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Vicky Su

Washington University in St. Louis

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SYNTHESIS OF LIGNIN MODEL POLYMER

Vicky Su

Mentor: Marcus Foston

In the project of synthesis lignin model polymer, precursors of lignin model polymers and co-polymers containing various monomers and molecular weights are made in order to help monitor and simulate the activity of lignin. Lignin is the only renewable source of aromatics, which is currently treated as waste. Therefore, it's more economically meaningful for second generation to convert lignin. To more efficiently use lignin and to convert it into value-added compounds is economically meaningful for second generation biorefinery. However, the high structural complexity and recalcitrance of lignin makes it spectacularly hard to understand its depolymerization process. In this study, various types of lignin model polymers with only β -O-4 with different molecular weight, topology, and repeating units were synthesized for understanding how the size and topology affect lignin depolymerization in future studies. The synthesis of the model compounds polymer involves using an S₂N reaction adapted from the work of Takao Kishimoto, which includes recrystallization, bromination, column filtration, polymerization, and reduction. 4-hydroxy-3-methoxy-acetophenone, and 4-hydroxyacetophenone were used as starting precursors to simulate the G and H units in lignin, which are the basic components of lignin in nature. The products from each step were tested by Thin-Layer Chromatography and/or Nuclear Magnetic Resonance, and yields were recorded for their purity over the duration of the research period. The majority of my time was devoted to work focusing on repeating the bromination step to generate monomer in large scale and increase its purity through the multiple recrystallizations of the brominated products. It is crucial to build a library of the brominated monomers for further research, and it is necessary to ensure the starting material of the polymerization is pure in order to obtain high yield of polymer in the polymerization step of the brominated products as well. While trying to balance the overall percent yield and the purity of the brominated products, adjustments to the bromination period and filtration methods were made. The optimal percent yield for H brominated monomer is 91.3% after two rounds of recrystallization. Overall, a library of 11.56g of pure brominated H monomer, 6.03g of pure brominated G monomer, and a model of cross-linked H polymer (with 1,3,5-trihydroxybenzene in 50:1 ratio) were made and tested during the research period, and these products will be applied in the future study.