ELECTRONIC SUPPLEMENTARY INFORMATION

Composite Fibers Spun directly from Solutions of Raw Lignocellulosic Biomass Dissolved in Ionic Liquids

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Materials

The wood samples were received from Seaman Timber Co. (Montevallo, AL) as shavings. Bagasse was donated by the Sugar Cane Growers Cooperative of Florida (Belle Glade, Florida). The biomass samples were ground into powder using a lab mill and sieved to particles less than 0.125 mm (wood) or 0.25 mm (bagasse), then dried overnight in an oven at 90 °C. The IL, 1-ethyl-3-methylimidazolium acetate ([C_2mim]OAc, ≥95%) was obtained from Iolitec (Ionic Liquids Technologies GmbH, Heilbronn, Germany). Microcrystalline cellulose (MCC) was purchased from Sigma-Aldrich Inc. (Milwaukee, WI), and Indulin AT (lignin from the kraft pulping process) from MeadWestvaco Corporation (Glen Allen, VA). All the other reagents were obtained from Sigma-Aldrich (St. Louis, MO) and used as received.

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Preparation of biomass composite fibers

The twelve solutions used to wet spin fibers were prepared as follows. Wood: 0.5 g of wood powder (pine, oak, or pretreated pine with particle size < 0.125 mm) was added to 10 g [C_2mim]OAc, then the mixture was placed into an oil bath and heated at 175 ^oC for 30 min with magnetic stirring. For oak, a lower concentration (0.33 g oak in 10 g [C_2mim]OAc) was used because the 0.5 g oak/10 g IL solution was too viscous, resulting in a considerable amount of undissolved particles in the spinning dope. Bagasse: 0.5 g bagasse (< 0.25 mm) was dissolved in 10 g [C_2mim]OAc either at 185 ^oC for 10 min or 110 ^oC for 16 h. Carbohydrate Rich Material (CRM): 0.5 g of the CRM obtained from the dissolution of pine at 175 ^oC for 30 min and regenerated in acetone/water was dissolved in 10 g of [C_2mim]OAc at 90 ^oC for 2 h. Standards: 0.3 g of cellulose pulp (DP = 1056) or 0.5 g of a 44/24/32 (wt%, the same weight ratio as in pine) mixture of pulp (DP = 1056)/xylan (for hemicellulose)/Indulin AT (for lignin) was dissolved in 10 g [C_2mim]OAc at 90 ^oC for 30 min.

Each biomass/IL solution was centrifuged (Clay Adams® Brand DYNAC centrifuge, Bohemia, NY) at 100 x g for 3 min after heating to remove any suspended particles, then the solutions were transferred to a 10 mL syringe and degassed upright in the vacuum oven at 80 ^oC for 2 h. The fibers were prepared using the same extrusion set up as described in our previous report.[1] Briefly, the syringe was attached to the syringe pump with a temperature controller at 70 ^oC. The fibers were extruded at 0.5 mL/min into a 0.6 m long DI water bath after a 5 cm air gap. The fibers were fed through the first two steps of the godet (recorded voltage: 4.5 V, 15 rpm) and wound onto a spool (2.9 V, 1.3 m/s). The fibers were further washed by soaking the reel in a 400 mL beaker filled with DI water overnight and then air dried.

Characterization

Tensile strength of the fibers. The tensile testing for the fibers was carried out as described in our previous report[1] using an MTS Q-Test 25 machine (Eden Prairie, MN). The uniformity of the samples was screened by checking the diameters of the fibers from ten samples from each spool. At least three 10 cm long samples were tested from each category. The average diameter of each sample was calculated from 5 measurements at different sections of the fiber, and a gauge length of 3 inches was used for the measurements. The selected fibers were attached to a pneumatic grip and subjected to a load cell of 5 lb. The cross head speed was 0.05 inch/min and the stress and strain data were obtained using a data acquisition system.

SEM imaging. SEM images of the composite fibers were taken at 300× magnification using a Philips XL30 SEM instrument (Eindhoven, The Netherlands) operated at 10 kV for the accelerating voltage. The fibers were sputter-coated with gold before imaging to avoid charging and degradation of the specimen.

Cellulose content determination. The cellulose contents of both the original/treated biomass and the prepared fibers were quantified using the method described in previous report.[2]

References

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[2] W. Li, N. Sun, B. Stoner, X. Jiang, X. Lu, and R. D. Rogers, *Green Chem.*, 2011, submitted.