Continuous Surface Densification of Wood: A New Concept for Large-scale Industrial Processing

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Fast growing and low-density species can be modified by various thermo-hydro-mechanical (THM) treatments. Wood densification is one of the promising techniques for broadening the application of these species. This study focuses on the use of a high-capacity continuous pressing technique that considerably increases the density in the region beneath the surface of poplar wood. Prior to densification at 185 °C, a softening stage was implemented, with water spraying followed by heating at a temperature of 205 °C to 235 °C. The density profile, set-recovery, and morphology of the densified surface were investigated. Densitometry revealed that an M-shaped density profile was created through the thickness, with a peak density of approximately 700 kg/m^3 close to the surfaces. The set-recovery after three wetting-drying cycles was 44%, which revealed that partial stress relaxation occurred during the densification. Scanning electron microscopy (SEM) confirmed that both sides of the wood were successfully densified and that after the wetting-drying cycles, the deformed cells did not completely recover.

Keywords: Poplar wood; Compression; Thermal-hydro-mechanical processing; Softening

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INTRODUCTION

There is considerable interest in developing thermo-hydro-mechanical (THM) treatments for low-density wood species to increase their density and thereby improve their mechanical properties and overall wood quality (Sandberg 1998; Sandberg *et al.* 2013). This paper introduces a new concept for a high-capacity surface densification process, which aims to improve the mechanical properties by compressing only the first few millimetres beneath the surface (Laine 2014). This process results in an increased abrasion resistance and an increased hardness of the wood surface. It is of particular interest in applications where the surface is exposed, such as tabletops and flooring. This allows the use of low-density species in applications where hardwood species, such as ash, beech, and oak, or other materials, have traditionally been used.

Generally, there is a strong correlation between the wood density and most of its mechanical properties, *i.e.,* high-density wood has better mechanical properties than low-density wood. There is an increasing environmental concern and demands for forest products and smaller volumes of high-quality wood from the forestry. These demands are providing motivation for the use of low-quality wood species as replacements for high-quality wood and fossil fuel-based materials (Kutnar and Sandberg 2015). To meet the required performance of the end products, the low-quality wood must in many cases be

modified.

The THM densification process is a widespread technique for modifying wood by compression in the transverse direction of the wood. Heat, steam, and mechanical compression are used to densify and fix the wood material. The principle of THM processing has been thoroughly described by Navi and Sandberg (2012), and some recent applications have been presented in Kutnar *et al.* (2015). The THM densification process has three main stages. The first stage consists of softening (plasticizing) the wood substance in a region that is to be compressed. The second stage is transverse compression of the softened wood structure to a degree that corresponds to the target density, normally *ca*. 1,000 kg/m^3. The final stage is stress relaxation and/or locking of the deformed structure to prevent it from returning to its original shape (*i.e.*, to prevent set-recovery) when subjected to moisture variations.

Wood is a porous material that consists mainly of the structural polymers cellulose, hemicelluloses, and lignin, and the behavior of these components can be altered by heat and moisture (Seborg *et al.* 1956). When wood is plasticized, the effect is mainly due to the changes in the properties of the lignin as result of the increase in temperature. However, the glass transition temperature is also strongly dependent upon the moisture level of the wood material, as shown by Salmén (1982) (Fig. 1). When the solid wood of sawn-timber dimensions is softened in an open system, the temperature must reach *ca*. 100 °C at a moisture content of 20% to 25% to achieve proper plasticization. After softening, the wood cells can be compressed radially by more than 50% without damage (Navi and Sandberg 2012).

