J Wood Sci (2011) 57:459–461
DOI 10.1007/s10086-011-1190-1

RAPID COMMUNICATION

Hisashi Miyafuji · Nobuyoshi Suzuki

Observation by light microscope of sugi (*Cryptomeria japonica*) treated with the ionic liquid 1-ethyl-3-methylimidazolium chloride

Received: December 7, 2010 / Accepted: March 28, 2011 / Published online: July 1, 2011

Key words Wood · Ionic liquid · 1-Ethyl-3-methylimidazolium chloride · Liquefaction · Morphology

Introduction

To establish an efficient method for effective utilization of lignocelluloses, many studies on lignocellulose conversion technology have been carried out. A promising novel method is treatment with ionic liquids, which are organic salts with melting points in the vicinity of ambient temperature.[1] Ionic liquids have many notable characteristics such as negligible vapor pressure, thermal stability, recyclability, and nonflammability. It has been found that some ionic liquids are able to dissolve cellulose,[2] and there have been several reports on applications of ionic liquids to cellulose.[3–6]

Previous work has shown that wood components are decrystallized and depolymerized as they are liquefied by treatment with ionic liquids.[7] The differences in the reaction behavior of two different wood species with ionic liquid have been studied. Significant differences in lignin liquefaction behavior were explained by differences in the chemical structure of the lignin in the two wood species.[8] The reaction atmosphere was also found to influence the liquefaction of wood.[9] Although many studies on ionic liquid treatment of wood have already been reported, the resulting morphological changes of wood have not been elucidated. For that reason, in the present study, the morphological changes in wood after treatment with the ionic liquid 1-ethyl-3-methylimidazolium chloride were studied by light microscopy.

H. Miyafuji (✉) · N. Suzuki
Division of Environmental Sciences, Graduate School of Life and Environmental Sciences, Kyoto Prefectural University, Hangi-cho, Shimogamo, Sakyo-ku, Kyoto 606-8522, Japan
Tel. +81-75-703-5646; Fax +81-75-703-5646
e-mail: miyafuji@kpu.ac.jp

Materials and methods

Wood specimens from sugi (*Cryptomeria japonica*) [approximately 5(R) × 5(T) × 20(L) mm] were extracted with ethanol/benzene (1/2 v/v) for 8 h in a Soxhlet apparatus. The extracted wood was oven-dried at 105°C for 24 h prior to use. 1-Ethyl-3-methylimidazolium chloride ([C2mim][Cl]) was purchased from Tokyo Kasei Kogyo.

Specimens of extracted wood were sectioned with a sliding microtome to give 15-μm-thick transverse sections that were mounted in a hemocytometer with a depth of 20 μm (Sunlead Glass). A total of 100 μl of [C2mim][Cl] heated to 120°C was poured onto the mounted section, and the hemocytometer was immediately closed with a cover glass; the time at this point was taken as the beginning of the treatment. The hemocytometer was placed in an oven at 120°C for a set period of time and then removed from the oven to examine the morphological changes in the wood section by light microscopy (BH-2, OLYMPUS). The perimeters of six neighboring cell walls were measured on latewood and earlywood using image analysis software (Motic Image Plus 2.25). From these observations, average cell wall perimeters and the corresponding standard deviations were calculated.

Results and discussion

Figure 1 shows light micrographs of latewood, earlywood, and the latewood/earlywood boundary after treatment with [C2mim][Cl] at 120°C for various times. The cell walls in latewood at 0 h treatment were well ordered, but they became disordered after 0.5 h treatment. Moreover, the distortion of latewood with some occlusion of cell lumens was observed in whole samples after 0.5 h treatment, although neither destruction nor flaking of the cell walls was seen. After 4 h treatment, however, some destruction or flaking was observed in the cell walls. By contrast, no significant morphological changes could be seen in earlywood. Even after 4 h treatment, earlywood had a similar form to that

