

## EFFECTS OF HEAT-TREATMENT ON PERMEABILITY OF UNTREATED AND NANOSILVER-IMPREGNATED NATIVE HARDWOODS

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### ABSTRACT

The effects of heat-treatment on permeability are the main topic of the present study. Longitudinal dowel shape pairs of specimens were prepared from three native species and their specific gas permeability values were measured. From each pair, one specimen was kept for heat-treatment and the other was impregnated with 200 ppm nano-silver suspension; the size range of nanoparticles was 20-80 nm. In six consecutive steps, each pair was heated at 50, 75, 100, 125, 150, and 185°C. Results showed that gas permeability increased when specimens were heated at 50°C as a result of the loss of bond-water and consequent shrinkage. Permeability sharply decreased when the specimens were heated at 75°C and gradually increased at each step up-to 150°C. At 185°C, permeability again decreased in. The sharp decrease in permeability at 75°C was possibly due to irreversible hydrogen bonding in the course of water movements within the vessel perforations and pore system of cell walls. The gradual increase in permeability at higher temperatures, though, might be due to higher internal stresses that are released as micro-cracks develop, thus leading to a greater capability of fluid-transfer. Nano-silver impregnation intensified this process.

**Keywords:** Nanoparticles, gas permeability, liquid permeability, perforation plate; wood modification.

### INTRODUCTION

Understanding wood permeability is of vital importance as it has great impact on its utilization in different industries (such as: wood preservation, wood drying, pulp and paper) (Chen *et al.* 1998, Dermoe *et al.* 2012). Gas permeability values in solid woods can be measured with 0.001 second precision Taghiyari *et al.* 2010, Taghiyari and Sarvari 2010, Taghiyari 2011a) and therefore can be helpful in scientific and industrial purposes. The point is that gases usually do not have interaction with cell wall materials while liquids may come to chemical and physical interaction with it, mostly its hydroxyl groups. Besides, most industries in which permeability is important, deals with impregnation of solid woods with liquid or extraction liquids from them. Therefore, finding a correlation between gas and liquid permeabilities can be helpful for industrial decision-making processes.

The effectiveness of nano-silver impregnation has been studied on wood heat-treatment (Taghiyari 2011b), hot-press time in composite boards (Taghiyari *et al.* 2011), fire-retardant properties (Taghiyari 2012a), gas and liquid permeability (Taghiyari 2012b), ice-blasting (Taghiyari *et al.* 2012a) but little or nothing have been done on the subsequent effects of nano-silver impregnation on gas or liquid permeabilities in heat-treated solid woods. The present study is therefore aimed at analyzing the effects of heat treatment on gas and liquid permeabilities of some untreated as well as nanosilver-impregnated native hardwoods, as well as possible relationship between gas and liquid permeabilities.

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The thermal modification of wood has long been recognized as a potentially useful method to improve the dimensional stabilization of wood and increase its decay resistance (Hill 2006). Although it has negative effects on the strength properties of wood, there are some techniques for mitigating these effects (Awoyemi and Westermark 2005, Awoyemi 2007).

Tiemann (1915) was one of the first workers to report on the effect of high-temperature treatment upon the physical properties of wood. He heated air-dry wood in superheated steam at 150°C for 4 hours, which reduced the subsequent moisture sorption by 10–25%, with relatively low reductions in strength found in most cases.

At around 270°C, there is a significant change in the reaction kinetics due to the onset of an exothermic reaction. What is less certain are the temperatures at which the different reactions become dominant (Stamm *et al.* 1946).

Thermal modification is usually carried out at temperatures between 180°C and 260°C. Temperatures lower than 140°C result in only slight changes in material properties and higher temperatures result in unacceptable degradation to the substrate. Studies of the thermal treatment of wood above 300°C are of limited value, due to severe degradation of the material. There is also evidence to show that there is an abrupt change in the degradation kinetics close to this temperature (Elder 1991). Modern thermal modification processes are limited to temperatures no higher than 260°C.

Temperature ranges from 150°C to 230°C are generally used for thermal modifications, because hydrolysis is very slow at lower temperatures. Cellulose degradation begins to occur in the region 210–220°C and becomes predominant at 270°C. A sharp increase in the free-radical content of the wood was also found when the wood was heated at temperatures above 200°C (Garrote *et al.* 1999). The heat-transfer property of nano-silver particles on mechanical properties of heat-treated poplar was also studied (Rassam *et al.* 2012, Taghiyari 2011a, Taghiyari *et al.* 2012abc, Taghiyari 2012ab, Taghiyari and Farajpour 2013).