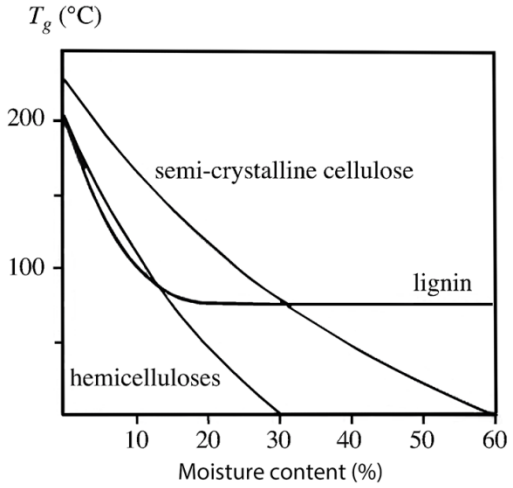

Fig. 1. Glass transition temperature (T_g) of isolated components of wood as a function of moisture content (Salmén 1982)

To create a high-density wood surface, an adequate volume of wood beneath the surface must be softened (Rautkari *et al*. 2009). Because of the low heat conductivity of wood, local moistening helps to soften the region of the wood and improve its compressibility, while the rest of the wood continues to resist compressive deformation (Wang and Cooper 2005; Lamason and Gong 2007). Once the desired volume of wood material beneath the surface has been softened, that volume should be compressed *via* the application of an external load.

When subjected to transverse compression, the cellular structure of wood undergoes a large deformation in precise stages (Sandberg *et al.* 2013). Wood under

compression starts to collapse in the weakest part of the material. A typical deformation pattern in the transverse compression of a softwood species, such as Scots pine, starts with the deformation of the first layer of the low-density earlywood cells, close to the annual ring boarders (Nilsson *et al.* 2011). This layer buckles when the yield point of the cell-wall material is reached. Moreover, the density is increased *via* diminishing the lumens until the cell walls totally collapse and come into contact. This new shape of the cells increases the density of the compressed layer so that it can withstand further compression. Furthermore, the next layers of the earlywood cells begin to collapse until the whole earlywood band is compressed. The compression deformation thereafter propagates into the latewood, and this stage is clearly distinguished in the load-deformation diagram by a drastic increase in the load (Müller *et al.* 2003).

Densified wood is capable of recovering its original shape even after a large deformation, especially at high humidity and under a high temperature. It exhibits two types of dimensional instability. (1) Wood swelling is an irreversible dimensional instability that is due to the hygroscopic nature of wood. (2) Densified wood also exhibits an irreversible dimensional instability related to its elastic-visco-plastic properties and due to the release of inner stresses stored in the wood during densification. When the load is removed from the compressed wood, the elastic deformation is instantaneously recovered (Bodig and Jayne 1982). This recovery of deformation is called spring-back. As time passes, additional deformation will be recovered as a consequence of the visco-elastic nature of wood. This part of the recovery involves a mix of delayed-elastic recovery and viscous recovery, but under constant humid condition recovery is in general complete and a residual deformation remains. The remaining deformation can be released if the wood is exposed to humidity (in absence of external force), and the densified wood then almost completely returns to its initial dimensions before densification. This re-shaping of wood cells is called compression-set recovery, set-recovery, or shape memory (Navi and Sandberg 2012). If some deformation remains after the complete re-moistening of the wood structure, the residual deformation can be related to the damage in the wood structure, or possibly to "true" plastic deformation.

The origin of the shape memory is to be found at the level of the cell-wall ultrastructure and molecular structure. The assumptions formulated by Norimoto *et al.* (1993) can be summarized as follows:

Moisture and temperature act in various manners on the matrix and the microfibrils. A rise in temperature under wet conditions softens the matrix (amorphous hemicelluloses and lignin) and the semi-crystalline zones of the cellulose, and these components pass from the glassy to a quasi-rubbery state. The cellulose microfibrils (crystalline regions), however, remain in their glassy state because of their crystalline nature and they are almost unaffected by the moisture and heat. When a compressive load is applied to wood in the transverse direction, the load is almost totally supported by the microfibrils. The softening of the matrix allows a relative displacement of the microfibrils so that the framework of microfibrils becomes elastically deformed to take up the local loads. As lignin is a polymer with slight cross-linkage, its deformation can be regarded as viscoelastic rather than plastic. The elimination of the water molecules during drying and the reduction of thermal activation energy due to cooling lead to a reformation of the hydrogen bonds between the molecules of the matrix components. As a result of the reduction in temperature during cooling, the densified wood returns to a glassy state, where the elastic strain of the microfibrils and the matrix are frozen. Consequently, no recovery can occur until the matrix is again softened. However, as soon as the matrix is

humidified and heated, the wood almost entirely recovers its initial form because of the release of the elastic energy stored in the microfibrils and the entropic and elastic molecular movements within the matrix.

