

Rheology and extrusion of high-solids biomass

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ABSTRACT: Economical biorefining of lignocellulosic biomass (LCB) requires processing high-solids particulate streams. We have developed new techniques and testing protocols to measure the rheological properties of high-solids LCB using a modified torque rheometer (TR). The flow field in the TR is similar to that of a twin-screw extruder and for modeling purposes can be adequately represented as a dual-Couette viscometer. Our experiments show that LCB exhibits Bingham plastic behavior with very large yield stresses. We observe that in the initial stages of mixing, torque values are extremely large and erratic. During this period, considerable particle-size reduction takes place with correspondingly large energy consumption. We show that the addition of a rheological modifier (e.g., carboxymethyl cellulose) reduces biomass apparent viscosity and mixing energy requirements. We take advantage of this effect to further investigate the viability of continuous processing by extrusion.

Application: This research was conducted to measure the rheological properties of high-solids biomass for biorefining applications. Determining the rheological properties of high-solids biomass is challenging. In this paper, we attempt to elucidate these challenges, as well as devise experimental techniques and quantitative measures for their determination.

Lignocellulosic biomass (LCB) is a nanoscale assembly of cellulose, hemicellulose, and lignin. The extraction and conversion of these components to fiber, carbohydrates, or other chemicals has significant economic value. The cost of processing LCB can be reduced by increasing the concentration of insoluble solids in various process operations by reducing the energy requirements associated with water removal. However, increasing solids concentration increases the apparent viscosity of biomass slurries, which makes mixing and conveying operations more challenging. Ideally, the necessary treatments and reactions would take place in a single continuous process. However, most biomass conversion processes require several stages of chemical, thermal, and mechanical treatments for particle-size reduction and extraction of products. Each of these unit operations requires an efficient method of conveying materials. Measures of flow characteristics (rheology) of biomass slurries throughout various stages of treatments would greatly facilitate the design, implementation, and characterization of industrial biorefining operations.

Many varieties of LCB are under consideration as potential feedstocks for biorefining applications, including agricultural residues (e.g., corn stover), annual crops (e.g., switchgrass), and hybrid woody plants (e.g., willow, aspen). Each of these materials has unique attributes that can influence their rheological properties: chemical composition, particle shape, particulate size distribution, and moisture content. For example, corn stover has a high cellulose content (~33%); contains diverse anatomical components (rind, core, nodes, leaf, and cob); is harvested with traditional agricultural equipment (balers or forage choppers); has high moisture (approximately 50

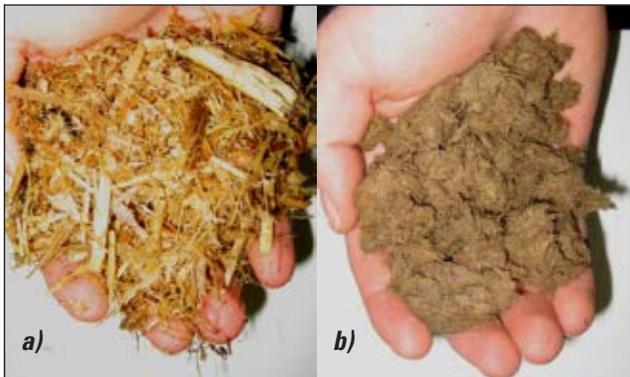
wt% solids) and low bulk density (usually less than 100 kg/m³) [1]. In its harvested condition, corn stover does not flow easily or predictably. This behavior makes it extremely challenging to develop handling systems, particularly in the primary treatment stages where coarse, saturated LCB must be conveyed. It is not until considerable size reduction occurs and/or the composition is sufficiently diluted that flow behaviors become more predictable and can be characterized by conventional rheometry.

Previously, we used a torque rheometer (TR) to evaluate the rheological behavior of various wood pulps, recycled papers, and paper mill residuals, generally in combination with one or more rheological modifiers [2, 3]. These tests revealed the extreme flow resistance of high consistency pulps and the large viscosity reductions achievable upon addition of water-soluble polymers (e.g., carboxymethyl cellulose) [4]. With this insight, we were able to process these materials using a twin-screw extruder [5]. These experiments have been expanded to include other forms of LCB. What follows is a discussion of new adaptations, techniques, and analyses developed to quantify the rheological properties of high-solids LCB using a torque rheometer. We also introduce the quantities Specific Mixing Energy (SME) and Specific Mixing Intensity (SMI) that are useful in comparing results from the torque rheometer and twin-screw extruder.

