Characterization and optimization of the glyoxalation of a methanol-fractionated alkali lignin using response surface methodology

ABSTRACT

The glyoxalation of a methanol-fractionated alkali lignin was executed at 60 °C for 8 h with different amounts of glyoxal (40% in water) and 30% NaOH. The weights of the lignin and water were fixed at 10.0 and 15.0 g, respectively. The gel permeation chromatography (GPC) results indicated that depolymerization of lignin molecules occurred during the glyoxalation process. However, a higher polydispersity index (Mw/Mn) of all glyoxalated lignins compared to the unmodified lignin (ML) showed that lignin polymers with a variety of chain lengths were generated through the crosslinking and through the repolymerization of lignin molecules via methylene (CH2) bridges and new, strong C-C bonds after the condensation reaction. This was confirmed by thermogravimetry analysis (TGA). Optimum amounts of glyoxal and NaOH to be used in the glyoxalation process were ascertained by quantifying the intensity of relative absorbance for the CH2 bands obtained from FT-IR spectra and by using response surface methodology (RSM) and central composite design (CCD), which facilitated the development of a lignin with appropriate reactivity for wood adhesive formulation. The experimental values were in good agreement with the predicted ones, and the model was highly significant, with a coefficient of determination of 0.9164. The intensity of the relative absorbance for the CH2 band of 0.42 was obtained when the optimum amounts of glyoxal and NaOH, i.e., 0.222 and 0.353, respectively, were used in the glyoxalation process.

Keyword: Lignin; Glyoxalation; Response surface methodology; Glyoxal; Wood adhesive