KRAFT PULP REACTIVITY ASSESSMENT USING COLD ALKALINE DISSOLUTION IN TEMPERATURE-DEPENDENT CROSS-POLARIZED LIGHT MICROSCOPY AND DYNAMIC SCANNING CALORIMETRY

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OBJECTIVE

Regenerated cellulose is produced by dissolving cellulose in a suitable solvent and precipitating in an antisolvent. Due to complex cellulose architecture caused by intra- and intermolecular cellulose hydrogen bonds, cellulose dissolves poorly in common solvents.

Kraft process is the dominant method for chemical pulp manufacturing. Kraft pulp fibres require pre-treatment to facilitate their dissolution, such as acid hydrolysis. The purpose of this study was to examine kraft pulp reactivity in dissolution using two methods: temperature-controlled cross-polarized light microscopy (CPLM) and dynamic scanning calorimetry (DSC).

EXPERIMENTAL PROCEDURE

Bleached softwood kraft pulp was acid hydrolysed to various degrees of polymerization (DP) in 0.5 M H_2SO_4. The DP was estimated from CED intrinsic viscosity values. A cellulose dissolution solvent was prepared by dissolving NaOH and ZnO in deionized water.

A small kraft fibre amount and a small droplet of NaOH/ZnO solvent was placed on the sample slide, and using a temperature-controlled stage, the sample temperature was dropped from 25 °C to -15°C at 10 °C/min rate. A cross-polarized light video was captured, and the video was converted frame-by-frame to mean relative luminance values corresponding with the dissolution.

Similar experiment was conducted combining kraft pulp with the solvent in a DSC pan. The pan was sealed, and a DSC program was run, dropping sample temperature at 10 °C/min rate. Exothermic peaks were detected, and dissolution enthalpy was calculated by integrating the peak area

MAIN RESULTS

Acid hydrolysis decreased cellulose molecular weight, enabling cellulose to dissolve. CPLM revealed four different sample dissolution modes: no dissolution, heterogeneous swelling via ballooning, complete dissolution via homogeneous swelling, and direct complete dissolution. DSC heat of dissolution energies correlated with CPLM results: more complete dissolution exhibited a larger energy output. The findings enable calculation of cellulose dissolution kinetics, including activation energy

CONCLUSIONS

The cellulose dissolution in an aqueous NaOH solution takes place within a specific temperature range. By controlling the temperature of the cellulose-solvent system, the dissolution reaction can be shown in real-time. The dissolution of cellulose leads to a loss of crystallinity, resulting in the disappearance of birefringence under cross-polarized light, and exothermic peaks in a DSC curve. Hydrolysed kraft pulp dissolution rate was shown to correlate inversely with sample DP: lower DP cellulose dissolves more completely at a faster rate

Keywords: : cellulose, dissolution, DSC, microscopy