PREPARATION OF LIGNOCELLULOSIC BIOMASS

Several types of LCB were collected, processed, and conditioned before testing. The specific details of these treatments are described elsewhere [6, 7] and are summarized here. Wood pulps were prepared by repulping recycled paper (usu-

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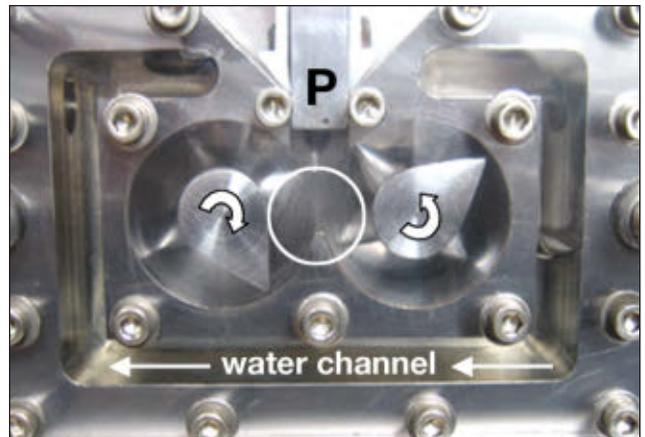
1. Hammermilled and washed corn stover (29 wt% solids) used in torque rheometry and extrusion trials. (a) raw stover, (b) extruded stover.

ally old corrugated containers [OCC] or newsprint) at 10 wt% solids in a hydrolpuler; dewatering in a bladder press to 25 wt% solids; and then shredding to produce a marble-sized crumb pulp. Corn stover was acquired in both baled (whole stalk) and ensiled (chopped) forms. The baled stover was hammermilled, washed, and dewatered before testing (Fig. 1). If solids contents above the level achieved by the bladder press were required, the LCB was pressed between dry blotter papers in a hydraulic press to remove excess water. Most of the rheometry and extrusion tests on LCB were conducted within the range of 20 to 40 wt% solids. When rheological modifiers were used, they were added as dry granulated powder. Addition levels were based on the total weight of saturated sample (wt%). In preparation for extrusion, the powders were simply “salted” onto the LCB and modestly mixed by hand in a bin, then stuffed into the extruder. All calculations related to mass-specific properties (i.e., SME and SMI) are based on the sample dry-weight mass.

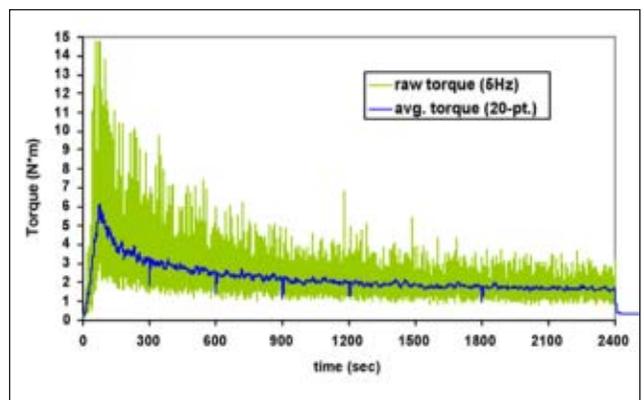
Torque rheometry

Torque rheometers (TRs) have been used to measure rheological properties of diverse materials, such as polymer melts [8, 9], food pastes [10], and wet granulations of pharmaceutical excipients [11]. For our purposes, we redesigned a Brabender Plasticorder (C.W. Brabender Instruments; South Hackensack, NY) to specifically measure the rheological properties of high-solids LCB (Fig. 2). This design incorporates a water-jacketed chamber for temperature control, a thermo-well to measure the temperature of the sample within the chamber, a polycarbonate cover to view mixing, and Teflon seals to prevent water leakage. We selected lobed-cam mixing elements to replicate the intense shear mixing that occurs in the kneading zones of a twin-screw extruder. We also reconfigured the gear head drive system to incorporate an in-line torque sensor and slip clutch. There is a 3:2 differential in rotational speed between the left cam (direct-driven) and the right cam (follower). The capacity of the mixing chamber is 100 cc.