Heat-treatment has an effect on fluid transfer properties of wood. Reduction of wood swelling with increasing temperature and duration of thermal treatment has often been attributed to hemicelluloses destruction. However, structural modifications and chemical changes of lignin are suggested to be also involved in the process (Repellin and Guyonnet 2005). Furthermore, Borrega and Karenlampi (2010) indicated that reduction in hygroscopicity is not only due to mass loss but another mechanism may also exist. They suggest that this mechanism might be related to irreversible hydrogen bonding in the course of water movements within the pore system of the cell walls.

Little study has been done on the effects of heat-treatment on gas permeability of solid woods. The present study is, therefore, aimed at finding the effects of heat-treatment on gas permeability of several hardwoods. Also, to find the effects of heat-transfer properties of silver nanoparticles, a separate set of specimens were also prepared that were first impregnated with nanosilver suspension before heat treatment was applied to them and untreated specimens.

## MATERIALS AND METHODS

### *Specimen Procurement*

Three hardwoods were chosen based on their importance in various industrial applications in Iran comprised of beech (*Fagus orientalis*), poplar (*Populus nigra*), and hornbeam (*Carpinus betulus*). Specimens of each species were divided into two main groups of heat-treated specimens (HT), and nano-silver-impregnated heat-treated specimens (NSI-HT). To minimize variation between HT and NSI-HT specimens, dowel-shape cylinders of 12 cm in length and 18 mm in diameter were procured in longitudinal direction of trees; two 5 cm long specimens were cut from each 12 cm long specimens; the upper specimens were kept as HT specimens, and the lower specimens were impregnated with nano-silver suspension once their specific gas permeability were measured. For every species, 20 pairs of specimens free from any knots, fissures, and checks were prepared. Furthermore, silicon adhesive was spread all around each specimen to prevent air flow through

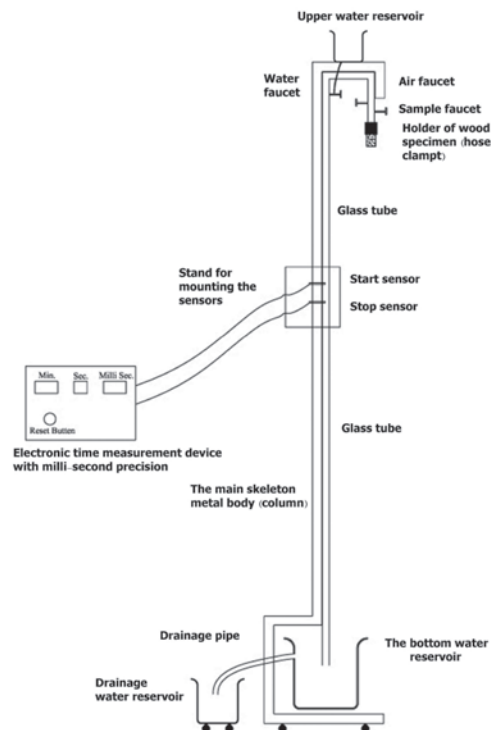
radial and tangential directions. In the meantime, to measure mass loss, some HT and NSI-HT specimens were randomly chosen to be put in oven along with gas permeability specimens. A separate set of specimens with the same size and specifications were procured for mass-loss measurement; only these specimens were not glued with silicon adhesive.

### ***Nano-silver Impregnation Process***

A 200 ppm aqueous dispersion of silver nanoparticles was produced and applied to the specimens using electrochemical technique in collaboration with Jafr Sorkhe Fajr Company. The size range of silver nanoparticles was 20-80 nm. The pH of the suspension was 6-7; two kinds of surfactants (anionic and cationic) were used in the suspension as stabilizer; the concentration of the surfactants was three times the nano-silver particles. Empty-cell impregnation process (Rueping method) was done in a 3 bar pressure vessel by Afshar Wood-Machinery Mfg. Co. (Ltd.). After impregnation, all HT and NSI-HT specimens were kept at room temperature for 4 months. Specific gas permeability values of NSI-HT specimens were again measured before heat-treatment process.

