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Study of chemical modifications and fungi degradation of thermally modified wood using DRIFT spectroscopy

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Abstract A mild thermal treatment of wood leads to improved macroscopic properties (dimensional stabilization and resistance against fungal degradation). The chemical modifications induced by the thermal treatment were investigated by means of DRIFT spectroscopy on wood blocks in order to explain the new macroscopic properties on a molecular level. The formation of new ether linkage was observed in addition to the well-known acidic hydrolysis reactions of wood. Fungal attack was found to take place even after thermal degradation of pentosanes by a standard oxidative way. A competition between depolymerization and condensation reactions was observed.

DRIFT Untersuchung chemischer Veränderungen sowie des Pilzabbaumechanismus von wärmebehandelten Hölzern

Zusammenfassung Eine milde thermische Behandlung führt zu verbesserten makroskopischen Holzeigenschaften (Dimensionsstabilität und Pilzwiderstandsfähigkeit). Chemische Veränderungen von Holzstücken während der thermischen Behandlung wurden mit Hilfe von DRIFT Spektroskopie untersucht, um die neuen Eigenschaften auf molekularer Basis zu erklären. Die Entstehung neuer Etherbrücken sowie bekannte saure Hydrolysereaktionen von Holz wurden beobachtet. Obwohl die Pentosan-Moleküle nach der thermischen Behandlung abgebaut waren, konnte ein Pilzangriff auf normalem oxidativen Weg stattfinden. Eine Konkurrenz zwischen Depolymerisierungs- und Kondensationsreaktionen konnte beobachtet werden.

1 Introduction

Heating wood at higher temperatures than normal drying conditions has been shown to appreciably reduce the hygroscopicity and the subsequent swelling and shrinking of the ligno-cellulosic material (Stamm et al. 1937, Stamm et al. 1946, Kubinsky 1971, Giebeler 1983, Bourmester 1974). The Ecole Nationale Supérieure des Mines of Saint-Etienne has developed a thermal process (Guyonnet et al. 1986) named 'retification' in order to produce heat modified wood called 'retified wood' (Bourgois et al. 1988). This thermally modified wood has undergone a mild pyrolysis in inert atmosphere at a temperature between 200°C and 260°C under atmospheric pressure. The thermal modifications of wood result in changes of lignin and hemicelluloses (De Groot et al. 1988) that become less hygroscopic (Nebesarova 1996). The new properties of such wood consist principally of a lower swelling and shrinking, and also of a good resistance against fungi (Dirol et al. 1993, Weiland et al. 1997) without strong losses in strength.

The retification process can therefore be considered as a wood preservative treatment without external addition of chemicals which allows the modifications of the properties of fast-growing non-durable wood (such as poplar) in order to produce a durable material which can compete with tropical woods (teak, red cedar...). This study was performed in order to understand the origin of the high durability of retified wood and chemical changes during thermal treatment using infrared spectroscopy in a diffuse reflection mode on solid samples.

2 Experimental

Maritime pine (*Pinus pinaster*) and beech (*Fagus silvatica*) were treated in a 0.3 m^3 laboratory reactor under various conditions of temperature in nitrogen as shown in Table 1.

Thermally treated and non-treated wood specimens' resistance against fungal degradation was estimated by normalized European tests NF-EN-113. Maritime pine specimens were degraded by a brown-rot fungus (*Poria placenta*) during 16 weeks.

Infrared spectra of the treated and non-treated wood specimens before and after fungal attack were obtained with a Digilab BIORAD FTS 185 spectrometer equipped with a DRIFT accessory composed of a modified HARRICK model in order to obtain spectra on wood blocks (dimension 5 mm×5 mm×1 mm). Each DRIFT spectrum was transformed into an absorption spectrum with the help of the Kubelka–Munk theory (Kubelka et al. 1931). The validity of the Kubelka–Munk theory was confirmed in the case of wood blocks for the whole infrared and near infrared spectral region by Tsuchikawa and coworkers (Tsuchikawa et al. 1996). This technique permits to obtain an excellent reproducibility of the recorded spectrum.

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