It has been suggested that semicrystalline microfibrils and lignin are deformed elastically without plastic flow even after a yield strain. Therefore, densified wood exhibits a large set-recovery in moist and warm conditions, due to the residual stress stored in the collapsed cells (Navi and Heger 2004). The set-recovery must be prevented if the wood is to remain densified. Three mechanisms have been proposed: 1) changing the hydrophilic agent to less water-accessible components, 2) the creation of covalent cross-links between the wood components, and 3) releasing the energy stored in the microfibrils and wooden matrix (Morsing 2000). The time and temperature have a large influence on the fixation of wood and can reduce the set-recovery. Inoue *et al.* (2008) reported that increasing the time and pre-steaming temperature reduces the set-recovery of transversely densified wood. A mechanical fixation *via* gluing or impregnation with adhesives is also a possible method to prevent set-recovery.

Surface densification relies on time- and energy-consuming batch processes. This eliminates the potential advantage of using low-density instead of high-density species or non-renewable materials (Neyses *et al*. 2016). For decades the wood-panel industry has focused on manufacturing boards with continuous presses because it is economically beneficial. Continuous presses process material continuously as it moves through the machine. The press provides a specific pressure, or thickness, with minimal variation during production to achieve the desired thickness reduction with a specialized hydraulic thickness-controlling system. Continuous presses also have controllable heating zones along their length. This adds another variable to control the product properties (Thoemen and Humphrey 2003; Thoemen *et al*. 2010). The large-scale THM surface densification of solid wood *via* continuous pressing has the potential to improve the quality, reduce the cost of production, and precisely control the processing parameters (Sandvik 2009).

The purpose of the present study was to develop a new industrial concept for continuous THM surface densification, where the normal equipment in the wood-panel industry is used. The objective was to verify if the equipment could be used for the softening and compression of the wood surface. There was less attention paid to the prevention of set-recovery at this stage.

EXPERIMENTAL

Materials

A total of 25 poplar (*Populus deltoides* Bartr. ex.) specimens with the dimensions of 21 mm (thickness (T)) x 100 mm (width (W)) x 300 mm (length (L)), and a density of 428 kg/m^3 at 12% moisture content (MC) that were used. All specimens were cut from sapwood with a similar grain orientation, and as uniform of an annual ring thickness as possible.

The annual ring orientation in the cross-section of the specimens was nearly horizontal (flat-grained wood). For the set-recovery and SEM studies, one specimen was randomly selected from the total group of specimens.

Methods

The densification process

A Siempelkamp A0361 continuous panel press (G. Siempelkamp GmbH & Co. KG, Krefeld, Germany) was used (Fig. 2). The press consisted of upper and lower heating devices with four separate adjustable heating sections, two sets of rolling elements to reduce the friction between the stationary heating plates, and movable steel belts. The steel belts transferred heat and pressure to the wood while it transported the wood through the press. The total length of the press was 18 m, with a pressing zone of 6 m. The temperatures of the zones could be adjusted separately along the length of the press. The press was also equipped with a distance system with 13 independent sections where the distance between the steel belts could be adjusted. The press was distance-regulated, *i.e.*, the pressing force was regulated so that the distance between the steel belts was kept at the desired value along the press. The process parameters used in the study are shown in Table 1.