### ***Gas Permeability Measurement***

Longitudinal gas permeability measurement was carried out by an apparatus with milli-second precision designed and built by the author based on the microstructure porosity of wood (Taghiyari and Efhami 2011) (Fig. 1). Measurements were conducted using the falling water volume displacement method instructions (Siau 1971, Taghiyari *et al.* 2010, Dashti *et al.* 2012ab). All gas permeability specimens were cylindrical, 18 mm in diameter and 50 mm long to get the ultimate permeability values (Taghiyari and Sarvari 2010). 20 pairs of specimens were cut randomly at scattered locations from each disk. Connection between the specimen and holder was made fully air-tight. A pressure gauge with milli-bar precision was connected to the whole structure to monitor pressure gradient ( $\Delta P$ ) and vacuum pressure at any particular time as well as height of water column.



**Figure 1.** A schematic drawing of the gas permeability apparatus (USPTO No. US 8,079,249, B2; Pub. No. 2010/0281951 A1) equipped with single-storey milli-second precision electronic time measurement device (Approved by The Iranian Research Organization for Scientific and Technology under certificate No. 47022)

Three measurements were taken for each specimen. Superficial permeability coefficient was then calculated using Siau's equations (Siau 1995) (Equations 1 and 2). The superficial permeability coefficients were then multiplied by the viscosity of air ( $\mu=1.81 \times 10^{-5}$  Pa s) for the calculation of the specific permeability ( $K=k_g \mu$ ).

$$k_g = \frac{V_d CL (P_{atm} - 0.074 \bar{z})}{t A (0.074 \bar{z}) (P_{atm} - 0.037 \bar{z})} \times \frac{0.760 \text{ mHg}}{1.013 \times 10^6 \text{ Pa}} \quad (1)$$

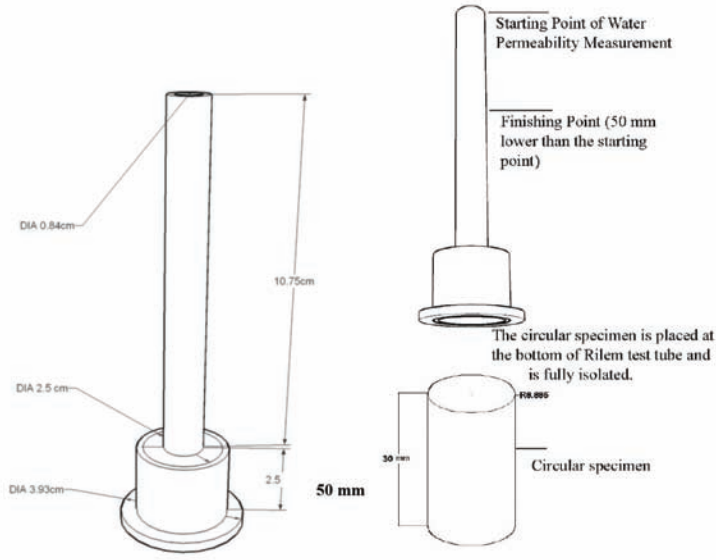
$$C = 1 + \frac{V_r (0.074 \Delta z)}{V_d (P_{atm} - 0.074 \bar{z})} \quad (2)$$

**Where:**

- $k_g$  = longitudinal specific permeability ( $\text{m}^3 \text{m}^{-1}$ )
- $V_d$  =  $\pi r^2 \Delta z$  [ $r$  = radius of measuring tube (m)] ( $\text{m}^3$ )
- $C$  = correction factor for gas expansion as a result of change in static head and viscosity of water.
- $L$  = length of wood specimen (m)
- $P_{atm}$  = atmospheric pressure (m Hg)
- $\bar{z}$  = average height of water over surface of reservoir during period of measurement (m)
- $t$  = time (s)
- $A$  = cross-sectional area of wood specimen ( $\text{m}^2$ )
- $\Delta z$  = change in height of water during time  $t$  (m)
- $V_r$  = total volume of apparatus above point 1 (including volume of hoses) ( $\text{m}^3$ )

### ***Liquid Permeability Measurement***

Liquid permeability was measured using RILEM test tube (Fig. 2). Two times were measured: 1- The time the first drop of water falls off the bottom surface of the 5 cm long specimen; 2- The time the level of water in RILEM tube lowers by 5 cm in the tube (that is, 6.6 CC of water). The test for each specimen was considered done when the liquid level was lowered by 5-cm in the RILEM tube; that is, if the 1st-drop took more that 5-cm lowering time, no 1st-drop time was registered for that individual specimen.



