Synthesis and Characterization of Lignin-Based Carbon Materials with Tunable Microstructure

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Results and Discussion

Lignin-based carbons can be used as a low-cost alternative to graphite and petroleum-based carbons enabling the production of sustainable, functional carbon materials for various applications. The microstructure development of these carbons can be controlled through chemical modification of the lignin precursor and choice of carbonization parameters. In this work, microstructured carbon materials are synthesized from lignin using a combination of chemical modification and carbon fiber processing techniques. Lignin is modified by incorporating different ester groups which results in a precursor highly compatible with melt processing using the fiber extrusion technique and conversion into microstructured carbons by oxidative stabilization and subsequent carbonization. Furthermore, the impact of esterifications on precursor chemistry and carbonizations is investigated. A nuclear magnetic resonance study of modified lignins shows characteristic spectral changes as a result of esterifications. Ultrahigh resolution Fourier transform ion cyclotron resonance mass spectrometry shows the modification process does not affect the polymeric character of the lignin backbone. Esterifications result in moderate shifts in O:C and H : C ratios. Thermogravimetric analysis of lignins reveals distinct differences in mass loss trends during oxidations and carbonizations.

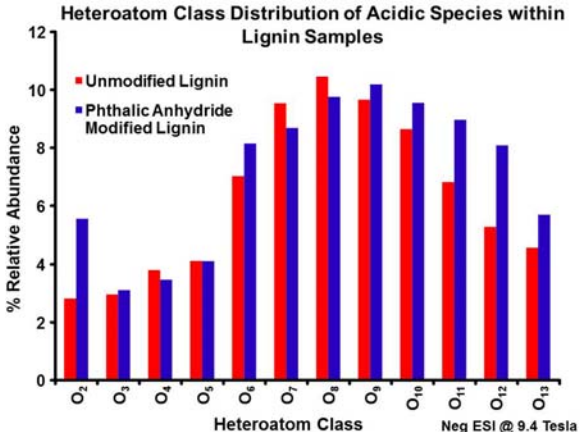

Figure 1. Heteroatom class distribution identified at >1% relative abundance in either lignin of phthalic anhydride-modified lignin derived from negative-ion ESI FT-ICR MS. The heteroatom class with the highest relative abundance detected by negative-ion ESI FT-ICR MS in both samples contains eight oxygen atoms (O_8) per molecule, with both samples containing between two and thirteen oxygen atoms. While NMR is an excellent tool for probing specific chemical functionalities, the complex branched nature of lignin biopolymers limits the use of NMR for the characterization of changes in the underlying polymer structure. Fourier transform ion cyclotron resonance mass spectrometry (FT-ICR MS) offers ultrahigh mass resolution and accuracy, which enables unique assignment of elemental compositions for thousands of fragments obtained from a single lignin sample. The high sensitivity of FT-ICR MS can reveal distinct changes in the elemental composition of lignin fragments as a result of chemical modification and its impact on the polymer characteristics, and capture compositional changes that occur to lignin after phthalic-anhydride modification.

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References

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