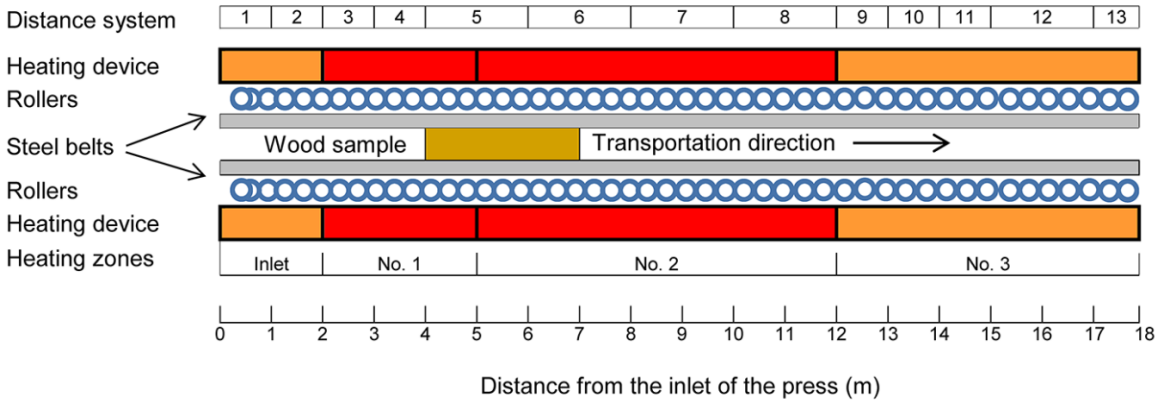

Fig. 2. A schematic side view (length-to-height section) of the continuous press used in the study

Table 1. Press Parameters Used in the Study

Conveyor Speed (m/min)	6.3			
Thickness Reduction (mm)	2.6			
Total Time in Press (min)	2.9			
Heating Zones	Inlet	Zone 1	Zone 2	Zone 3
Temperature (°C)	205	235	228	182
Time in Zone (s)	19	29	67	57
Compression	No	No	No	Yes

Before the specimens entered the press, both surfaces were sprayed with approximately 50 g/m^2 of water, and the specimens were heated to a temperature of 205 °C to 235 °C (heating-device temperature) as they passed through the inlet, zones 1 and 2, for 115 s without compression. The heated specimens were gradually compressed to a thickness of 18.4 mm over 57 s (Fig. 3). Finally, the specimens were cooled to room temperature. The pith side of the specimens was oriented toward the lower steel belt.

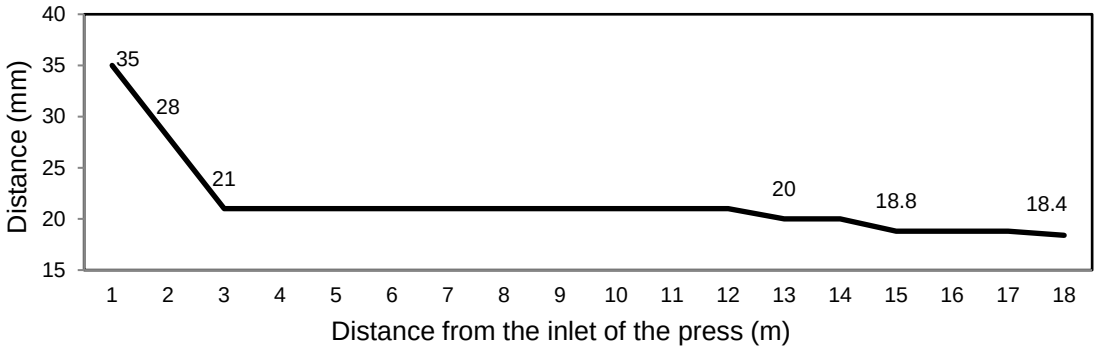

Fig. 3. Distance between the upper and lower steel belt along the press

Evaluation of the specimens

The thickness was measured before and after pressing, and an ocular examination of the specimen was performed.

To evaluate the density profile through the thickness, the densified specimens were scanned by X-ray computer tomography (CT) after being conditioned at 20 °C and 65% relative humidity (RH). Cross-sectional scanning was performed in the length direction at every 1 mm, and the images were analysed with the ImageJ image-processing software (Rasband 1997). Four samples were cut from the densified wood to be evaluated for set-recovery. The samples were dried to a constant weight at 103 °C before the first of three soaking-drying cycles in water at 20 °C for 24 h, with intermediate drying at 103 °C for 24 h. The set-recovery was calculated as follows,

$$\text{Set Recovery} = \frac{t_S - t_{C_0}}{t_I - t_{C_0}} \quad (1)$$

where t_s is the oven-dry thickness after soaking (mm), t_{c_o} is the thickness of the oven-dry compressed wood (mm), and t_I is the thickness of the initial uncompressed wood (mm) at 12% moisture content (MC).