**Figure 2.** Liquid permeability measurement apparatus (RILEM test tube)

### ***Heat-Treatment Process***

All specimens (HT, NSI-HT, and mass-loss) were randomly arranged in an oven to be heated for 24 hours. Heat-treatment was done at consecutive steps of 50, 75, 100, 125, and 150°C. At the final step, specimens were heated for 4 hours at 185°C. After each step, gas permeability of each specimen was measured once the specimens were cooled off. Also, adhesive glue was checked, and if necessary replaced after each step to make sure no air flow through lateral directions.

### ***Mass-Loss Measurement***

Separate specimens with the same size and specifications were prepared from all three species for weight measurement after each heat-treatment steps. Mass-loss specimens of both HT and NSI-HT groups were randomly arranged in the oven along with gas permeability specimens. They were weighed with 0.0001 g digital scale after each heat-treatment step. Mass-loss specimens were free from adhesive glue.

### ***Statistical Analysis***

Statistical analysis was conducted with SAS software program, version 9.1 at 99% level of confidence. Regression analysis and hierarchical cluster, including dendrogram and using Ward methods with squared Euclidean distance intervals, was carried out by SPSS/16.

### RESULTS AND DISCUSSION

Results showed that specific gas permeability in longitudinal direction of all specimens had a sharp increase at two stages: first when NSI-HT specimens were impregnated by the nano-silver suspension, and second, when specimens were first heated at 50°C (Figures 3, 4, and 5). The sharp increase after nano-silver-impregnation process was because some of the extractives were washed out of wood specimens by the empty-cell process. Greatest increase was found in poplar specimens (136%). In beech specimens, however, 4.2% decrease was observed which was due to the settlement of silver nanoparticles within pits and scalariform perforation plates (Fig. 6) (Taghiyari 2012b).

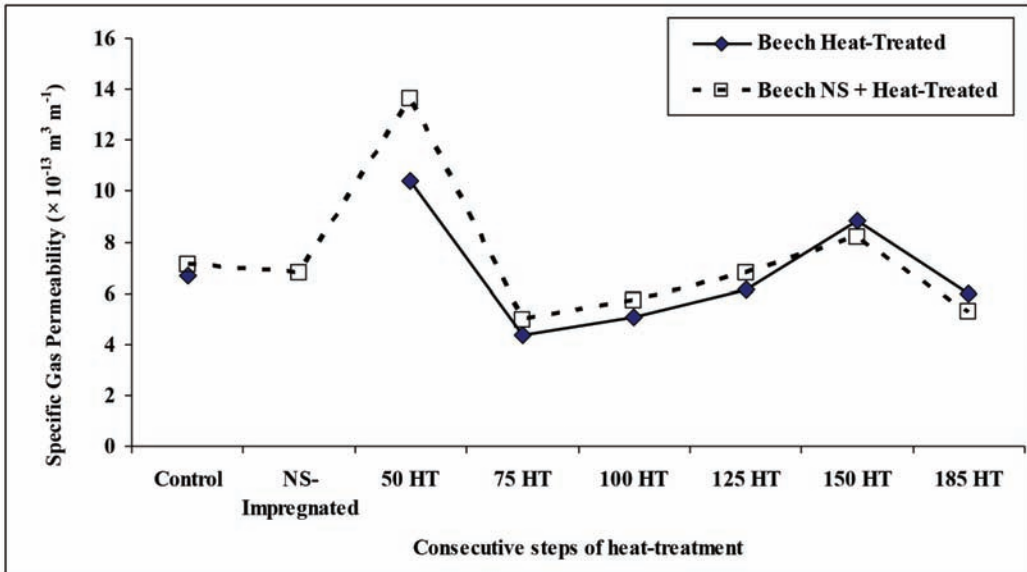


Figure 3. Specific gas permeability values for HT and NSI-HT beech specimen-pairs.

