Modification of Domestic Timbers by Impregnation using Supercritical Carbon Dioxide - A Comparison

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ABSTRACT

Domestic timbers are often not suited for construction trade applications because of low dimensional stability, low durability or low mechanical strength and are squeezed out of the market by tropical lumbers or other materials (such as plastics or metals). Therefore it is of considerable interest to improve their characteristic properties in order to open new potential markets. One possibility to accomplish this aim is the impregnation of wood with silicon containing substances such as silane based nanosols or silicones. During this process, the silicon compounds are deposited in the lumina and the cell walls causing a change of wood properties. In many important domestic wood species, particularly in spruce wood, conventional procedures using water or organic solvents achieve only superficial penetration with depths of only a few millimetres. Thus a solvent is needed, which enables the complete penetration of the wood without damaging its structure. It was previously shown, that supercritical impregnation with saline and silicone compounds resulted in reduced water uptake and complete penetration of the specimens. Thus, the objective of the investigations described here is to verify the suitability of supercritical carbon dioxide ($scCO_2$) as solvent for the impregnation process and to study the achieved improvement of wood properties. In comparison to the classical vacuum-pressure impregnation the supercritical infiltration shows the same load (weight percentage gain), but in shorter time and with less amount of chemicals used in the treatment process. Another advantage of supercritical impregnation is that no additional drying after the process is needed.

INTRODUCTION

Carbon dioxide becomes a supercritical fluid at a temperature above 32 °C and a pressure exceeding 7.2 MPa (72 bar, 1044 psi), a physical state which has the properties of either gas or liquid. Under pressures from 10.0 to 40.0 MPa and at temperatures of 35 to 70 °C supercritical carbon dioxide can be used as a solvent for non-polar substances, and is applied in established processes like the recovery of natural flavours or the decaffeination of coffee and tea. The process temperature does not need to be higher than 40 °C. Because of its low surface tension, supercritical CO_2 intrudes deeply into the wood without swelling it.

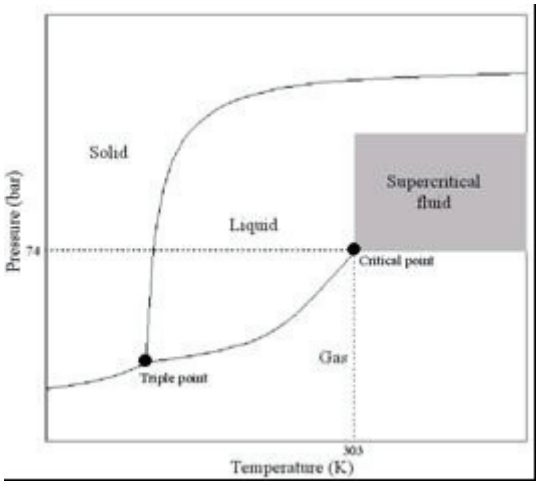


Figure 1: Phase behaviour of carbon dioxide

Any mechanical damage is minimised if care is taken during the pressure built-up and pressure release during the impregnation. Another advantage of supercritical carbon dioxide is that supercritical carbon dioxide becomes a gas after pressure release and there is no solvent left in the wood.

EXPERIMENTAL

For impregnation with supercritical carbon dioxide at Fraunhofer UMSICHT the pilot plant station depicted in figure 2 was used. First the carbon dioxide from the storage tank (D1) was cooled to get liquefied and pump able. After that the carbon dioxide was heated to up to 40 °C and pressurized up to 15 or 20 MPa. It flows than through the vessel (V1) with the impregnation agent (silicone or silica sol) to get into vessel A1. When impregnation time was over the pilot plant station was depressurized and the impregnation agent precipitated on and in the wood.