To study the deformation of cells after densification, samples with dimensions of 3 mm × 5 mm (tangential × radial) were cut from the middle of the surface region in the width direction of the specimen. All samples were cut to size with a razor and then gold-sputter-coated for 40 s. The SEM images were taken in a Jeol-JSM5200 (Peabody, MA, USA) at 15 kV. To investigate the micromorphological changes after three cycles of water soaking and drying, similar samples were cut from the reversed specimens and studied in the same way as the densified samples.

RESULTS AND DISCUSSION

The densified surfaces had a smooth, light-brownish appearance after the compression and thermal treatment. A density profile of a sample is shown in Fig. 4. The density peaks on each side of the specimen indicated a clear surface densification. The surface directed towards the lower side of the press showed a slightly higher density than that of the upper side. This was expected, as the lower side of the specimen was in direct contact with the heated steel belt at the inlet of the press (Fig. 2), and was thereby heated for a longer time than the upper side of the specimen.

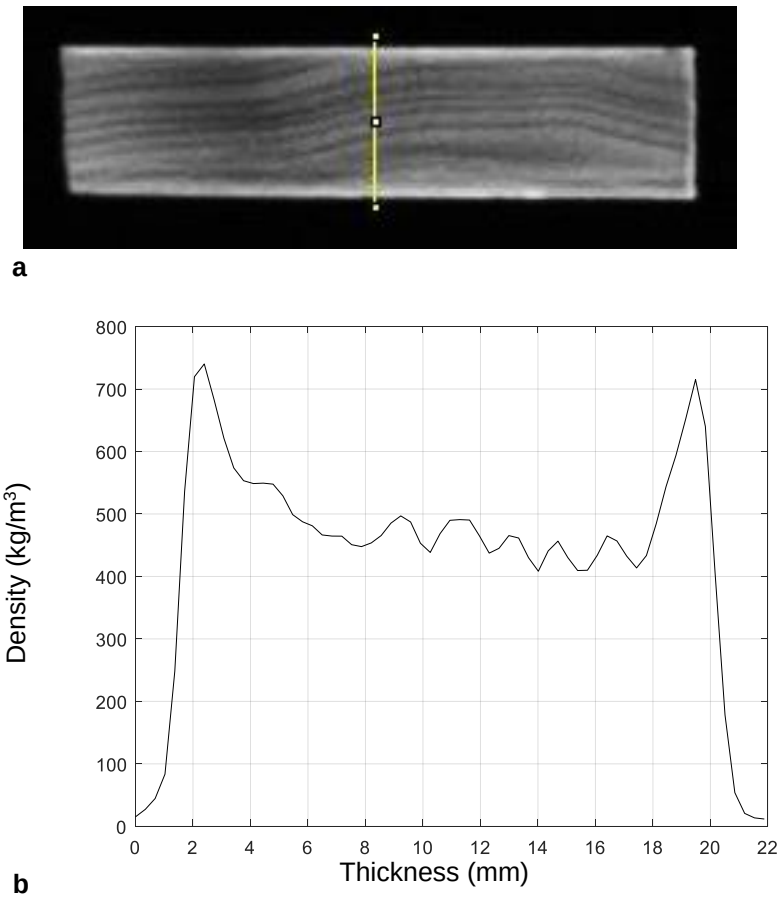

Fig. 4. (a) The CT image of a cross-section of surface-densified poplar, and (b) the density profile through the thickness along the indicated line. The zero point is the surface of the specimen on the lower side of the press

The set-recovery after the first soaking-drying cycle was 37%, and after a total of three cycles it was 44% (Fig. 5). This was a considerably lower set-recovery compared to the results from untreated Scots pine (Laine *et al*. 2013). The cited authors reported a set-recovery of approximately 80%.