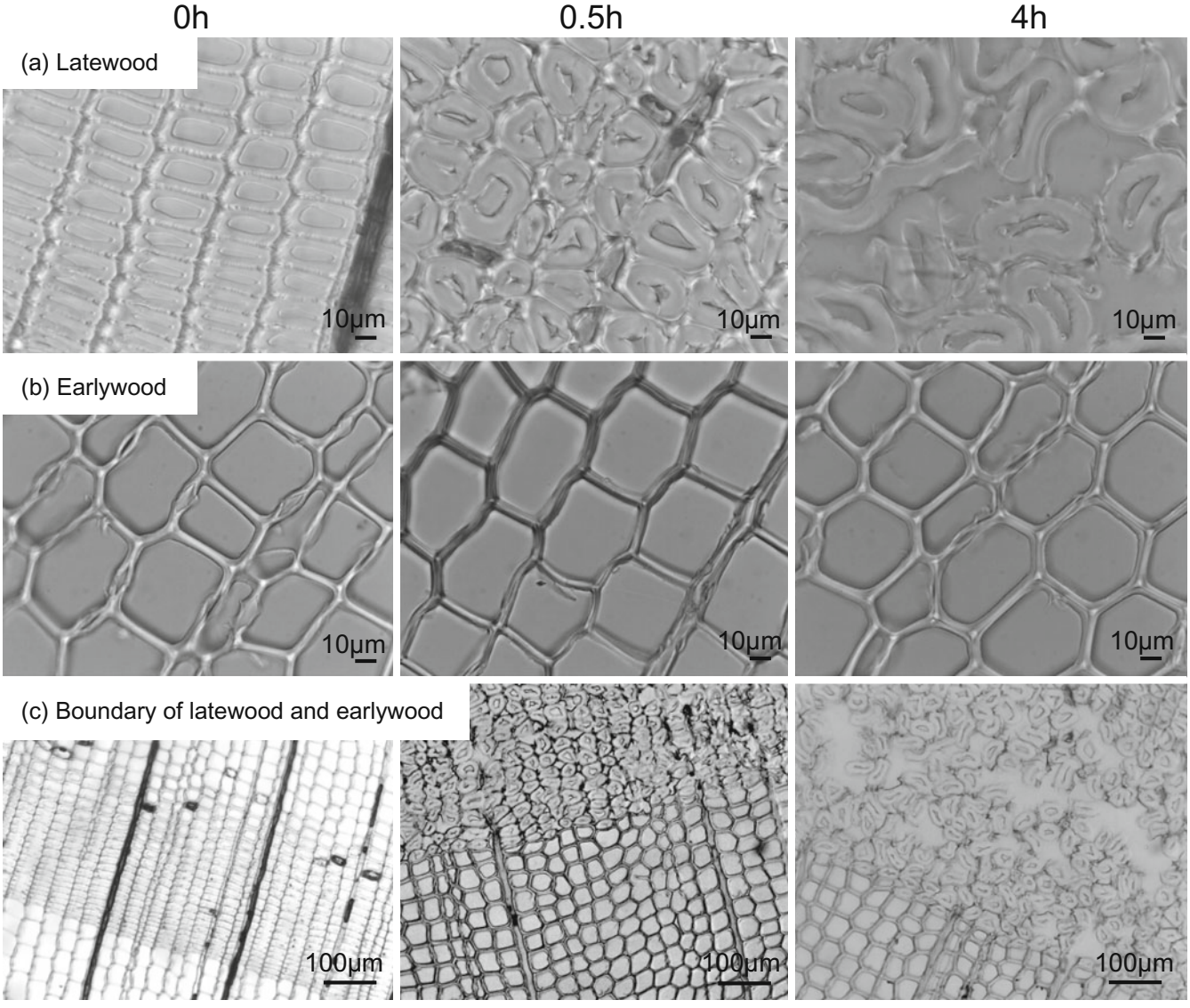

Fig. 1. Light microscopy images of wood treated with 1-ethyl-3-methylimidazolium chloride at 120°C for various times. **a** Latewood, **b** earlywood, **c** boundary of latewood and earlywood

Table 1. Changes of cell wall perimeter in latewood and earlywood

Treatment time (h)	Cell wall perimeter (μm)			
	Latewood		Earlywood	
	Average	S.D.	Average	S.D.
0	75	10	146	9
0.5	101	13	145	11
4	107	14	141	12

S.D., standard deviation

before treatment. These differences in latewood and earlywood can be easily seen in the images of the boundary regions of latewood and earlywood.

Table 1 shows the cell wall perimeters in latewood and earlywood after various treatment times. The cell wall perimeter in latewood was found to increase significantly, whereas no substantial changes were found in earlywood. These results indicate that the cell walls in latewood swelled as a result of [C2mim][Cl] treatment. In a previous study on the swelling of wood by water, it was reported that latewood swells much more than earlywood because the specific gravity of the former is higher than that of the latter.[10] [C2mim][Cl] is thought to exhibit a similar swelling effect to that of water, and the different swelling behavior of latewood and earlywood is assumed to cause the difference in morphology shown in Fig. 1.

In our previous study, more than 80% of western red cedar was found to be liquefied by [C2mim][Cl] treatment at 120°C for 24 h.[8] The light micrographs in Fig. 1 are thought to show the morphological changes of the cell walls before they disappear through liquefaction. Thus, the results of the present study revealed that the liquefaction of wood by [C2mim][Cl] is not homogeneous, and marked distortion and dissociation of the cell walls in latewood occurs at an early stage of liquefaction. In our next report, the swelling

behavior and distortion of the cell walls will be investigated in detail to clarify the mechanism of liquefaction of wood in [C2mim][Cl] from the morphological viewpoint.

Acknowledgment This research was supported in part by the TOSTEM Foundation for Construction Materials Industry Promotion.

References

1. Seddon KR (1997) Ionic liquids for clean technology. J Chem Tech Biotechnol 68:351–356
2. Swatloski RP, Spear SK, Hobrey JD, Rogers RD (2002) Dissolution of cellulose with ionic liquids. J Am Chem Soc 124:4947–4975
3. Turner MB, Spear SK, Holbrey JD, Rogers RD (2004) Production of bioactive cellulose films reconstituted from ionic liquids. Biomacromolecules 5:1379–1384
4. Wu J, Zhang J, Zhang H, He J, Ren Q, Guo M (2004) Homogeneous acetylation of cellulose in a new ionic liquid. Biomacromolecules 5:266–268
5. Heinze T, Schwikal K, Barthel S (2005) Ionic liquids as reaction medium in cellulose functionalization. Macromol Biosci 5:520–525
6. Barthel S, Heinze T (2006) Acylation and carbanilation of cellulose in ionic liquids. Green Chem 8:301–307
7. Miyafuji H, Miyata K, Saka S, Ueda F, Mori M (2009) Reaction behavior of wood in an ionic liquid, 1-ethyl-3-methylimidazolium chloride. J Wood Sci 55:215–219
8. Nakamura A, Miyafuji H, Saka S (2010) Liquefaction behaviour of Western red cedar and Japanese beech in the ionic liquid 1-ethyl-3-methylimidazolium chloride. Holzforsch 64:289–294
9. Nakamura A, Miyafuji H, Saka S (2010) Influence of reaction atmosphere on the liquefaction and depolymerisation of wood in an ionic liquid, 1-ethyl-3-methylimidazolium chloride. J Wood Sci 56:256–261
10. Skaar C (1983) Wood–water relationships. In: Rowell R (ed) The chemistry of solid wood. American Chemical Society, Washington, D.C., pp 127–172