CHARACTERIZATION OF ORGANOSOLV SWITCHGRASS BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY/MULTIPLE STAGE TANDEM MASS SPECTROMETRY USING HYDROXIDE-DOPED ELECTROSPRAY IONIZATION

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Many research efforts focus on the extraction, degradation and catalytic transformation of lignin, hemicellulose and cellulose. Unfortunately, these processes result in the production of very complex mixtures. Testing the usefulness of these processes requires analytical methods that can be used to rapidly characterize the complex mixtures produced. In this study, high-performance liquid chromatography/multiple stage tandem mass spectrometry has been implemented for organosolv mixtures of switchgrass. In this study, HPLC coupled with negative-ion mode MS^n analysis for the characterization of lignin degradation products is demonstrated. This approach hinges on the use of a hydroxide doped electrospray ionization method that ionizes all the mixture components to only yield one ion/analyte with no fragmentation. Analytes eluting from the HPLC were detected by UV light absorbance by using a PDA detector and by detecting all the ions generated upon ESI in the mass spectrometer. In addition to the acquisition of mass spectra for the compounds eluting from HPLC (MS^1), ions formed from these compounds were subjected to isolation and CAD experiments (up to MS^3) until no further fragmentation products were observed. MS^n was utilized to provide structural information for the components of a real degradation mixture on a chromatographic time scale. On the same time scale, elemental compositions were acquired for all analytes by transferring ions to a high-resolution instrument. This methodology significantly improves the ability to analyze complex product mixtures that result from degraded biomass. A switchgrass degradation product mixture was analyzed and molecular structures were proposed for its main components. The mixture contains primarily coumaric and ferulic esters. These unextracted components prevent the analysis of the lignin components in the mixture due to their high abundance. Further modification to the extraction procedure would need to be made before complete analysis of the lignin components can be accomplished.