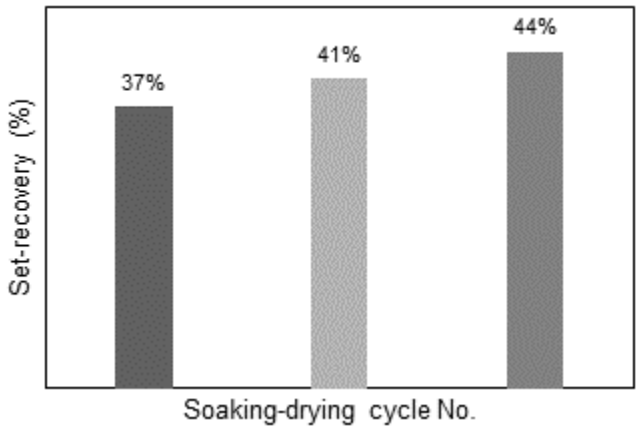

Fig. 5. Set-recovery of surface densified wood after 1, 2, and 3 soaking-drying cycles

The lowered set-recovery in the present study was probably due to a combination of pre-wetting by spraying water onto the surface and a long pre-heating period before the compressive force was applied. Research by Inoue *et al*. (2008) supports this, showing a pre-treatment of the wood with steam at 120 to 220 °C for a period of 5 to 10 min reduces the set-recovery.

The SEM study of the densified poplar also showed a distinct densified layer beneath the surfaces of the specimens. Figure 6a shows that most of the vessels and fibre cells were locally densified during compression as a consequence of the combination of high temperature and moistening of the surface before compression, which softened the wood in the region just beneath the surface. The first rows of earlywood adjacent to the annual-ring borders in poplar have large and thin-walled vessels, which were susceptible to collapse. These vessels collapsed during compression even below the densified zone (Fig. 6b) after the softened area beneath the surface was completely densified. The contact points between the cell walls of the collapsed cells increased the rigidity of the softened area beneath the surface and helped to transfer further deformation to the weakest layers further inside the specimen.