In a typical test, LCB is loaded into the mixing chamber



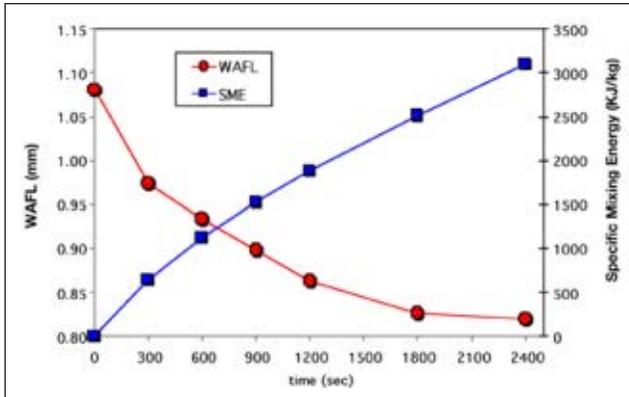
2. Front view of stainless steel torque rheometer (TR) with water channel for heating and cooling. Rotation arrows indicate the counter rotation direction of impellers. Circle indicates location of intense mixing and plug formation.



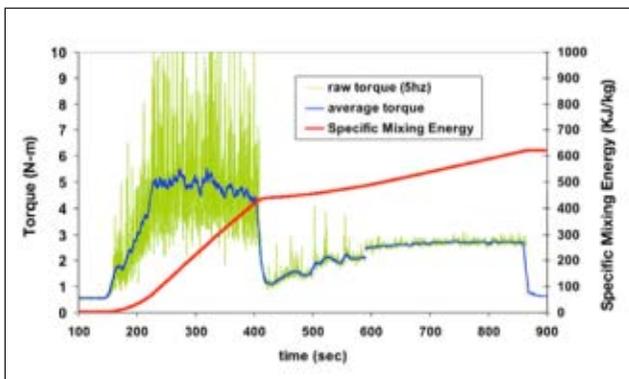
3. Torque response of newsprint pulp at 20 wt% solids.

while the cams rotate slowly. Once loaded, the plunger (P) with attached thermo-well is inserted. A data acquisition system records the torque and temperature (usually at 5 Hz) as the test proceeds. To smooth the torque data, an average torque was calculated by a 20-point (4-s) moving average and is shown in each data graph. For the tests reported here, the mixing chamber temperature was held at 40°C. Several procedures were developed to test high-solids LCB using a torque rheometer. Three of the most common tests are described in the following paragraphs.

The first test simply demonstrates the intense mixing action that occurs within the torque rheometer and reveals the extent of fiber damage that can occur in this mixing environment. A charge of newsprint crumb pulp (20 wt% solids) is quickly fed into the mixing chamber as the drive speed is held at a constant 55 rpm (Fig. 3). In this test, the mixing chamber is filled to only half capacity (50 cc). As is typical for raw pulps, the torque quickly peaks and becomes highly erratic. Under these conditions, adding more material would exceed the capacity of the torque sensor and likely stall the mixer. As the test proceeds, the torque gradually diminishes, but the erratic fluctuations remain. To quantify the extent of



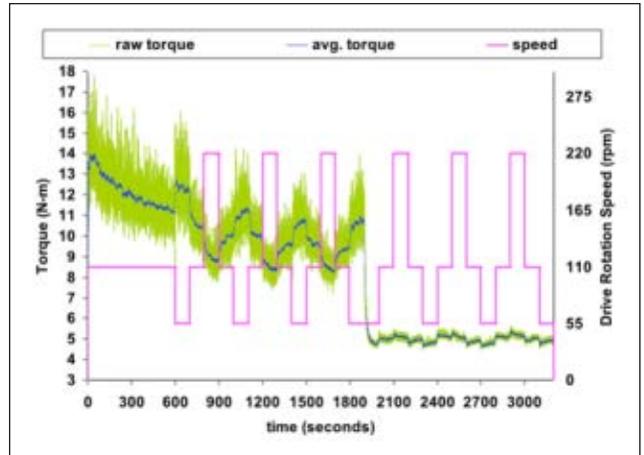
4. Weight-average fiber length (WAFL) and specific mixing energy (shown cumulative) from periodic sampling throughout the TR newsprint test.



