Electronic Supporting Information

Preparation and comparison of bulk and membrane gels based on Kraft- and ionic-liquid-isolated lignins

Xiaoping Shen,[a,b] Paula Berton,[a,c] Julia L. Shamshina,[d] Robin D. Rogers[a,c,1]

[a] Department of Chemistry, The University of Alabama, Tuscaloosa, AL 35487, USA

[b] Key Laboratory of Bio-based Material Science and Technology (Ministry of Education), Northeast Forestry University, 26 Hexing Road, Harbin 150040, China

[c] Department of Chemistry, McGill University, 801 Sherbrooke St. West, Montreal, QC H3A 0B8, Canada

[d] 525 Solutions, Inc., 720 2nd Street, Tuscaloosa, AL 35401, USA

[1]Corresponding author; E-mail: robin.rogers@mcgill.ca

EXPERIMENTAL

Fourier-Transform Infra-Red Spectroscopy (FT-IR). FT-IR spectra of ETPEG, lignin, and lignin/ETPEG hydrogels (air-dried) were recorded in the range of 500–4000 cm^{-1} using a Bruker Alpha FT-IR instrument (Bruker Optics Inc., Billerica, MA, USA) with an attenuated total reflectance (ATR) sampler.

Powder X-Ray Diffraction (PXRD). PXRD analysis of lignin powder was performed at room temperature using a Rigaku D/MAX-2BX horizontal X-ray diffractometer (Rigaku Co., Spring, TX, USA) equipped with Cu-Kα radiation (λ= 1.5418 Å). The samples were scanned within 5–30° (2θ) in continuous mode with a step size of 0.02° and step time of 3 degrees s^{-1}.

Synthesis of epoxide terminated PEG (ETPEG). ETPEG was synthesized by a slightly modified, reported procedure.[1] NaOH (1.80 g; 45 mmol) and ECH (4.16 g; 45 mmol) were transferred into a round-bottom flask equipped with a Teflon-coated magnetic stirring bar, under magnetic stirring. PEG (15 mmol PEG_{400} or PEG_{1000}: 6.0 or 15.0 g, respectively) was then added, and the mixture was stirred at 40 °C for 2 h. After the reaction time, the resultant mixture was dissolved in acetonitrile, followed by centrifugation to remove excess NaOH and the byproduct NaCl. Acetonitrile was then evaporated under reduced pressure using a rotary evaporator at 50 °C. The resulting products were coded as $ETPEG_{400}$ and $EFPEG_{1000}$, respectively, and used without further purification. The degree of substitution (DS; substituting –OH with $–OCH_2CHCH_2O$) of the product was calculated using eqn. (S1):

$$DS\ (\%) = \frac{\Delta m}{n_{PEG} \times 2 \times (73 - 17)} \times 100\% \qquad \text{(S1)}$$

where Δm (g) is the mass gain of ETPEG relative to the original PEG added, n_{PEG} (mol) is the mol number of original PEG (considered to be equal to the mole number of ETPEG), 17 and 73 g mol^{-1} are the molecular weight of –OH and $–OCH_2CHCH_2O–$ groups, respectively. Both $ETPEG_{400}$ and $ETPEG_{1000}$ had a high DS, calculated to be 80(3)% and 75(2)%, respectively.

RESULTS AND DISCUSSION

PXRD of lignin powders

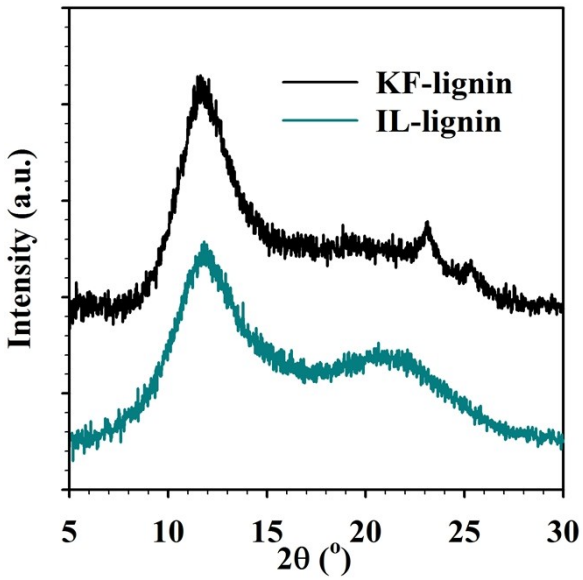


Figure S1. PXRD diffractograms of KF- and IL-lignin powder.