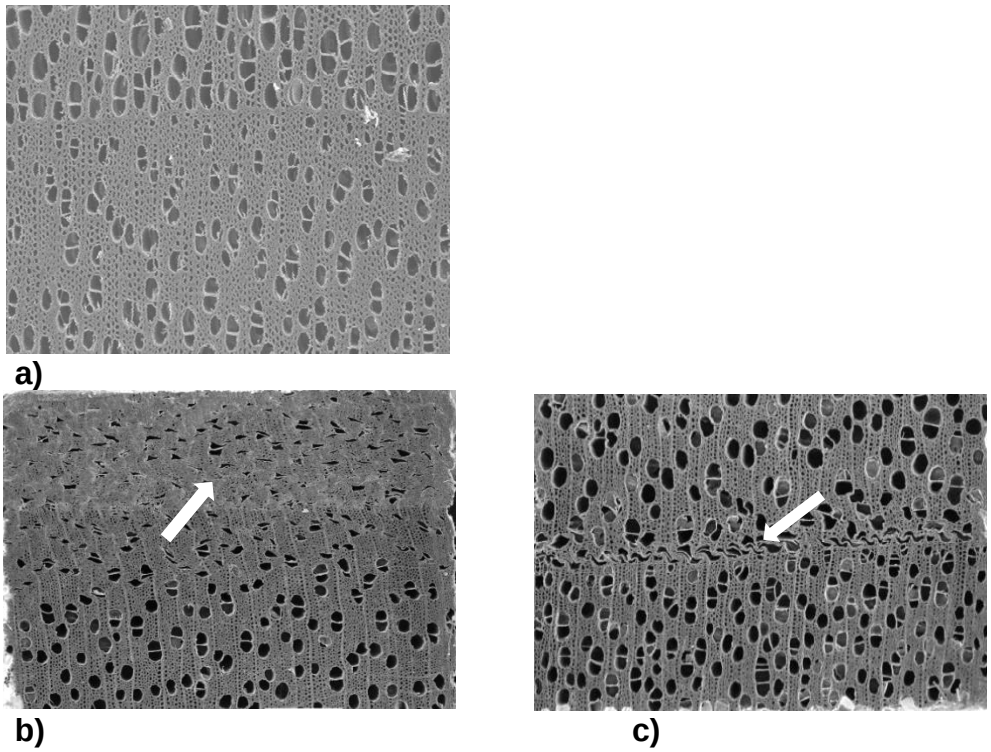

Fig. 6. Cross-section views of poplar: a) before densification, b) after densification showing cell deformation close to the surface, and c) cell collapse adjacent to an annual-ring border further inside the specimen

Figure 7 shows an example of the cell structure after three soaking-drying cycles, where vessels and fibres in both the earlywood and latewood could not fully recover to their original shape. The partial set-recovery after the soaking-drying cycles indicated that stress relaxation took place during the densification process. The large vessels adjacent to the annual-ring borders showed signs of cell wall damage that enabled relaxation to occur under pressure.

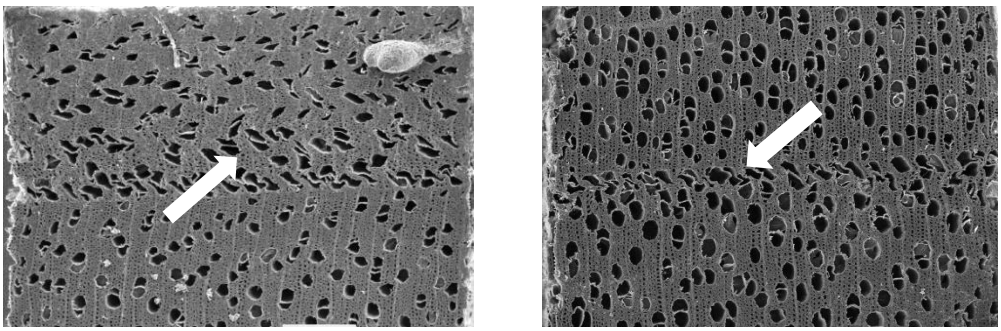

Fig. 7. Cross-section views of densified poplar after three soaking-drying cycles: a) set-recovery of densified cells beneath the surface, and b) set-recovery of collapsed vessels adjacent to annual-ring border further inside the specimen

CONCLUSIONS

1. This study showed that it was possible to densify low-density poplar using a continuous press that was normally used for the industrial manufacture of wood panels, such as particleboard.
2. This process could be regulated so that the density is increased within a few mm thick zone beneath the surface of the wood, *i.e.*, sub-surface densification is achieved.
3. Set-recovery of the densified cells occurred after the soaking-drying cycles, but was considerably lower than in the other densification studies performed under static conditions. It was suggested that the lowered set-recovery was a consequence of the specific process conditions, *i.e.*, a combination of pre-wetting by spraying water onto the surface and a long pre-heating period before the compressive force was applied.
4. The SEM study confirmed that the wood cells, in both earlywood and latewood, beneath the surfaces were satisfactory plasticized and compressed.

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REFERENCES CITED

Bodig, J., and Jayne, B. A. (1982). *Mechanics of Wood and Wood Composites*, Van Nostrand Reinhold Company Inc., New York, Cincinnati.

Inoue, M., Sekino, N., Morooka, T., Rowell, R. M., and Norimoto, M. (2008). "Fixation

of compressive deformation in wood by pre-steaming," *Journal of Tropical Forest Science* 20(4), 273-281.

Kutnar, A., and Sandberg, D. (2015). "Next steps in developing thermally modified timber to meet the requirements of the European low-carbon economy," *International Wood Products Journal* 6(1), 8-13. DOI: 10.1179/2042645314Y.0000000079

Kutnar, A., Sandberg, D., and Haller, P. (2015). "Compressed and moulded wood from processing to products," *Holzforschung* 69(7), 885-897. DOI: 10.1515/hf-2014-0187

Laine, K. (2014). *Improving the Properties of Wood by Surface Densification*, Ph.D. Dissertation, Aalto University, Espoo, Finland.