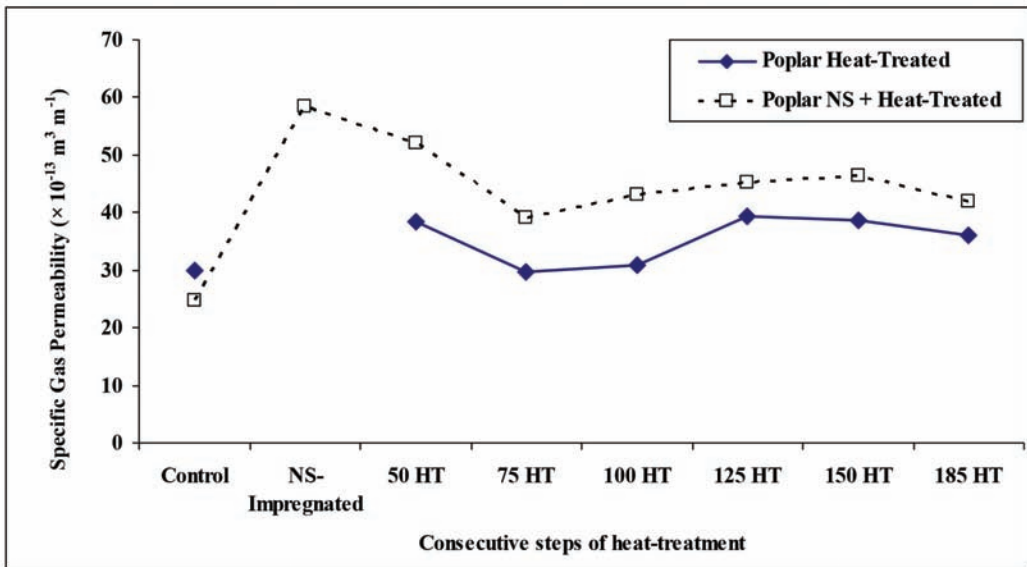


Figure 4. Specific gas permeability values for HT and NSI-HT poplar specimen-pairs.

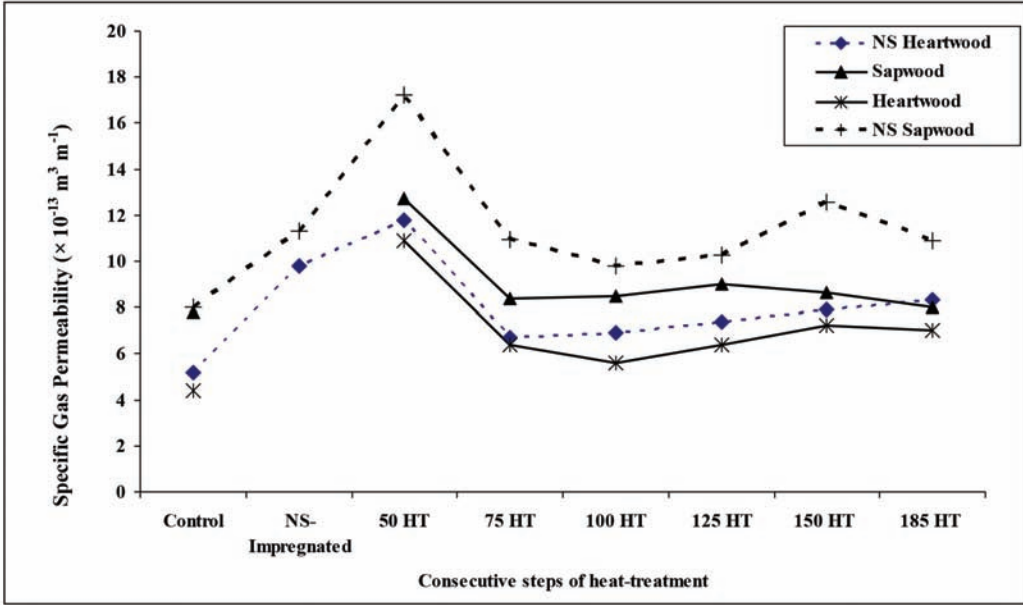


Figure 5. Specific gas permeability values for HT and NSI-HT hornbeam sapwood and heartwood specimen-pairs.