5. Torque response and specific mixing energy (shown cumulative) for pulp made from old corrugated containers (OCC) at 25 wt% solids. Sodium-carboxymethyl cellulose (CMC) powder is added at 400 s. At 600 s, the mixing chamber was full.

fiber damage, the test was stopped periodically to withdraw a small sample for measuring the fiber length distribution with a Kajaani fiber-length analyzer (FS-100) from Metso (Helsinki, Finland). The length-weighted average fiber length (WAFL) was measured for each sample withdrawn (Fig. 4). Initially, there is a sharp decrease in WAFL. Throughout the test, WAFL continues to decrease until the average torque level becomes relatively stable (at about 1800 s). Also shown in Fig. 4 is the specific mixing energy (SME), which represents the accumulation of mechanical energy transferred to the sample mass as mixing proceeds. SME is calculated as the time-integrated mixing power per sample mass, where mixing power is calculated as the product of average torque and mixer speed.

The second test demonstrates the viscosity changes that can be affected by the addition of a rheological modifier. In this test, a charge of OCC crumb pulp (25 wt% solids) was gradually added to the mixing chamber as the drive speed was held at 55 rpm (Fig. 5). Again, filled to only half capacity, the torque quickly peaks and becomes highly erratic. After about 250 s of mixing, a small amount (0.5 wt%) of sodium-carboxymethyl cellulose (CMC, Aqualon 7H4F; Ashland, Wilmington,



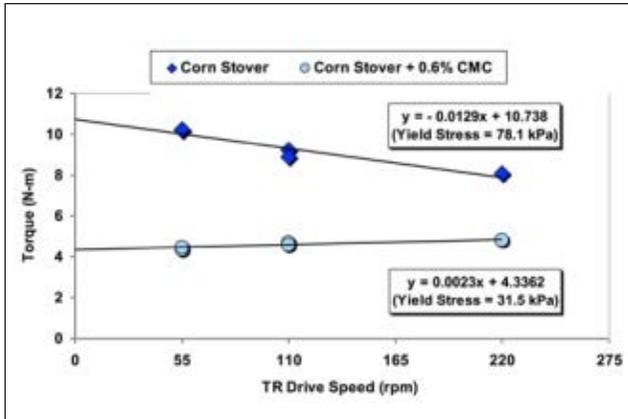
6. Torque response for hammermilled corn stover (29 wt% solids). At 600 s, the drive rotation speed was cycled in three steps (55, 110, and 220 rpm) at 100-s intervals. At 1900 s, CMC powder was added. At 2000 s, three more speed cycles were repeated.

DE) was added. Within seconds, there is a rapid torque drop, the torque fluctuations diminish, and a homogeneous fiber “paste” forms. At this point, it is possible to add fresh crumb pulp into the mixing chamber and fill the chamber to capacity without risking of stalling the TR. Once full (at about 600 s), a stable torque level is achieved.

Fig. 5 also shows the cumulative SME. We find that during the initial period of erratic torque fluctuations, SME increases steeply. However, once CMC is added, SME increases more slowly. For similar tests with other water-soluble polymers acting as rheological modifiers we have observed a range of torque drop rates, final torque levels, and torque fluctuations [4, 12].

The third test illustrates a new protocol developed to use the torque rheometer as a traditional Couette viscometer, but with dual-concentric cylinders. This approach was originally tested for Newtonian fluids using a similar TR [13] and has since been used to measure rheological properties of complex fluids [9]. We have adopted this approach to further characterize the rheological properties of LCB. However, two additional assumptions must be considered. We first assume that each lobed-cam impeller can be modeled as a rigid cylinder. An effective radius can then be determined by measuring the torque response at various rotation rates for Newtonian calibration fluids [6, 7]. We further assume that the rheological behavior of LCB can be modeled as a yield stress (Bingham) plastic material. As such, the torque-rotation rate data can be fit with a Bingham model (using the effective radius) to extract yield stress and plastic viscosity [6, 7].

