Rheological Characterization of 1-Ethyl-3-Methylimidazolium Acetate and Lignocellulosic Biomass mixtures

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What is Rheology?

Rheology is the science of deformation and flow of matter, investigating the response of materials to applied stress or strain Rheological properties describe flow characteristics and textural behavior of substances and principles of rheology are used by industries for process design and quality control. Rheological characterization is based on a response to an applied load, force, or deformation and substances can thus be rheologically classified as, elastic (ideal solids), viscous (ideal fluids) or viscoelastic.

What is Viscoelasticity?

Typically, most materials lie between the ideal scenarios defining viscous fluids and elastic solids and are called viscoelastic materials.

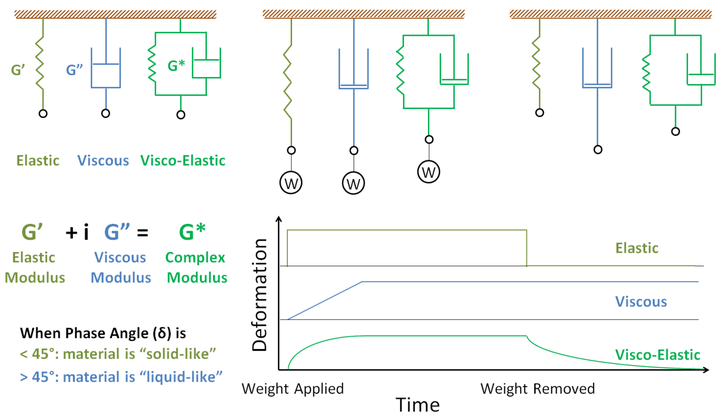


Figure 1. Illustration of a viscoelastic material at its expected behavior under stress

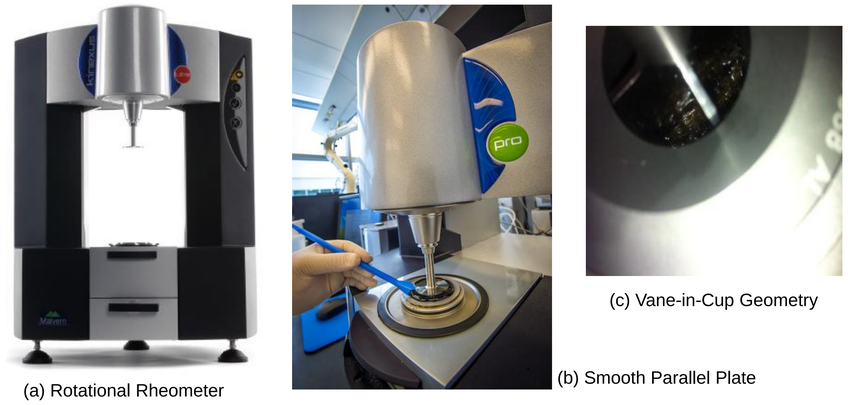
Can we measure Viscoelastic Properties?

Viscoelastic property data can be obtained by testing materials on sophisticated instruments called rheometers. Rheometers are generally classified as: rotational and extensional. Oscillatory rotational rheometers drive an attachment at a constant rate or stress and measure the resistance offered by the material. Some such attachments are parallel plate, cone and plate, mixer type, bob-in-cup (coquette and searle), and vane-in-cup geometries (see Figure 5). Mixtures of five biomass types mixtures and 1-ethyl-3-methylimidazolium Acetate (Ionic Liquid or IL) were pretreated in 10 L Mixing Parr Reactors (Moline, IL) at a working volume of 6 L (see Table 1) . Samples taken before and after pretreatment were analyzed for viscoelastic properties with smooth plate geometry.

Table 1. Pretreatment Reaction parameters of IL and Biomass Mixtures

Mixture type	Biomass type	Biomass loading (% w/w)	Reaction Temperature (°C)	Reaction Time (hour)
A	Sigma Aldrich Avicel	15	120	3
PS 1 (Temp 1)	Putnam Switchgrass	15	120	3
PS 2 (Temp 2)	Putnam Switchgrass	15	160	3
S	INL Switchgrass	10	140	1
E	INL Eucalyptus	10	140	1
MF	INL Mixed Feedstock	10	140	1

Oscillatory Stress Controlled Rheometer Measures Viscoelasticity of Ionic Liquid – Biomass Mixtures