WVT determination

Figure S2. Water vapor transmittance (WVT) test set up.

Calibration curve of DPPH/methanol solutions

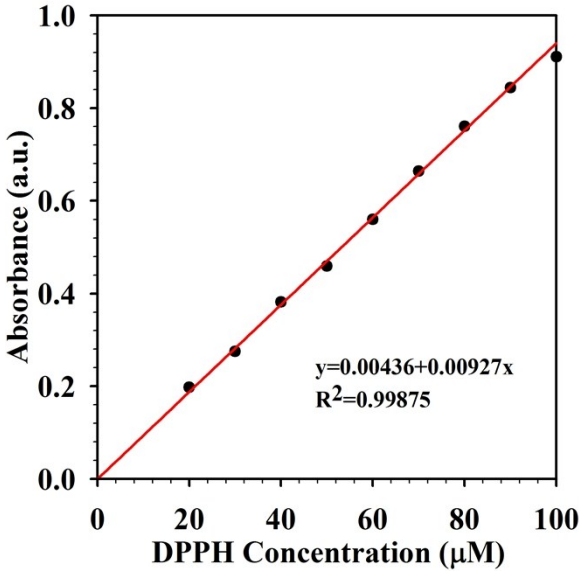


Figure S3. Calibration curve of DPPH/methanol solutions at 23 °C.

Shrinkage of bulk gels using various drying methods

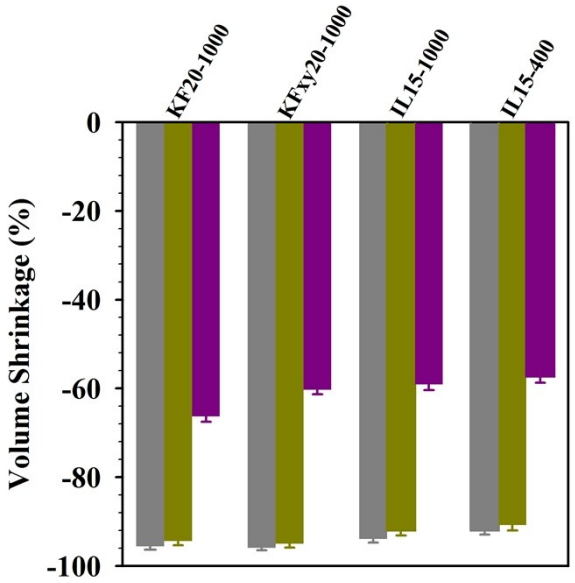


Fig. S4. Shrinkage of lignin/ETPEG bulk hydrogels after various drying methods (■) air-drying, (■) $ScCO_2$-drying, (■) freeze-drying.