To measure the rheological properties of LCB using the third test approach, a three-step variable rotation rate test was developed (Fig. 6). In this test, the mixing chamber is first completely filled with hammermilled corn stover (29 wt% solids) and conditioned for 10 min (600 s) at 110 rpm until a relatively stable torque response is obtained. The rotation rate



7. Torque/speed response of corn stover (29 wt% solids) with linear fit of plastic viscosity.



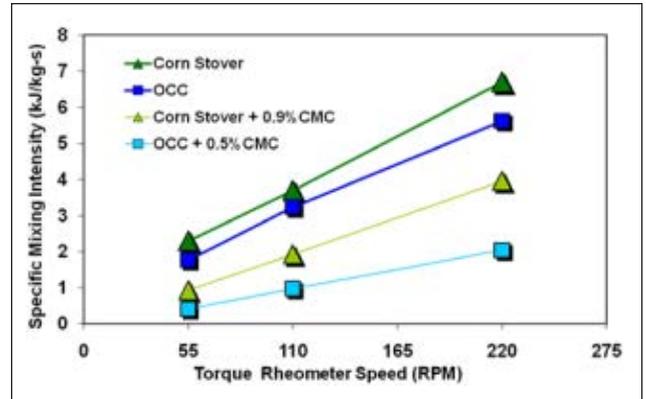
8. Homogeneous corn stover "paste" recovered after a TR test with CMC added.

is then cycled three times between 55, 110, and 220 rpm as shown. We fit the torque-rotation rate data of the third cycle with the Bingham model to extract yield stress and plastic viscosity (Fig. 7). After the third cycle, the test can be extended by adding a rheological modifier (0.6 wt% CMC, shown at 1900 s in Fig. 6). After three more cycles, we again fit the torque/rotation rate data from the last cycle with the Bingham model to extract yield stress and plastic viscosity. At the completion of each test, the material is collected and saved for further testing (Fig. 8).

The three-speed variable speed test also makes it possible to calculate specific mixing intensity (SMI), or the instantaneous rate at which mechanical energy is transferred to the sample at different mixing speeds. For TR tests, SMI is calculated as the ratio of mixing power to sample mass. Figure 9 shows SMI values for corn stover and a similar test on OCC.

Twin-screw extrusion

Twin-screw extruders (TSEs) are a specialized category of continuous processing equipment that is especially suited for aggressive mixing under reactive conditions. They contain synchronous, parallel axis shafts with intermeshing screw



9. Specific mixing intensity versus TR rotation rate for corn stover (29 wt% solids) and OCC (25 wt% solids) compositions.

elements that can be configured to impose very high compression and shear forces on materials. TSEs are widely used in the polymer and food processing industries but have only recently been considered for continuous processing of LCB. For our extrusion trials, we use a Davis-Standard (Pawcatuck, CT), 32-mm, co-rotating TSE. This extruder has interchangeable screw elements that can be configured into multiple conveying and kneading zones to control mixing intensity. It also has a variable speed drive capable of speeds to 500 rpm and is instrumented to measure motor load (reported as a percentage of the maximum motor load). A torque wrench was used to convert the percentage of motor load to screw torque. All extrusion trials were run at 40°C.

We have previously shown that two key factors must be considered when extruding high-solids LCB. These are screw configuration and the judicious selection of a rheological modifier [5]. If particle-size reduction or homogenization is desirable, an aggressive screw configuration can be assembled. But this will also increase mixing intensity. The addition of a rheological modifier is necessary to moderate these extreme shear stresses and prevent the extruder from plugging. To illustrate a typical extrusion trial on LCB, we assembled an aggressive screw configuration containing three kneading zones with progressively increased mixing severity. The purpose for selecting this configuration is to examine the extrusion characteristics of two very different LCB feedstocks: hammermilled corn stover and a wood pulp blend (75 wt% OCC + 25 wt% newsprint). Carboxymethyl cellulose was used as the rheological modifier for both trials. Table I lists the composition of the LCB feedstocks and the steady-state extrusion conditions recorded at each screw speed. For these trials, we recorded the steady-state motor load (torque) and measured the mass flow rate (throughput) over a range of four screw speeds (100, 200, 300, and 400 rpm). Mixing power, SME, SMI, and residence time were calculated from these measurements.

