concentration during the pretreatment reaction is plotted in Fig. 5A and zoomed in for the low pressure drop conditions in Fig. 5B. Fig. 5C shows the temperature profiles corresponding to the reactions plotted in Fig. 5A and B. At pretreatment temperature and pressure, the pressure drop decreases slightly for reactors initially loaded with 140 kg/m^3 solids or less as expected from the decrease in water viscosity. However, at 150 kg/m^3 the pressure shoots up uncontrollably at the end of the reaction at about 175 °C. At 170–200 kg/m^3 the pressure shoots up uncontrollably at the beginning of the reaction as the temperature increases above 100 °C. This sharp rise in pressure is unacceptable in a practical context because it would lead to a highly unstable pretreatment process. This sets an upper bound of 140 kg/m^3 on the bagasse concentration acceptable for practical pretreatment operation in a FT mode. Since the skeletal density of bagasse is about 1500 kg/m^3 (Rasul et al., 1999), this corresponds to a low bound of 6:1 on the allowable mass ratio of liquid to solids for FT operation. The sharp rise in pressure seen at pretreatment conditions is a consequence of the non-ideality of the reaction fluid mechanics and indicates that the pressure drop at reaction conditions cannot be predicted from the reactor behavior at room temperature.

4. Conclusions

Bagasse and switchgrass require more water than poplar to process in a flow-through mode. Higher pressure drop was associated with greater biomass compaction, greater water absorption, more fine particles and smaller average particle size. The instability of the bagasse bed during pretreatment above 140 kg/m^3 sets an upper bound on the allowable concentration for continuous stable flow. The data provided in this study provide useful information to identify conditions for which FT pretreatment can be implemented in a practical context. Further work on thermodynamic and economic considerations is required to define the feasible operating region for FT pretreatment.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.biortech.2014.01.035.

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