

Ionic liquid pre-treatment to reduce the elastic spring-back and set-recovery of surface densified Scots pine

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Although it is not a new research area in the field of wood science, in recent years, surface densification of wood became a subject of increasing popularity. It was shown that creating a layer of densified wood cells of a few millimetre thickness just beneath the surface can lead to an increase in hardness by approximately 100% (Gong *et al.* 2010, Laine *et al.* 2013).

One of the major obstacles regarding the wide-spread commercialisation of surface-densified wood products is the reduction of the elastic spring-back and set-recovery of the deformed wood cells in the context of an industrial process. The elastic spring-back can be greatly reduced by introducing a cooling stage before releasing the pressure at the end of the densification process (Neyses 2016). The set-recovery can be nearly eliminated by applying different methods, such as chemical modification or by thermal-hydro-mechanical treatments of the densified wood cells (Kutnar *et al.* 2015). A theory describing the underlying mechanisms of the elastic spring-back and the set-recovery was presented by Navi and Sandberg (2012) and further described by Navi and Pizzi (2014). It is suggested that the set-recovery occurs due to elastic deformation of cellulose, which is 'frozen' inside the plastically deformed matrix of lignin and hemicellulose. Plasticisation of the wood after it has been densified, e.g. through moisture, leads to a recovery of the elastic deformation. Hence, the set-recovery can be greatly reduced if plastic deformation of the cellulose can be achieved. This is, however, difficult to accomplish with the established methods of plasticising wood, such as the combination of moisture and heat, or treatment with gaseous ammonia.

In the past decade, several studies explored the possibility of plasticising cellulose by treatment with so-called ionic liquids (Swatloski *et al.* 2002, Kilpeläinen *et al.* 2007). Apart from being considered 'green solvents' of cellulose, ionic liquids are reported to have a positive effect on antifungal and antimicrobial activity, and on fire and UV degradation resistance. Ou *et al.* (2014) reported that ionic liquid treatment transforms wood into a thermoplastic material, without or only little elastic deformation when put under load. This behaviour suggests that the elastic spring-back and the set-recovery can be largely reduced.

The objective of the present study was to evaluate the effect of ionic liquid pre-treatment on the elastic spring-back and set-recovery of surface-densified wood.

The study was carried out on Scots pine (*Pinus sylvestris* L.) specimens treated with a 20% weight solution of 1-Ethyl-3-methylimidazolium chloride, solved in ethanol. The specimens were soaked in the solution prior to being surface-densified on the sapwood side in a hot press at up to 250°C for 2 to 7 minutes.

As no cooling stage was applied before releasing the pressure, the elastic spring-back of the untreated control group was 50-60% (based on the compressed thickness). The ionic liquid treatment reduced the spring-back to less than 20%, similar to values obtained if a cooling stage would have been applied. Depending on the pressing time, the set-recovery could be reduced from about 90-100 % (control group) to 20-30%, where it appears that part of the remaining set-recovery can be attributed to inadvertent densification of earlywood regions in the untreated core of the specimens. The high pressing temperature led to thermal degradation of the densified wood cells, which could be an additional contribution to the reduction in set-recovery. Visual inspection and Brinell hardness tests suggest that the specimens treated with ionic liquids have a more pronounced density peak close to the surface in comparison to the untreated specimens.

The results of the present study indicate the potential of ionic liquids as a strong plasticiser of wood, reducing the elastic component of deformation of wood under load. In this way, the elastic spring-back and set-recovery are to a certain extent prevented from occurring in the first place. Further studies are needed to optimize the treatment and to gain a better understanding of the treatment mechanism.

References

- Gong, M., Lamason, C., Li, L. 2010. Interactive effect of surface densification and post-heat-treatment on aspen wood. *Journal of Materials Processing Technology*, 210, 2: 293-296.
- Kilpeläinen, I., Xie, H., King, A., Granstrom, M., Heikkinen, S., Argyropoulos, D. S. 2007. Dissolution of wood in ionic liquids. *Journal of Agricultural and Food Chemistry*, 55, 22: 9142-9148.
- Kutnar, A., Sandberg, D., Haller, P. 2015. Compressed and moulded wood from processing to products. *Holzforschung*, 69, 7: 885-897.
- Laine, K., Rautkari, L., Hughes, M. 2013. The effect of process parameters on the hardness of surface densified Scots pine solid wood. *European Journal of Wood and Wood Products* 71: 13-16.
- Navi, P., and Sandberg, D. 2012. *Thermo Hydro Mechanical Processing of Wood*, EPFL Press, Lausanne, Switzerland.
- Navi, P., Pizzi, A. 2014. Property changes in thermo-hydro-mechanical processing. *Holzforschung*, 69, 7: 863-873.
- Neyses, B. 2016. *Surface-Densified Wood – From Laboratory-Scale Research Towards a Competitive Product*. Licentiate Thesis, Luleå University of Technology.
- Ou, R., Xie, Y., Wang, Q., Sui, S., & Wolcott, M. P. 2014. Thermoplastic deformation of poplar wood plasticized by ionic liquids measured by a nonisothermal compression technique. *Holzforschung*, 68, 5: 555-566.
- Swatloski, R. P., Spear, S. K., Holbrey, J. D., Rogers, R. D. 2002. Dissolution of cellulose with ionic liquids. *Journal of the American Chemical Society*, 124, 18: 4974-4975.