For extrusion purposes, mixing power is calculated as the product of screw torque and screw speed (similar to torque rheometry). However, in extrusion processing, material flows

					WET	DRY						
Speed (rpm)	Speed (rad/sec)	Torque (% load)	Torque (N-m)	Power (watt)	Throughput (g/min)	Throughput (g/min)	Throughput (kg/h)	Specific Mixing Energy (kJ/kg)	Residence Time (sec)	Barrel Volume (ml)	Specific Mixing Intensity (kJ/kg-s)	
100	10.5	18	32.3	338	400	100	6.0	203	113	750	1.80	
200	20.9	10	17.6	369	786	197	11.8	112	57	750	1.96	
300	31.4	12	21.3	668	882	221	13.3	182	51	750	3.56	
400	41.9	22	39.7	1661	1508	377	22.6	264	30	750	8.86	
Corn stover + 0.9% CMC (29% solids)												
Speed (rpm)	Speed (rad/sec)	Torque (% load)	Torque (N-m)	Power (watt)	Throughput (g/min)	Throughput (g/min)	Throughput (kg/h)	Specific Mixing Energy (kJ/kg)	Residence Time (sec)	Barrel Volume (ml)	Specific Mixing Intensity (kJ/kg-s)	
100	10.5	63	115.0	1204	86	25	1.5	2897	523	750	5.54	
200	20.9	28	50.7	1061	182	53	3.2	1207	247	750	4.88	
300	31.4	33	59.9	1881	277	80	4.8	1405	163	750	8.65	
400	41.9	31	56.2	2354	212	61	3.7	2297	212	750	10.82	

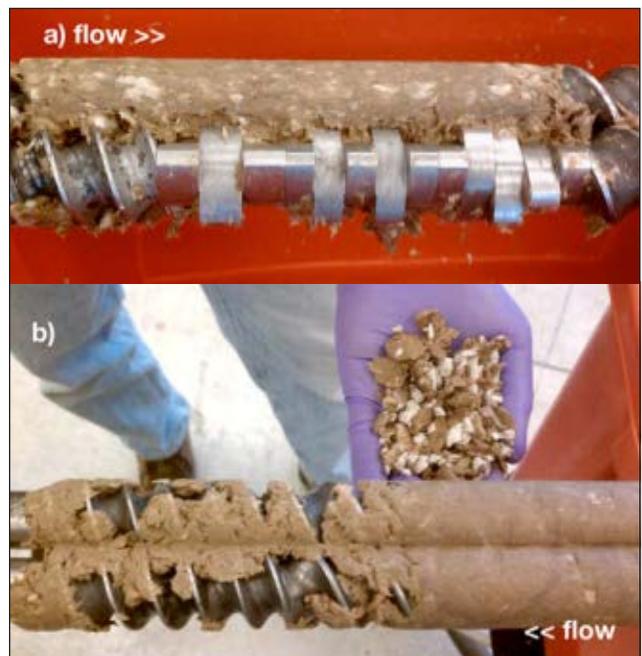
I. OCC + news + 0.5% CMC (25% solids)

continuously through the barrel and experiences many levels of mixing intensity (compression and shear) depending on the screw configuration, and more specifically, the configuration of the kneading zones. Mechanical energy is transferred to the LCB as it is conveyed along the length of the barrel. However, the instantaneous rate of energy accumulation (SMD) at any position within the extruder is unknown. Therefore, for extrusion purposes, SME is defined as the total mechanical energy transferred to the LCB for the length of time it is resident in the extruder barrel. Under steady-state conditions, SME is constant and can be calculated as the ratio of mixing power to throughput (Table I). Since an instantaneous value of SMI cannot be measured, an average value is instead calculated by dividing SME by residence time.

