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Isolation and characterization of hemicelluloses extracted by hydrothermal pretreatment

Ming-Guo Ma [a,*], Ning Jia [a], Jie-Fang Zhu [b], Shu-Ming Li [a], Feng Peng [a], Run-Cang Sun [a,c]

[a] Institute of Biomass Chemistry and Technology, College of Materials Science and Technology, Beijing Forestry University, Beijing 100083, PR China
[b] Department of Chemistry, the Ångström Laboratory, Uppsala University, Uppsala 75121, Sweden
[c] State Key Laboratory of Pulp and Paper Engineering, South China University of Technology, Guangzhou 510640, PR China

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ABSTRACT

The dewaxed sample from Triploid of *Populus tomentosa* Carr. was extracted by using organic alkaline solvent (Dimethylformamide, DMF) via hydrothermal pretreatment. Neutral sugar compositions and molecular weight analysis demonstrated that the hemicellulosic fractions with a higher Uro/Xyl ratio, namely the more branched hemicelluloses, had higher molecular weights. Interestingly, these results were different from the previous report, in which the ratio of Uro/Xyl in the water-soluble hemicellulosic fraction was more than that of the alkali-soluble hemicellulosic fraction. Spectroscopy (FTIR, ^{1}H NMR, ^{13}C NMR, and HSQC) analysis indicated that the hemicellulosic fractions were mainly composed of (1 → 4)-linked α-D-glucan from starch and (1 → 4)-linked β-D-xylan attached with minor amounts of branched sugars from hemicelluloses. In addition, thermal analysis implied that linear hemicelluloses showed more thermal stability than the branched ones during pyrolysis.

1. Introduction

Lignocelluloses has been receiving considerable attention as low cost and renewable feedstocks for producing biofuels, bio-based chemicals, and high value-added biomaterials to meet global energy and chemical needs (Himmel et al., 2007). The major components of lignocelluloses are the polysaccharides (cellulose and hemicelluloses), and lignin. It is well known that lignin, as a network polymer, binds with hemicelluloses and cellulose to form a tight compact structure (Adler, 1977). It makes the components of lignocelluloses difficult to be separated and thus restrict its applications. Therefore, development of pretreatment methods for the extraction and isolation of lignocelluloses is of great importance for broadening its industrial applications.

Until now, some successful pretreatment methods such as alkali extraction (Sun et al., 2000a), steam explosion (Biermann et al., 1984), hot water (Leppänen et al., 2011), enzymatic hydrolysis (Nhuan et al., 2011), bioconversion method (Himmel et al., 2007), and the hydrothermal method (Feria et al., 2012; Pińkowska et al., 2011; Pronyk and Mazza, 2012) were employed for the extraction and isolation of lignocelluloses. Among of these pretreatment methods, the alkali extraction included the concentrated solutions of sodium or potassium hydroxide, and alkaline hydrogen peroxide solution. The main advantages of the alkali extraction are easy to act and cost-effective. Alkaline peroxide treatment could separate cellulose, hemicelluloses, and lignin, and achieve complete utilization of lignocelluloses without the negative impact on the environment (Sun et al., 2000b). In addition, the hemicellulosic fractions obtained from alkali by ultrasound-assisted extraction showed a relatively lower content of associated lignin, a higher molecular weight, and a slightly higher thermal stability than those obtained without ultrasonic irradiation (Sun and Tomkinson, 2002). The steam explosion is another potential method to isolate of hemicelluloses from cereal straw and wood samples (Glasser et al., 2000). Higher yields of hemicelluloses can be achieved at shorter extraction times and lower temperatures. However, compared with the conventional method, the hydrothermal method is a promising technology for the preparation of lignocelluloses due to its advantages of short reaction time, high percent conversion, and relatively low reaction temperature.

In recent years, rapid progress has been made in the hydrothermal pretreatment for lignocelluloses (Feria et al., 2012; Goto et al., 2004; López et al., 2004; Makishima et al., 2006; Pińkowska et al., 2011; Pronyk and Mazza, 2012; Sasaki et al., 2003). For example, hydrothermal pretreatment was usually operated by exposing lignocelluloses to a chemical at certain temperature for a period of time depending on the physiochemical structure of the biomass (López et al., 2004). In addition, hydrothermal pretreatment to produce a series of xylooligosaccharides had been reported as an effective fractionation method for the major components (cellulose, hemicelluloses, and lignin) of lignocelluloses (Goto et al., 2004; Sasaki et al., 2003). Makishima et al. (2006)

* Corresponding author. Tel.: +86 10 62336592; fax: +86 10 62336972.
E-mail address: mg_ma@bjfu.edu.cn (M.-G. Ma).

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