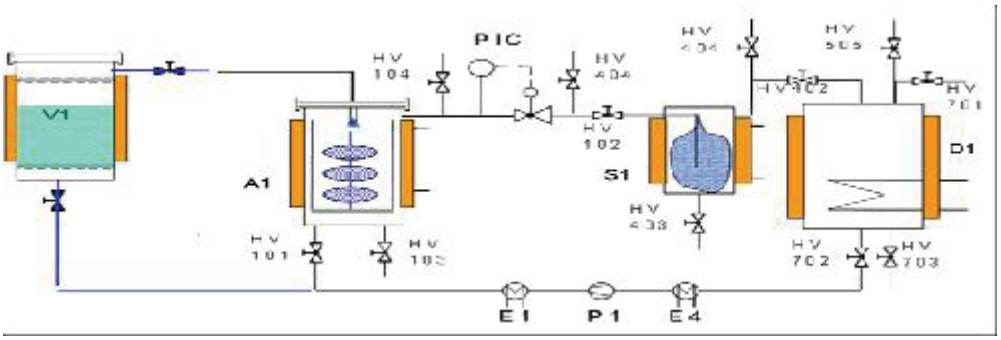


Figure 2: Pilot plant station for the impregnation used at Fraunhofer UMSICHT

To measure and compare the intrusion depth regarding to the direction (longitudinal, radial, tangential), five sides of the specimen were sealed with a specific varnish as depicted in figure 3.

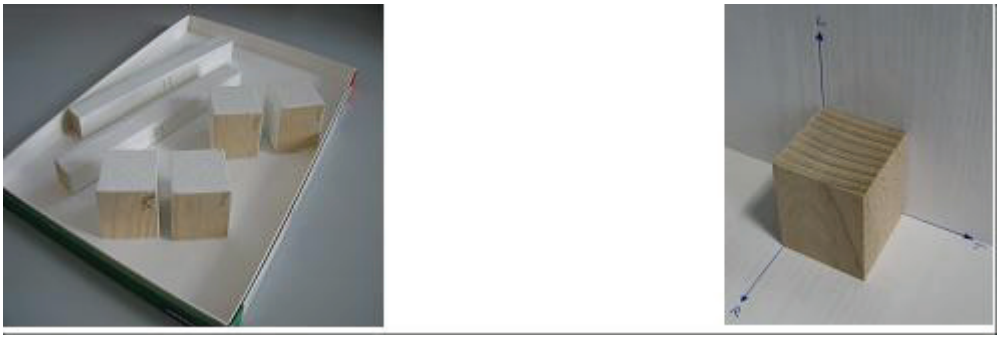


Figure 3: Specimen for determining intrusion depth

In every experiment the specimen were arranged in the same way (figure 4). This makes the interpretation of intrusion easier and the results more comparable.

Figure 4: Beech specimen before and after $scCO_2$ impregnation

Table 1: Dimensions of the specimen

Sample	Dimensions [mm]
Longitudinal	20*20*200
Radial	40*40*40
Tangential	40*40*40

The impregnation process described above and depicted in figure 2 was not suitable for the impregnation with the silica sol. The silica sol was dispersed in ethanol and with the supercritical carbon dioxide a so called gas-antisolvent-reaction occurs and some particles precipitated in vessel V1 before contacting the wood in A1.

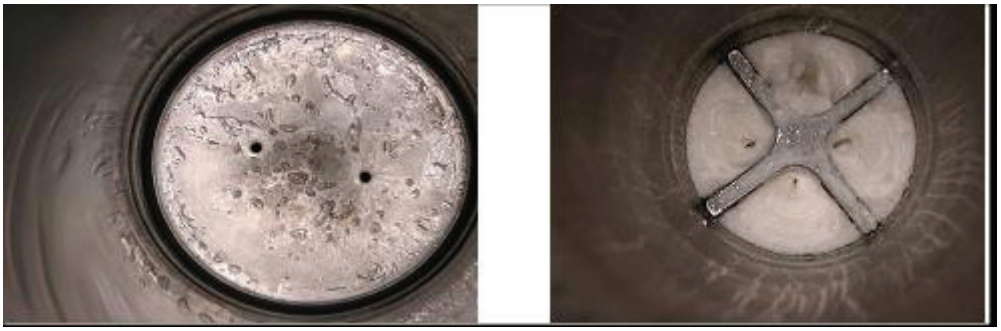


Figure 5: Precipitated silica sol particles in Vessel V1

To solve this problem variations of impregnation processes were tested. First trial was to spray the silica sol overhead into the vessel with parallel flow to the carbon dioxide. The second trial was to put the wooden specimen between two sieves and overlaying the chemicals (figure 6).