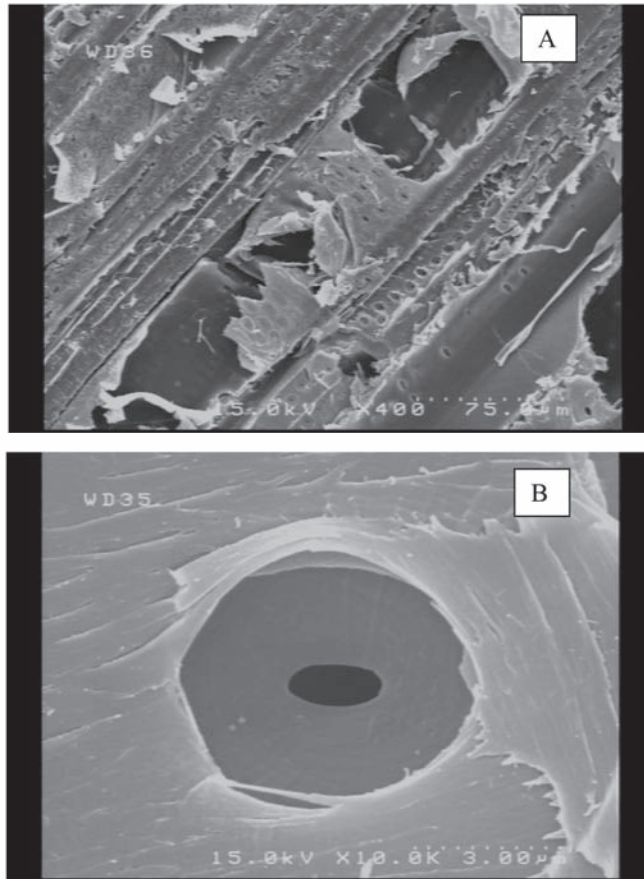


Figure 6. SEM micrographs of beech showing perforation plates in vessels (A), and a pit in the cell wall (B)



All nano-silver-impregnated treatments showed an increase in gas permeability when heated at 50°C, but poplar NSI-HT specimens showed a 10.9% decrease (Fig. 4). The sharp increase at this step could have been due to loss of bound water causing vessel perforations and pits to become wide open by great shrinkage. There was however a sharp decrease in permeability at 75°C in all specimens. Structural modifications and chemical changes of lignin may have played an important role (Repellin and Guyonnet 2005). Furthermore, the irreversible hydrogen bonding in the course of water movements within the pore system also may have affected the fluid transfer process (Borrega and Karenhampi 2010). These processes may have occurred at lower temperatures in poplar, with the lowest density in the present study, causing the above mentioned slight decrease at 50°C. Permeability values were then steadily increased in the further heat-treatment steps after 75°C, and up-to 150°C. In these steps, higher temperatures increase high internal stresses that are released as cracks (Oltean *et al.* 2007). These cracks facilitated fluid transfer process through the porous material causing the gradual increase in permeability.

Heat-treatment at the final step of 185°C made permeability values in nearly all species decrease (Fig. 5). Heat-conductivity of nano-silver particles made these processes intensified. Figures 7 and 8 show that heat-treated sapwood specimens has a great mass loss when they were heated at 150 and 185°C. Similarly, a decreasing trend in gas permeability value may be observed in figure 5. This may clearly imply that every mass loss should not necessarily end up in increase in permeability.

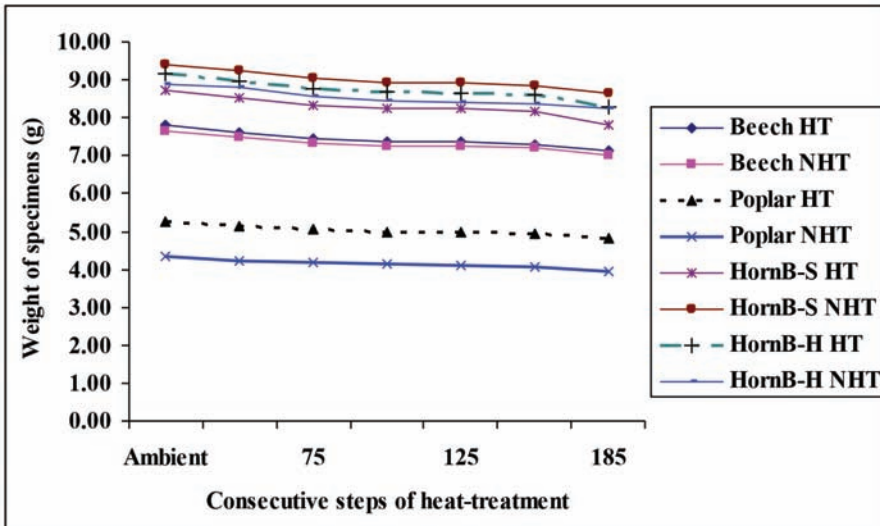


Figure 7. Weights of specimens at different steps of heat treatment (g)



