SIZE EXCLUSION CHROMATOGRAPHY OF LIGNIN IN N-N-DIMETHYLFORMAMIDE/LITHIUM BROMIDE

Carolina Cateto (1,2,3) • Filomena Barreiro (1) • Naceur Belgacem (3) • Alírio Rodrigues (2)

(1) LSRE - Bragança Polytechnic Institute (2) LSRE - School of Engineering - University of Porto (3) Materiaux Polymères - EFPG-INPG

OBJECTIVES

To inspect the effect of some working conditions: temperature, LiBr concentration and sample preparation details (lignin concentration and dissolution time) on the obtained chromatographic profile.

LIGNIN SAMPLES

ALCELL

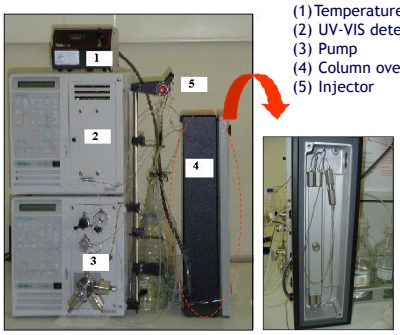
INDULIN-AT

Table 1. Properties of the technical lignins used in the SEC study

Lignin sample	Total OH (mmol/g)	Phenolic OH (mmol/g)	COOH (mmol/g)	Ash content (% w/w)
Alcell	5.26	3.81	0.23	0.05
Indulin AT	6.99	3.95	0.39	3.06

Indulin AT is a softwood lignin obtained by the kraft process (MeadWestvaco)
Alcell is a hardwood lignin obtained by an organosolv process (Repap Enterprises)

SEC ANALYSIS

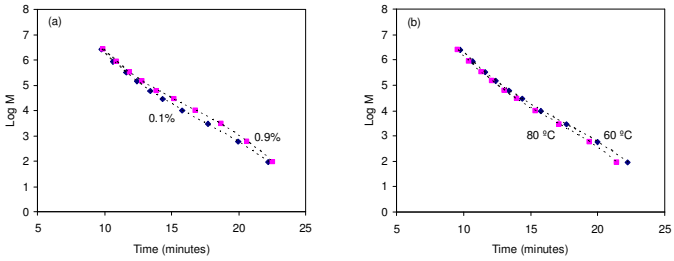


(1) Temperature controller
(2) UV-VIS detector
(3) Pump
(4) Column oven
(5) Injector

SEC measurements were performed on VARIAN Prostar 240 liquid chromatograph equipped with a UV-VIS detector working at 280 nm. Two PLgel 5µm MIXED-D columns (300x7.5 mm) and a pre-column in series were used.

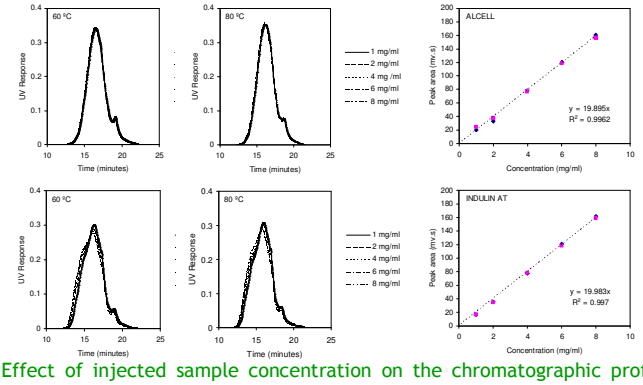
Typical analysis conditions: Sample concentration: 4 mg/ml; Dissolution time: 24 hours; Flow-rate: 1 ml/min; Injection volume: 20 µl.
Studied variables: Temperature (60 and 80 ºC); LiBr concentration (0.1 and 0.9 %, w/w); Sample concentration and dissolution time.

CALIBRATION WITH PS STANDARDS



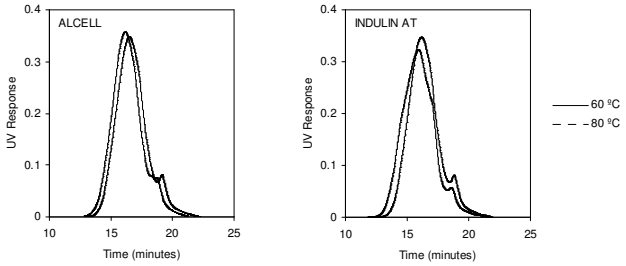
Effect of LiBr concentration at 60 ºC and eluent temperature using 0.1 % LiBr.

SAMPLE CONCENTRATION EFFECTS

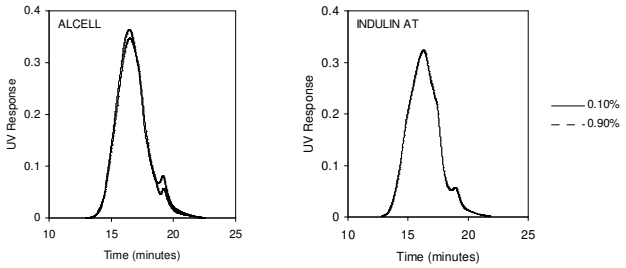


Effect of injected sample concentration on the chromatographic profile for 60 and 80 ºC (LiBr concentration of 0.1%) and peak area as a function of concentration. (Squares: 60 ºC; Lozenges 80 ºC).

LIGNIN ANALYSIS



Effect of temperature on the chromatoghrafic profile of Alcell and Indulin AT (LiBr concentration of 0.1%).

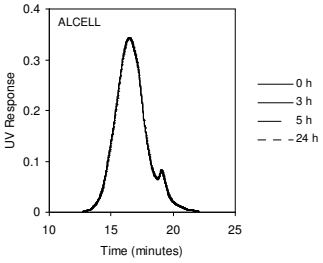


Effect of LiBr on the chromatoghrafic profile of Alcell and Indulin AT (Temperature of 60 ºC).

CONCLUSIONS

1. Calibration with PS standards:
•For a fixed temperature, the calibration trace shifts towards lower hydrodynamic volume as the LiBr concentration increases.
•For a fixed LiBr concentration, the calibration trace shifts towards higher hydrodynamic volume as the temperature increases.
2. Sample concentration effects:
•For Alcell the chromatographic profile did not change with sample concentration, whereas for Indulin AT the opposite was observed.
•For both lignins a linear dependence of the UV response as a function of temperature was observed. Moreover, the achieved response factor is approximately the same (19.90 for Alcell and 19.98 for Indulin AT).
3. Dissolution time:
•For Alcell the used dissolution time prior to analysis did not influence the chromatographic profile. This lignin could be inject just after sample preparation.
4. Lignin analysis:
•For Alcell the chromatographic profile as a function of temperature and ionic strength can be analysed considering only hydrodynamic volume contributions.
•For Indulin AT some differences where found that pointed out for the presence of association phenomena.
•Alcell lignin in DMF/LiBr can be used as a suitable model system to perform molecular weight investigations, particularly those dealing with comparative studies using different characterization techniques.

DISSOLUTION TIME



Dissolution time prior to sample analysis (60 ºC, 0.1% LiBr)

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