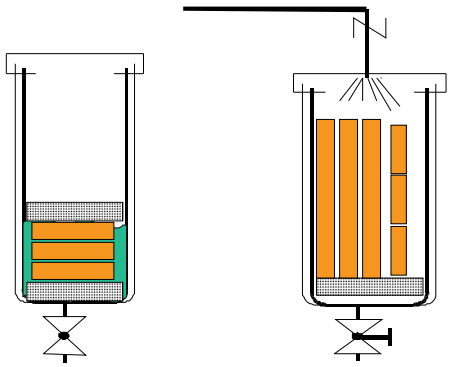
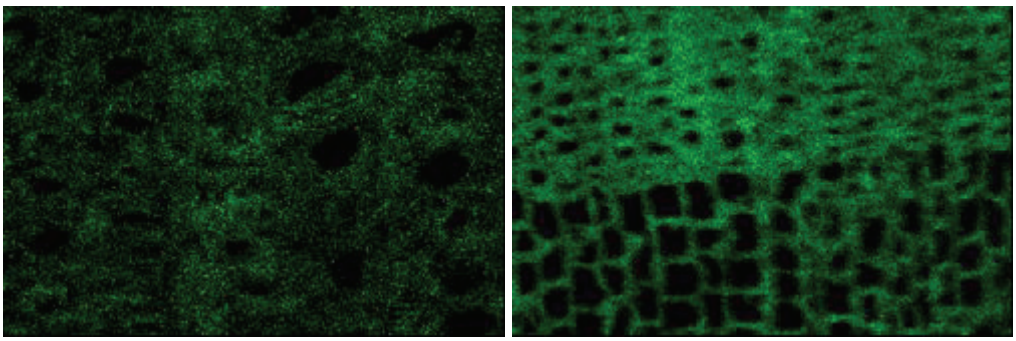


Figure 6: Variation of impregnation procedures

Depending on the amount of sol particles in the dispersion the procedure has to be adapted.

RESULTS AND DISCUSSION

There are two main results. It could be shown that Silica was precipitated in the cell wall of the wood. And on the other hand that supercritical carbon dioxide penetrates through the wood completely.



A B

Figure 7: SEM-EDX Si-mapping images:: Silicon oil in Scots pine with SCC treatment (A); Siliconoil M50 in Scots pine with vacuum pressure treatment(B)

The intrusion was measured with FT-IR-spectroscopy (peak area). As depicted in figure 8 there are two characteristic bands of Si which can be used for the calculation. The peak area of the normalised spectra gives a hint for the uptake of the chemical.