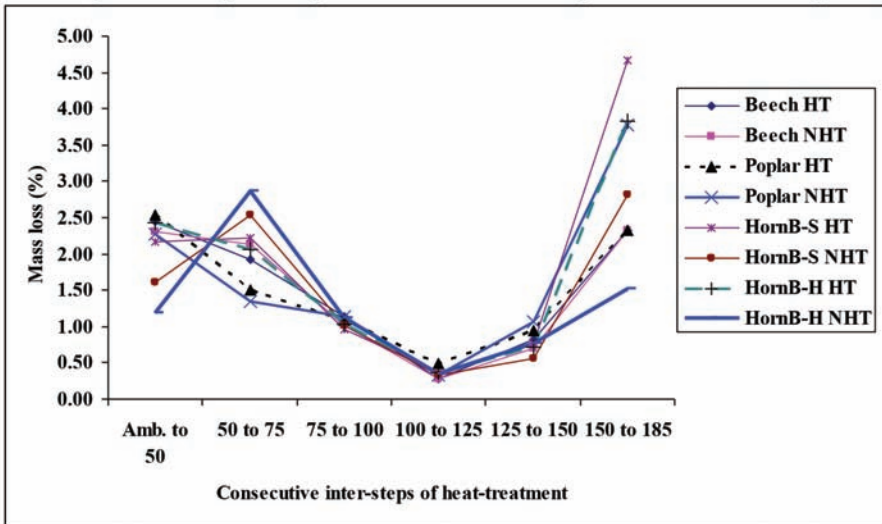


Figure 8. Percent of mass loss between different steps of heat treatment

Liquid permeability times (s) were measured once the specimens were heat-treated and finished with gas permeability measurement. Maximum liquid (water) permeability times were found in NSI-HT poplar specimens. Regression analysis between specific gas permeability time with the two liquid permeability times, after the heat-treatment, showed high relationship between gas permeability and 5-cm lowering times (Table 1). Cluster analysis of all the treatments based on gas and liquid permeability time values showed great similarity between HT and NSHT beech and poplar specimens, as well as HT and NSHT poplar specimens.

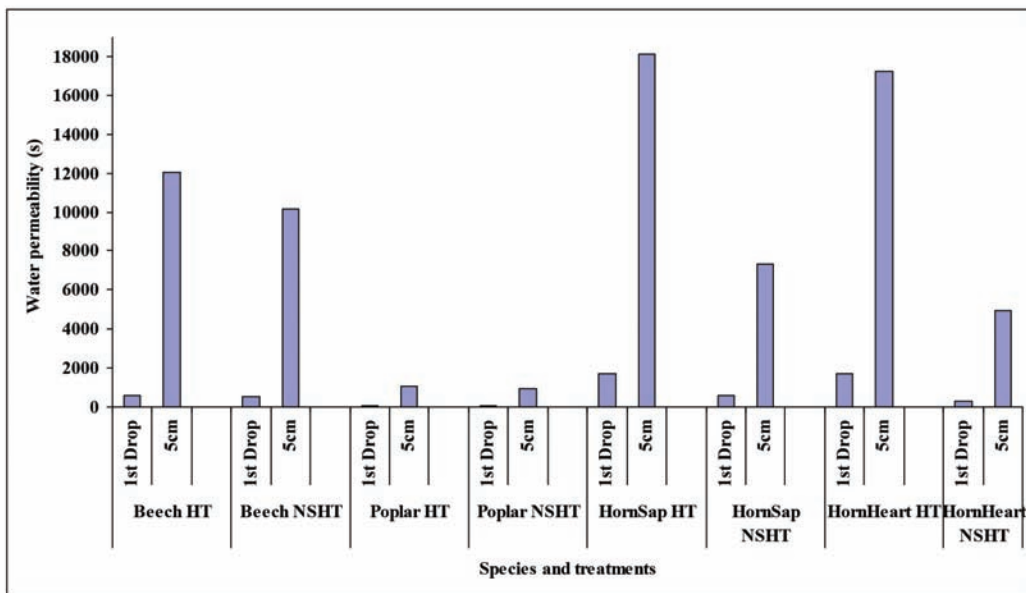


Figure 9. Liquid permeability times (s) of the different treatments of the present study (HT=Heat-Treated; NSI-HT=Nano-Silver-Impregnated Heat-Treated) after 185°C heat treatment

**Table 1.** Regressions analysis results for correlations between gas with liquid permeability (1st drop as well as 5-cm lowering) times after heat-treatment at 185°C.