At the end of the wood pulp trial, the extruder was stopped fully packed and the screws were removed to qualitatively examine the levels of dispersion in each kneading zone. **Figure 10** shows the first and third kneading zones. In the first kneading zone (Fig. 10a), the OCC and newsprint crumb pulp blend is not well dispersed. However, by the third kneading zone (Fig. 10b), the blend is more homogeneous.

DISCUSSION

The torque rheometry and extrusion trials reported here were selected to demonstrate various methods that can be adopted to characterize the rheological properties and process characteristics of high-solids LCB. In the TR test on recycled newsprint at 20 wt% solids, initial torque levels can peak quickly and become erratic. We believe the erratic torque fluctuations are caused by the formation and compression of discrete fiber “plugs” that intermittently occur in the intense mixing zone of the TR (circled area shown in Fig. 2). During peak torque



10. Sections of screws exposed after extrusion trial with OCC/newsprint pulp blend. (a) first kneading zone showing coarse mixing elements, packed with relatively unmixed pulps, (b) third kneading zone (not exposed, but packed with well dispersed pulp) shown next to handful of crumb pulp feedstock.

events, free water is pressed out of these plugs, creating an instantaneous elevation of the local solids level that results in an increase in flow resistance and network (floc) strength [14]. This condition illustrates the principal limitation encountered in pumping biomass slurries. Pump pinch points are known to locally dewater slurries, making them exhibit solid-

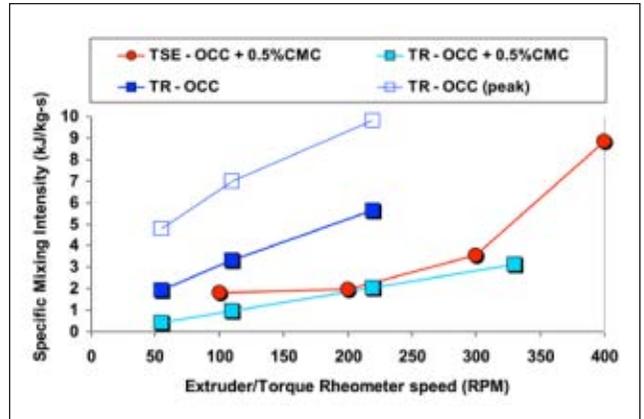
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like behaviors. Furthermore, it is important to note that although we use the average torque values to calculate mixing power, the peak torque values are at least double the average torque values, and at the same rotation rate, correspond to peak power levels that are also more than double the average mixing power.

Processing high-solids LCB can be greatly facilitated by the addition of a rheological modifier. The torque rheometer is a useful instrument to quantify these effects. For the OCC pulp studied here, addition of only 0.5 wt% CMC caused an immediate torque drop and diminished torque fluctuations, resulting in the formation of a homogeneous fiber paste. The time required for the torque drop to occur is significant, considering that continuous industrial processes (e.g., extrusion) may allow only seconds for the rheological modifier to disperse, hydrate, and alter viscosity. The mechanisms by which certain polymers (e.g., CMC) reduce the viscosity of LCB suspensions is still under investigation, but the evidence suggests they hydrate rapidly and yet have a strong affinity to bind to LCB, thereby reducing interfiber friction [15] and preventing plug formation.

By assuming that the torque rheometer functions as a dual-Couette viscometer and developing a multi-speed, multi-cycle TR test protocol, it is possible to model LCB as a Bingham material and extract conventional measures of rheological properties such as yield stress and plastic viscosity. For the test on corn stover described here, the yield stress of raw corn stover (78.1 kPa) is considerably reduced by the addition of a small amount of CMC (31.5 kPa). The multi-speed, multi-cycle test protocol is also useful for calculating SME and SMI. We introduced these quantities as means to further characterize and compare batch mixing in a torque rheometer with continuous mixing in a twin-screw extruder. For instance, in the torque rheometer, a biomass sample is typically mixed for a much longer time period than during extrusion. Also, the conditions under which the LCB is mixed can be readily altered at any time (i.e., CMC added). In TR tests, both SME and SMI can be easily determined. However, in a twin-screw extruder, the mass flow is continuous and the instantaneous rate at which energy is transferred to the mass as it travels the length of the barrel is unknown. For this reason, SMI is calculated as the average rate at which energy is transferred to the biomass as it passes through the extruder. The significance of SMI, for both extrusion and torque rheometry, is that it defines the rate at which energy is transferred to a unit sample mass during mixing.