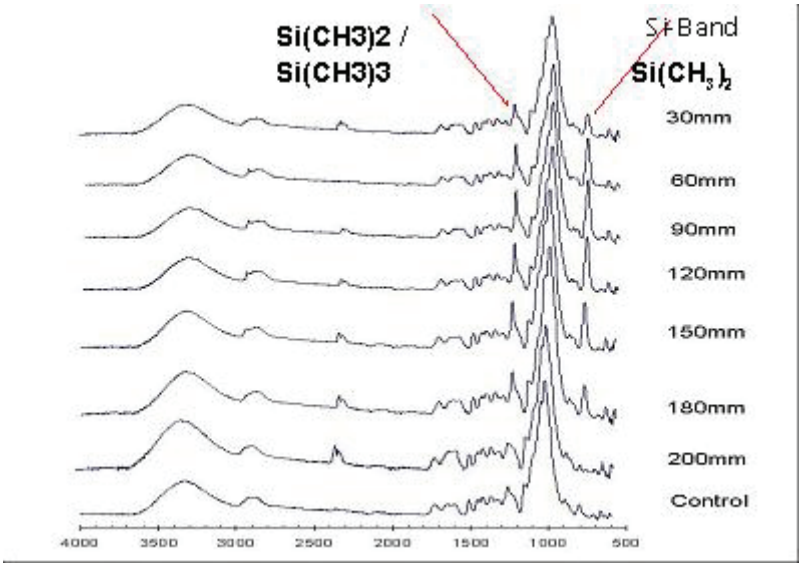


Figure 8: Spectra of longitudinal intrusion measurement

In comparison to the vacuum-pressure method the amount of uptake was less. But the intrusion of supercritical carbon dioxide is very good with shorter time of impregnation. As an example of intrusion depth the following picture shows the longitudinal intrusion of a silicon oil. The figure depicts also that with supercritical carbon dioxide a real intrusion profile is recognizable and not with vacuum pressure method. For now not known reasons the amount of chemical is increasing to the end of the specimen. This may be caused of little damages of the specimens varnish and the silicone could penetrate through the cracks. This has to be proven.

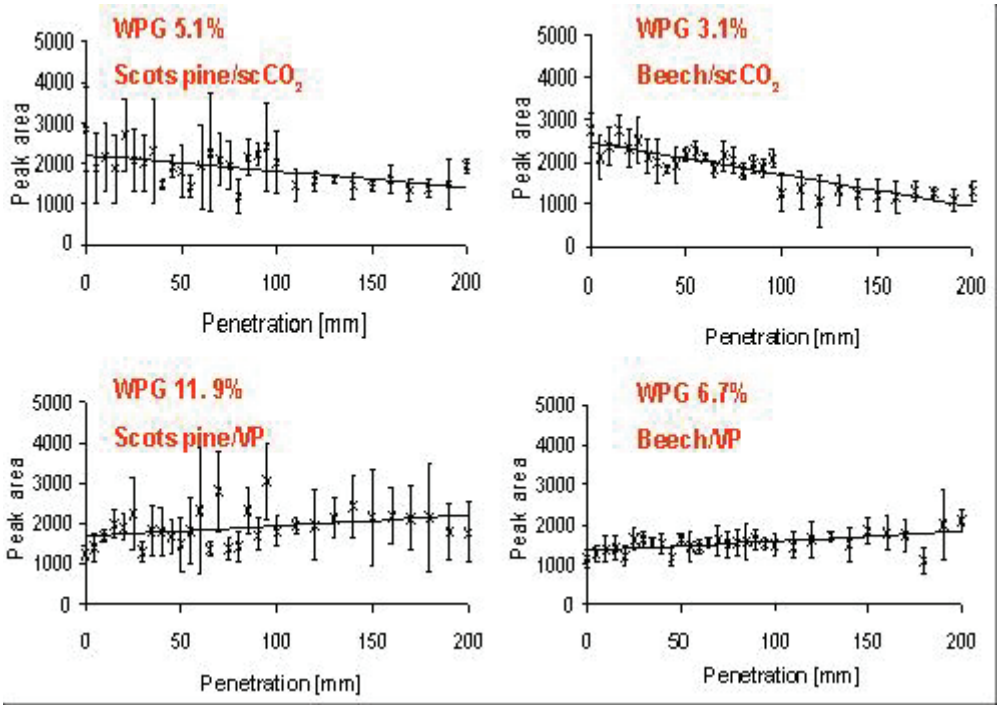


Figure 9: Longitudinal penetration

The weight percentage gain was calculated regarding to equitation 1.

$$WPG\ (\%) = ((M_1 - M_2)/M_1) \times 100 \qquad (1)$$

CONCLUSIONS

On the one hand it could be shown, that impregnation of wood with supercritical carbon dioxide is suitable. But the impregnation process has to be revised. And on the other hand that supercritical carbon dioxide penetrates through the wood completely. In comparison to the vacuum-pressure method the amount of uptake was low. But the intrusion of supercritical carbon dioxide is very good with shorter time of impregnation. Further investigations are still running.

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