(a) Rotational Rheometer (b) Smooth Parallel Plate (c) Vane-in-Cup Geometry

Figure 2. Malvern Kinexus Rheometer (Boston, MA) with attachments

Material Viscosities and Solid Liquid Characteristics

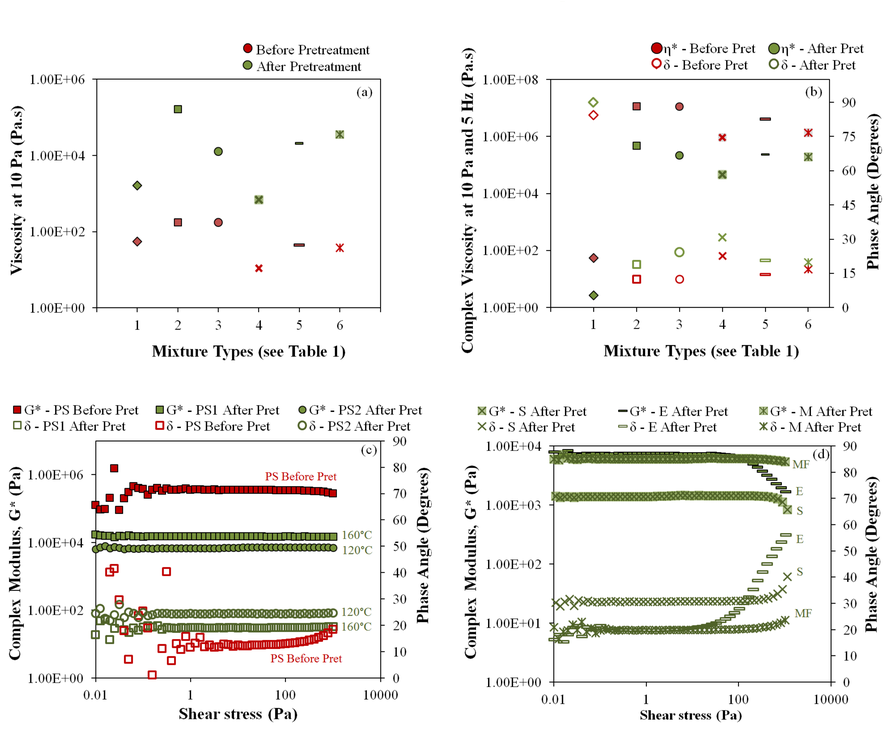


Figure 3. (a) Shear Viscosities and (b) Complex Viscosities and Phase Angles of mixtures measured at 25° C. Complex Modulus and Phase Angles over three orders of shear stress @ 5 Hz in the linear viscoelastic region of (c) Putnam Switchgrass and (d) INL feedstocks.

IL Pretreatment and Removal Reduces Biomass Rigidity

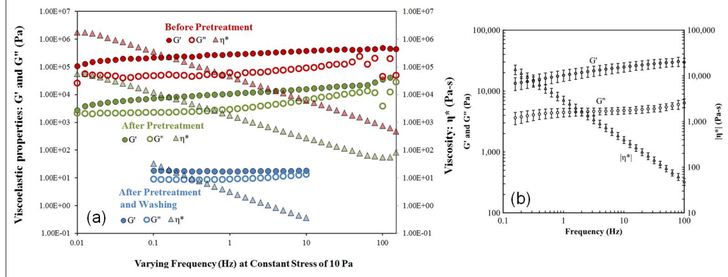


Figure 4. Comparison of rheological properties of
(a) IL pretreated Switchgrass pretreated at 160° C (Mixture C) with
(b) Dilute acid treated corn stover (17% w/w) from Stickel et al. (2009)

Real-Time Rheological Data Provides Composition Related Information

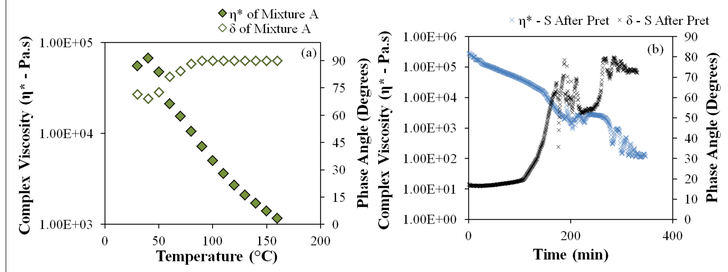


Figure 5. Real-Time Complex Viscosity and Phase Angle during
(a) Pretreatment of Avicel with Increasing Temperature to 160° C in 30 minutes and (b) Enzymatic Hydrolysis of IL Pretreated and Washed Switchgrass with Novozymes Ctec2 at 54 mg enzyme/ g glucan

Implications of these Studies include:

(1)Characterizing biomass to obtain specifications for scale-up biomass mixing and handling equipment, such as pumps, feeders, hoppers, motors, etc.
(2)Identifying the stress required to change the material from "solid-like" to "liquid-like" form for better mixing during pretreatment and saccharification and better handling
(3)Understanding the influence of pretreatment temperature and feedstock variation
(4)Utilizing real-time data to correlate to composition and Degree of Polymerization
(5)In other applications including Carbon-Fiber spinning

Acknowledgement and Reference

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1.J. J. Stickel, et al. Rheology measurements of a biomass slurry: an inter-laboratory study *Rheologica Acta* 2009, 48 (9), 1005-1015.