



## Translucent UV light absorbant coatings for wood

A. Ullrich, B. Reinhard, M. Wittmar



### Abstract

Efficient protection of fast growing domestic woods against the effects of weathering and UV irradiation is of increased significance to obtain improved dimensional stability and durability comparable to tropical wood. To advance the properties of these woods, a combined treatment process was developed. First, samples of pine sapwood were impregnated by project partners in the University of Göttingen to improve the dimensional stability and to avoid fungal infection of the wood, then a coating containing zinc oxide nanoparticles was applied on the impregnated wood samples at INM. Due to the UV light absorption of zinc oxide nanoparticles, the coated wood substrates are distinguished by enhanced colour stability. Together with the impregnation the coating prevents wood from deterioration by UV-irradiation and changing climates while retaining optical appearance.

The water-repellent properties of the coatings interfered with the impregnation and reduced the ingress of water. This resulted in a stabilising reduction of the swelling and shrinking and avoided the formation of cracks in both wood and coating. The improved characteristics were demonstrated by artificial weathering. A prototype of an impregnated and coated window has been manufactured and is currently exposed to outdoor weathering.

### Introduction

European wood species show considerably poorer durability and dimensional stability in applications in outskirts areas compared to tropical wood species.

Therefore, the aim of this project was the development of a novel combined treatment for woods used for window constructions and in the structural-facings sector. The first step in this two-stage process was pressure impregnation to chemically modify the wood and lead to enhanced solidness and stability. The nanoparticulate composite coating was applied in the second step. It should ensure improved surface characteristics such as hydrophobic properties and absorption of UV light.

The impregnations were performed at the Department for Wood Biology and Wood Technology at the University of Göttingen [1-3]. The main task of the INM was the development of a functional coating providing improved water repellent properties and UV resistance.

Hydrophobic properties lead to decreased moisture uptake and consequently improved dimensional stability. The absorption of UV light should prevent photo-induced degradation of wood due to the decay of the lignin and the elution of its decomposition products. Further requirements for the coating included good adhesion on impregnated wood, permeability to water vapour, and additionally, boehmite particles should result in improved abrasiveness.

## Experimental

High quality samples of pine sapwood with a size of 75 x 40 x 10 mm<sup>3</sup> were used for the experiments. The samples were impregnated at the University of Göttingen.

The coating consisted of an inorganic-organic hybrid matrix structure with embedded nanoparticles, including nano zinc oxide as an UV light absorbing component. The matrix was built of phenyltriethoxysilane, tetraethoxysilane and (3-glycidoxypropyl)-trialkoxysilanes, crosslinked via bisphenol A. The zinc oxide nanoparticles (ethanolic suspension with ZnO-content of 32 % by weight) were provided by Bühler-Partec GmbH, Saarbrücken. The coatings were applied using dip-, roll- or spray-techniques and cured at 120 °C in a furnace.

The properties of the coatings were characterized using artificial weathering [4] in a UV cabinet (alternating condensation atmosphere and UV irradiation) and a suntest chamber. Evaluation of the samples and especially the surfaces by crack characterisation was performed at regular intervals. The surface colour and brightness was determined with a Micro Color device according to the Commission International de l'Eclairage (CIE) based on the three dimensional LAB colour space [5]. Brightness is represented by the L axis from black (0 %) to white (100 %). Chromaticity is characterized by the two coordinates a (representing the red-green-axis) and b (representing the blue-yellow-axis). Chroma can be calculated according to the following equation:

$$C^* = \sqrt{(a^*)^2 + (b^*)^2}$$

Volume changes due to absorption of moisture were determined using a climate chamber with standard climatic conditions (20 °C, 65 % relative humidity) and humid climatic conditions (20 °C, 90 % relative humidity) [6]. Weight and