SEM of bulk gels after air-drying

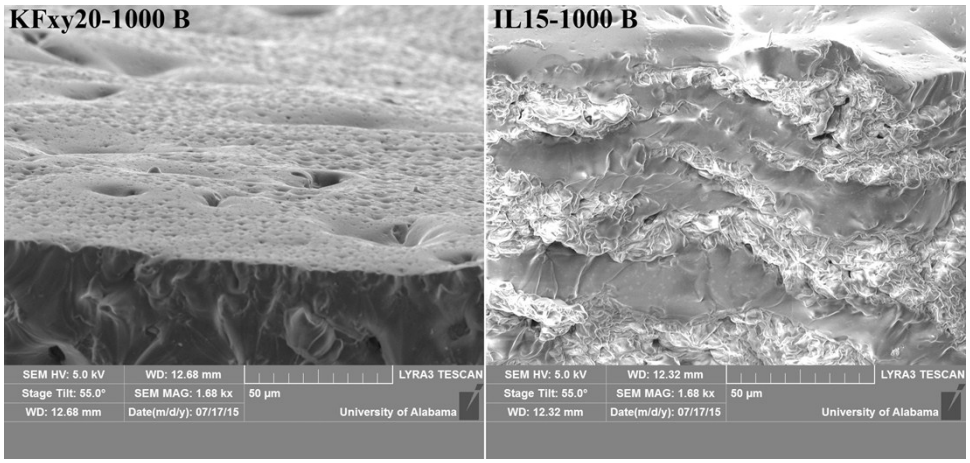
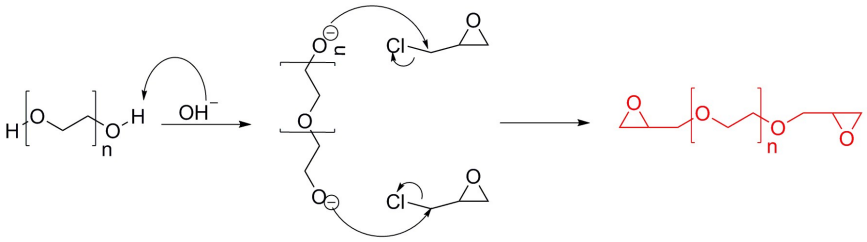


Figure S5. SEM images of lignin/ETPEG bulk (B) hydrogels after air-drying (Left: KFxy20-1000 B; Right: IL 15-1000 B).

Characterization of ETPEG



Scheme S1. Preparation reaction of epoxy-terminated poly(ethylene glycol) (ETPEG), adapted from Ref. 1.

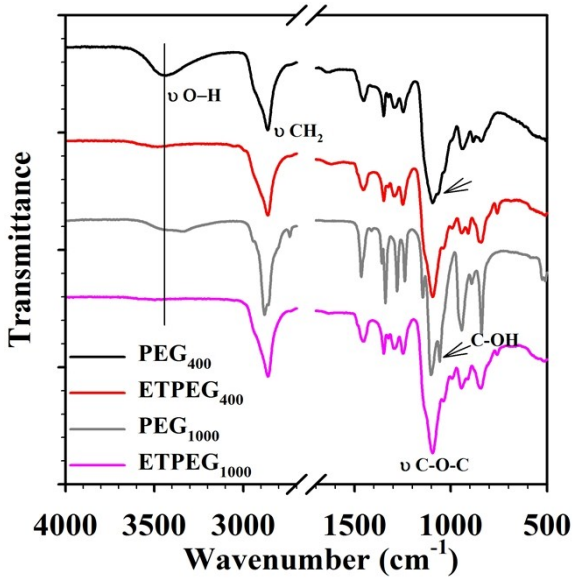


Figure S6. FT-IR spectra of ETPEG.

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FT-IR of lignin/ETPEG hydrogels

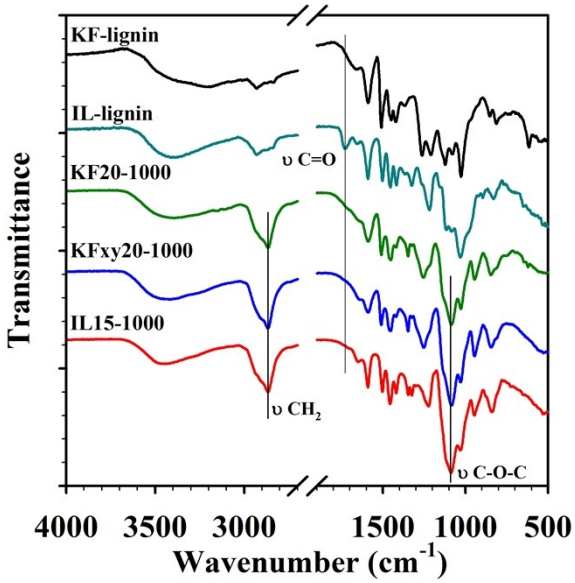

Figure S7. FT-IR spectra of lignin/ETPEG hydrogels.

References

1. K. Y. Cho, C. H. Kim, J. W. Lee and J. K. Park, *Macromol. Rapid Commun.*, 1999, **20**, 598-601.