Laine, K., Rautkari, L., Hughes, M., and Kutnar, A. (2013). "Reducing the set-recovery of surface densified solid Scots pine wood by hydrothermal post-treatment," *European Journal of Wood and Wood Products* 71(1), 17-23. DOI: 10.1007/s00107-012-0647-2

Lamason, C., and Gong, M. (2007). "Optimization of pressing parameters for mechanically surface densified aspen," *Forest Products Journal* 57(10), 64-68.

Morsing, N. (2000). *Densification of Wood: The Influence of Hygrothermal Treatment on Compression of Beech Perpendicular to the Grain,* Ph.D. Dissertation, Technical University of Denmark, Copenhagen, Denmark.

Müller, U., Gindl, W., and Teischinger, A. (2003). "Effects of cell anatomy on the plastic and elastic behavior of different wood species loaded perpendicular to grain," *IAWA Journal* 24(2), 117-128. DOI: 10.1163/22941932-90000325

Navi, P., and Heger, F. (2004). "Combined densification and thermo-hydro-mechanical processing of wood," *MRS Bulletin* 29(5), 332-336. DOI: 10.1557/mrs2004.100

Navi, P., and Sandberg, D. (2012). *Thermo Hydro Mechanical Processing of Wood,* EPFL Press, Lausanne, Switzerland.

Neyses, B., Hagman, O., Sandberg, D., and Nilsson, A. (2016). "Development of a continuous wood surface densification process- the roller pressing technique," in: *Proceedings of the 59th International Convention of Society of Wood Science and Technology*, Curitiba, Brazil, pp. 17-24.

Nilsson, J., Johansson, J., Kifetew, G., and Sandberg, D. (2011). "Shape stability of modified engineering wood product (EWP) subjected to moisture variation," *Wood Material Science and Engineering* 6(3), 42-49. DOI: 10.1080/17480272.2011.552122

Norimoto, M., Ota, C., Akitsu, H. and Yamada, T. (1993). "Permanent fixation of bending deformation in wood by heat treatment," *Wood Research* 79, 23-33.

Rasband, W. S. (1997). "ImageJ," U. S. National Institutes of Health, Bethesda, MD, USA, (http://imagej.nih.gov/ij/). Accessed on June 16, 2016.

Rautkari, L., Properzi, M., Pichelin, F., and Hughes, M. (2009). "Surface modification of wood using friction," *Wood Science Technology* 43, 291-299. DOI: 10.1007/s00226-008-0227-0

Salmén, L. (1982). *Temperature and Water Induced Softening Behaviour of Wood Fiber Based Materials,* Ph.D. Dissertation, Royal Institute of Technology (KTH), Stockholm, Sweden.

Sandberg, D. (1998). *Value Activation with Vertical Annual Rings- Material, Production, Products*, Ph.D. Dissertation, Royal Institute of Technology (KTH), Stockholm, Sweden.

Sandberg, D., Haller, P., and Navi, P. (2013). "Thermo-hydro and thermo-hydro-mechanical wood processing: An opportunity for future environmentally friendly wood products," *Wood Material Science and Engineering* 8(1), 64-88. DOI:

10.1080/17480272.2012.751935
Sandvik, A. B. (2009). *Steel Belts for the Production of Wood Based Panels*, Sandvik Process Systems, Sandvik, Sweden.
Seborg, R. M., Millett, M. A., and Stamm, A. J. (1956). *Heat Stabilized Compressed Wood (Staypak)* (Report No. 1580), U.S. Department of Agriculture, Forest Products Laboratory, Madison, WI.
Thoemen, H., and Humphrey, P. E. (2003). "Modelling the continuous pressing process for wood-based composite," *Wood and Fiber Science* 35(3), 456-468.
Thoemen, H., Irle, M., and Sernek, M. (2010). *Wood-Based Panels-An Introduction for Specialists*, Brunel University Press, London, England.
Wang, J., and Cooper, P. A. (2005). "Vertical density profiles in thermally compressed balsam fir wood," *Forest Products Journal* 55(5), 65-68.

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