Species and Treatments	Poplar (HT)	Poplar (NSI-HT)	Beech (HT)	Beech (NSI-HT)	Hornbeam Sapwood (HT)	Hornbeam Sapwood (NSI-HT)	Hornbeam Heartwood (HT)	Hornbeam Heartwood (NSI-HT)
R square	.960	.998		1.000	.281	.240		.396
(Gas – 1 <sup>st</sup> drop)	** (+)	** (+)	-----	** (+)	NS (-)	NS (-)	-----	NS (+)
R square	.960	.998	.963	.970	.608	.124	.858	.946
(Gas – 5 cm)	** (+)	** (+)	** (+)	** (+)	** (+)	NS (+)	NS (+)	** (+)

\*\* Statistically significant at 1 % confidence level.

\* Statistically significant at 5 % confidence level.

**HT** Heat-Treated.

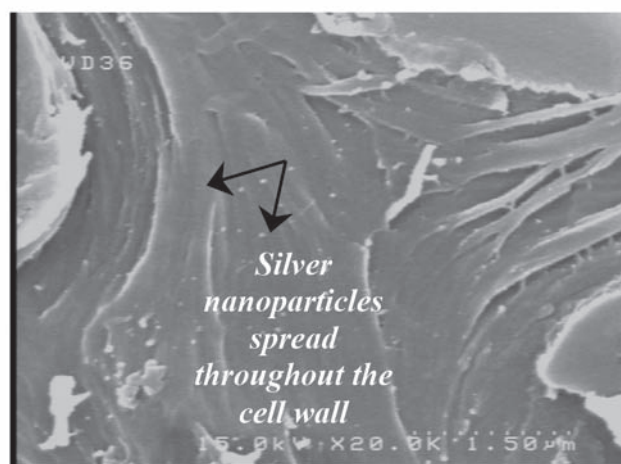
**NSI-HT** Nano-Silver-Impregnated Heat-Treated.

**NS** Not Significant.

( ) positive (+) or negative (-) correlation

Mass loss of all species and treatments in the present study showed constant decrease from ambient temperature up-to 185°C (Fig. 7). Calculation of the percent of mass loss though showed that the maximum mass loss took place at 3 steps: 1- when specimens were first heated at 50°C, 2- when they were heated at 75°C, and 3- when they were heated at 185°C (Fig. 8). Apart from the first step when the specimens were heated from ambient temperature to 50°C causing a sharp increase in permeability, the other two peaks of mass loss shown in figure 8 (when heated at 75°C and 185°C) resulted in considerable decrease in gas permeability (Figures 3, 4, and 5).

SEM micrographs showed spread of nano-silver particles over the surface area of specimens. The effects of nano-silver particles were quite different at lower temperatures in comparison with higher temperatures. At lower temperatures of less than 125°C, NS particles accelerate the process of heat-transfer to the inner parts of specimens and therefore evaporation of volatiles was more easily done also in the core of specimens. At temperatures more than 125°C, on the other hand, heat-conductivity property of NS-particles made concentration of heat to less degrees on the surface layer of specimens and therefore lower mass-loss could be expected in the specimens (Fig. 8).



**Figure 10.** SEM micrograph of nano-silver-impregnated beech showing silver nanoparticles all over the cell wall

Oltean *et al.* (2007) indicated that only a few papers dealt with the influence of temperature during drying on both the mechanical properties of wood and on the occurrence of cracks. Gas permeability may be considered a suitable means to estimate the extent of degradation of wood components and cracking. At lower temperatures, when gas permeability increased, possibly significant degradation of wood components occurred. At higher temperatures when permeability showed a gradual increase, the internal stresses might have resulted in (micro-) cracks within cell walls.

Based on the above mentioned findings on the effects of heat-treatment on permeability, when wood specimens are to be dried for industrial or scientific purposes for further processes, it would be more logical to heat them at temperatures less than 60°C even if that means longer times of keeping the samples in oven or kiln because this would keep specimens on the safe edge of not being irreversibly changed in their structure and chemical components, as well as permeability and impregnability properties.

## CONCLUSIONS

Heat-treatment affects permeability significantly due to loss of bound water and irreversible hydrogen bonding in the course of water movements within the vessel perforations and pore system of cell walls.

Heat-conductivity of nano-silver particles intensifies the effects of heat-treatment.

For heat-treated specimens, liquid permeability of 5-cm lowering time gives a better estimate of specific gas permeability value.

In processes where the permeability of solid woods are important, the wood should not be heated at temperatures higher than 60°C to prevent irreversible structural or chemical changes in the cell wall components.

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