

MOLECULAR WEIGHT DETERMINATION BLEACH EFFLUENTS

Effluent molecular weights were measured by an aqueous gel permeation chromatography method developed by Schwantes and McDonough (i). The method passes the effluent through a G-50 and G-15 Sephadex columns using a 0.1 M LiCl eluent. The discharge of the columns flows into a total organic carbon (TOC) detector, which measures on a frequency of approximately 1 sample every 2.3 min. Low flow rates (0.65 mL/min) allow for increased resolution due to the slow periodicity of the TOC detector. The column was calibrated using polyethylene glycol standards and methanol giving a calibration range for molecular weights of 32 to 19,700 atomic mass units (amu). Tabulation of the amount of the original sample's TOC recovered within the calibration range gives an indication of holdup in the column. Number average molecular weight (MW_N) and weighted average molecular weight (MW_W) were calculated using equations 1 and 2 (ii).



$$MW_W = \sum w_i M_i \quad (1)$$

$$MW_N = \sum x_i M_i \quad (2)$$

where: w_i is the weight fraction of fraction i ; x_i is the mole fraction of fraction i
 M_i is the molar mass of fraction i

Isolated effluent lignins were dissolved in a 0.1N NaOH at a concentration of ~2000 ppm. The solutions were analyzed for molecular weights in a similar manner to the effluent samples using the GPC/TOC method. The GPC/TOC LC tests took approximately 6 hr for a sample to traverse the columns. After a sample was run, the columns were run an additional 6 hr to flush out any organic matter that was held up in the column. Additionally, the amount of TOC that passed through the column during the test was quantified. This was accomplished by measuring the original samples' TOC and comparing it to the summation of the TOC measured. The recovered TOC ranged from 79.6 to 86.5%.

A repeatability study was performed on the effluent molecular weight procedure by analyzing four independent DE effluent samples and calculating a LSD value. The data shown in Table indicate 95% LSD values of 621 and 718 amu for the MW_N and MW_W averages, respectively.

Table 1. Repeatability Study Molecular Weight Averages

| Stage | MW _N (amu) | MW _W (amu) | Polydispersity |
|--------------------|-----------------------|-----------------------|----------------|
| DE - 1 | 3353 | 7248 | 2.161 |
| DE - 2 | 3230 | 6994 | 2.165 |
| DE - 3 | 3441 | 7258 | 2.166 |
| DE - 4 | 3142 | 6984 | 2.169 |
| Average | 3292 | 7121 | 2.170 |
| Standard Deviation | 132 | 153 | 0.003 |
| LSD @ 95% | 621 | 718 | 0.014 |

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- i. Schwantes, T.A.; McDonough, T.J. *Development of a Method of Aqueous Gel Permeation Chromatography for the Determination of Molecular Weight Distributions of Bleaching Effluents. 1994 International Environmental Conference. TAPPI Press, Atlanta, 1994. pp.683-695.*
- ii. *Shaw, D.J. The Colloid State. Introduction to Colloid and Surface Chemistry 4th Ed. Butterworth Heinemann, Oxford, 1992. pp. 1-10.*