
ABSTRACTS (MASTER THESIS)

Selective acetylation of matrix components in softwood ground pulps

**(Graduate School of Agriculture,
Laboratory of Active Bio-based Materials, RISH, Kyoto University)**

Tomohiro Yagi

The use of lignocellulose biomass remains a challenge in wood chemistry. Wood, the most abundant lignocellulosic resource, has long been used and researched as a raw material. However, wood has certain disadvantages such as dimensional stability, fungal resistance, photostability, and weathering. Chemical modifications can overcome such shortcomings. Specifically, acetylation, the simplest esterification, is an efficient method for improving the properties of wood (i.e., lignocellulose materials). In fact, acetylated wood is now commercialized.

This study prepared matrix-acetylated ground pulp to follow its acetylation process from the initial stage to the final stage of the reaction. Ground pulps, whose component ratio is approximately the same as raw wood, was selected as the sample. Acetylation was chosen as the most typical esterification. In detail, we prepared partially acetylated ground pulps with degree of substitution values of 1.2 (where it was expected that the matrix components were acetylated completely) and elucidated the position that the acetyl groups were introduced under the acetylation of ground pulps. We also investigated the effect of acetylation on thermal stability. Thermal stability is important for the use of lignocellulose materials as filler because plastics are molded from 150 to 300 °C. An increase in thermal stability will make the application to more plastics possible.

The acetylation of ground pulp was followed by heteronuclear single quantum coherence-nuclear magnetic resonance (HSQC-NMR) spectroscopy to investigate the relationship between the degree of acetylation and the introduction positions of acetyl groups during this acetylation. We also examined the effect of acetylation on thermal stability. Seven kinds of acetylated ground pulps with degree of substitution values of 0.19, 0.29, 0.59, 0.74, 1.04, 1.11, and 1.19 were prepared and analyzed using HSQC-NMR spectroscopy. Acetylation was found to occur first on the primary hydroxyl groups of polysaccharides and lignin, followed by the secondary hydroxyl groups of polysaccharides, and finally the hydroxyl groups at the α -position of lignin. The thermal stabilities of the acetylated ground pulps were analyzed using thermogravimetric analysis. The 1% weight loss temperatures of the acetylated ground pulps were higher than that of the ground pulps. The acetylation of the primary hydroxyl groups in polysaccharides and lignin and the hydroxyl groups at the α -position of lignin was more effective in improving thermal stability than that of the secondary hydroxyl groups in polysaccharides.