Supercritical Fluid Chromatography of Lignin-Derived Phenols from Alkaline CuO Oxidation

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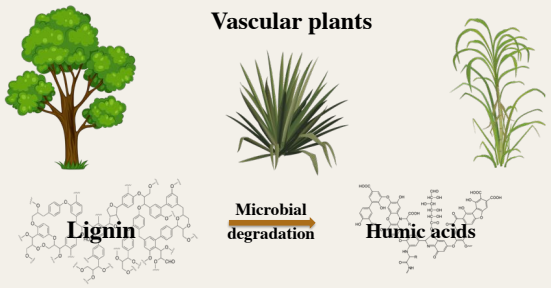
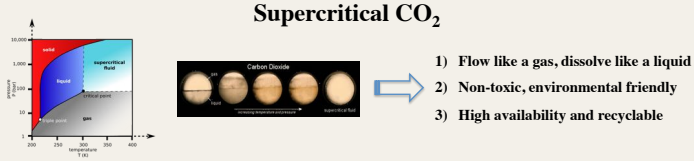




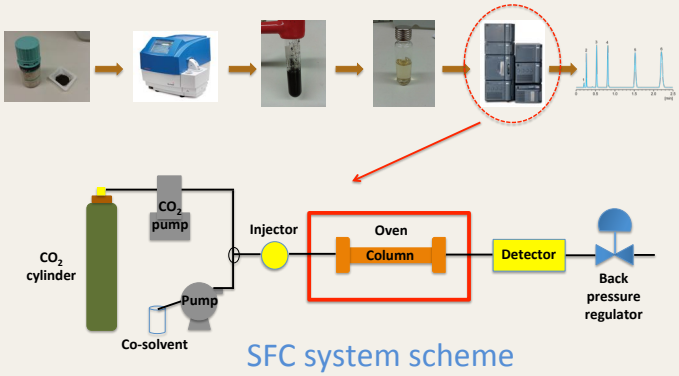
Introduction

Major lignin-derived phenols produced from Cupric Oxide (CuO) oxidation are analyzed to trace back the type of original plant tissues in geoscience. Methods reported are gas chromatography (GC), high-performance liquid chromatography (HPLC) and capillary electrophoresis (CE), which suffer either from a need for derivatization, long analysis time or poor precision of identification.

This work reports a simple, robust and fast supercritical fluid chromatography (SFC) method for separation and quantification of lignin-derived phenols from CuO oxidation.

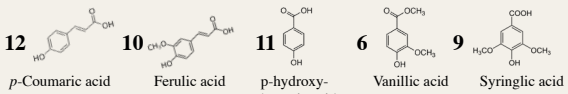
Experimental

Humic acid sample was added to a NaOH solution together with $Fe(NH_4)_2(SO_4)_3 \cdot 6H_2O$ and CuO. Oxidation was carried out in a Biotage Initiator Microwave Reactor. After acidification, extraction, solvent evaporation and re-dissolution, the samples were analyzed on a Waters Ultra Performance Convergence Chromatography System (UPC^2) with a Diode Array Detector.

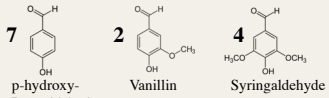


SFC system scheme

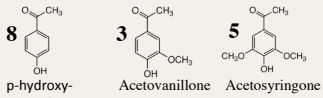
Acids

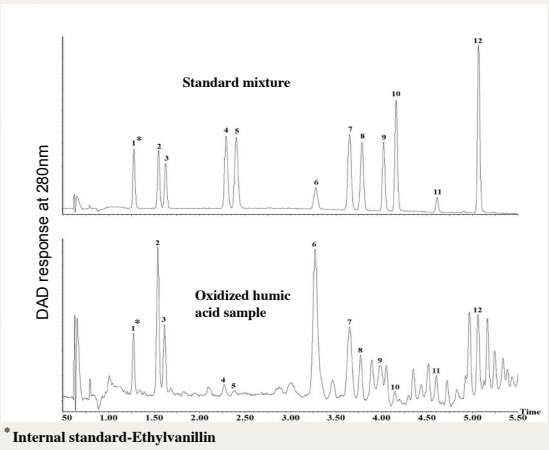


Aldehydes



Ketones





*Internal standard-Ethylvanillin

Results and discussion

It can be clearly observed that the 12 phenolic compounds (11 lignin phenols and ethyl vanillin as internal standard) were well separated in 6 minutes with all peaks being highly symmetrical. The resolution factors of each pair of adjacent two peaks varied between 1.38 and 10.93. The limit of detection (LOD) for each phenol ranged from 0.5 to 2.5 µM and limit of quantification (LOQ) 2.0 to 5 µM.

The applicability of the method developed was tested by analysis of phenol content in a humic acid sample. The lignin-derived phenols were identified with both elution times and characteristics of absorption spectra from external standard runs. No interfering peaks were found that co-elute with the lignin phenols of interest.

Conclusions

The SFC method described gave rapid and clear separation of the 11 major lignin-derived phenols from cupric oxide oxidation with good selectivity and resolution. Application of this method in the analysis of humic acid sample proved its great suitability and potential in the analysis of complex environmental samples.

Acknowledgement

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