Figure 11 shows a comparison of SMI values obtained from TR and TSE trials on wood pulp. For compositions with CMC, the SMI values closely match. Interestingly, there is a sharp increase in the TSE-SMI value at 400 rpm. This may be due to the extremely short (30 s) residence time encountered at that speed, perhaps not enough time for the CMC to effectively reduce viscosity. Figure 11 also shows TR-SMI values calculated from the average power and peak power for the second TR test on OCC without CMC added (Fig. 5).



11. Specific mixing intensity values from TR and twin-screw extruders trials with OCC compositions at 25 wt% solids.

CONCLUSIONS

Biorefining of lignocellulosic biomass requires the integration of numerous chemical, thermal, and mechanical treatments. The cost of processing LCB can be reduced by increasing the concentration of insoluble solids in various process operations. However, increasing the solids concentration increases the apparent viscosity of the biomass, which makes mixing and conveying operations more challenging. An understanding of the rheological properties of high-solids LCB and how these properties can be controlled or altered in all stages of processing can facilitate the design and integration of biorefining operations.

We have successfully developed new techniques and testing protocols to measure the rheological properties of high-solids LCB using a modified TR. We have determined that the torque rheometer functions much like a twin-screw extruder and for quantifying rheological properties can be adequately represented as a dual-Couette viscometer. In addition, the flow behaviors of LCB are indicative of high yield stress (Bingham) fluids and can be characterized as such. We also observe that in the initial stages of mixing, torque values are extremely large and erratic. During this period, considerable particle-size reduction takes place with correspondingly large energy consumption. These peak torque events are an indication of the peak power requirements for process equipment and are of considerable concern to biomass processors. The addition of a rheological modifier, however, quickly and efficiently reduces biomass viscosity, resulting in reduced mixing power and energy requirements. Furthermore, the torque fluctuations are significantly reduced as homogeneous fiber paste forms. We have used a torque rheometer to evaluate the rheological behaviors of many different varieties of LCB, including wood pulp, corn stover, switchgrass, and model fiber systems [4, 12]. We have also investigated a variety of LCB pretreatments (including dilute-acid and enzymatic hydrolysis) [7, 16, 17], and LCB compositions containing rheological modifiers and other additives [3, 12].

Measuring the rheological behavior of high-solids LCB in a torque rheometer is important to better understand, characterize, and minimize the energy and mass transport problems associated with processing these materials. To demonstrate the value of these techniques, and to evaluate the viability of continuous processing, we conducted several extrusion trials on various LCB compositions. We found that high-solids LCB can be easily extruded if a rheological modifier (e.g., CMC) is used. We introduce new measures of SME and SMI to correlate torque rheometry and extrusion results. Although SME and SMI values are not considered to be material properties, we suspect that their calculation at peak and average power levels may be useful in predicting the extent of mixing severity that may be encountered in an extrusion process or similarly intense mixing device. **TJ**

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ABOUT THE AUTHORS

In the early 1990s, we were investigating alternative methods for recycling "high-solids" wastepaper and paper mill residuals, and extrusion seemed like an intriguing idea. We initially used a meat grinder to simulate an extruder, but failed miserably by plugging it on every attempt. Not to be deterred, we were next compelled to investigate torque rheometry as a means to characterize the rheological properties of concentrated fiber suspensions (and distract us from plugging another extruder).

The biggest challenge we face is to identify effective rheological modifiers that will quickly alter pulp viscosity and significantly lower mixing power and energy requirements, thus making extrusion possible. We have recently expanded our investigations to design a rheometer for measuring the in-situ rheological properties of high-solids biomass undergoing various treatments (acid-hydrolysis, enzymatic hydrolysis, etc.).

In addition, we continue to expand a simulation theory that predicts the consequences of fiber interac-



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tions in suspension. This work is providing new insight into the rheological behavior of high-solids fiber suspensions and the mechanisms that influence these behaviors.

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