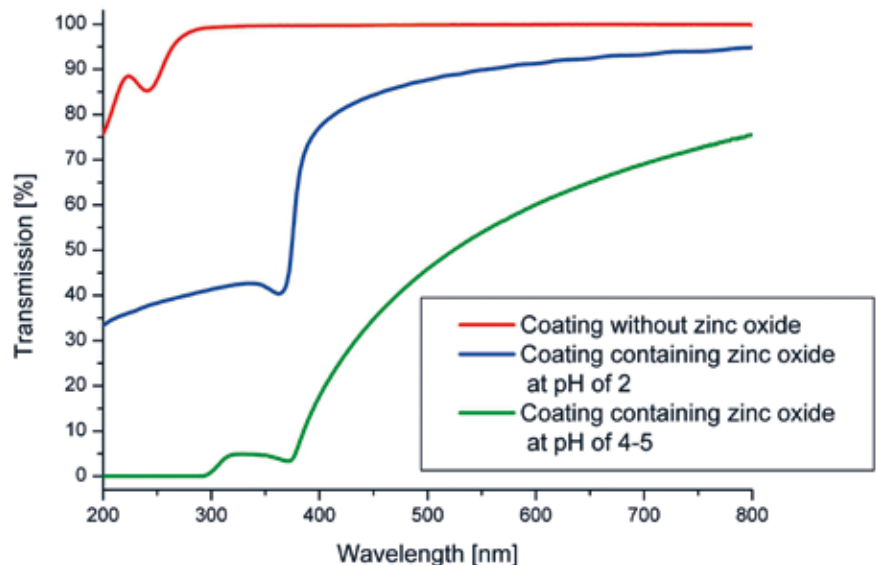


Figure 1: Transmission spectra of coatings on quartz glass sides.

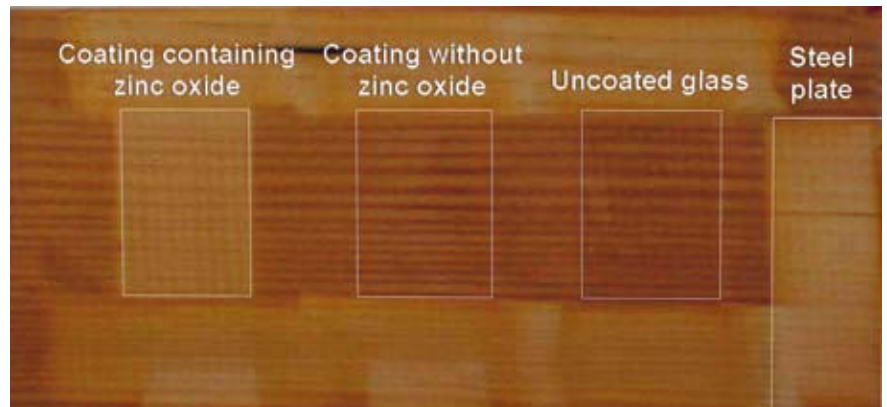


Figure 2: Wood sample after sun test; the areas were covered with different plates and glass sides during the test.



dimension gains were measured after reaching constant weight.

The abrasion resistance was determined using taber abrader tests [7]. The coating was applied to 10 x 10 cm glass plates and subjected to the wearing stress of two abrading arms each loaded with 500 g pressure. The loss of weight was measured after 1000 cycles.

### Results and discussion

UV light absorption by the zinc oxide nanoparticles was tested by UV spectroscopy and using a suntest chamber. A 20 micron thick coating was applied on quartz glass sides. The transmission of UV light was measured with an UV spectrometer (Figure 1).

Within a wavelength range of 800 to 200 nm, 100 % transmission of light was observed without any UV absorbing agent. Using a sol containing zinc oxide particles, at a pH value of 2 due to the acidic reaction catalyst, a loss of transmission beginning at approximately 400 nm was observed. However transmission of UV light remained at almost 40 %. This reduction was due to the low pH value partly dissolving the zinc oxide. Setting the pH between 5 and 6 resulted in slowly decreasing absorption down to 5 % at 400 nm and finally to non measurable transmission at 300 nm.

In further experiments in the sun test chamber only slight browning was observed on areas covered with coated quartz glass. Figure 2 shows a sample after 8 weeks of irradiation. Two further areas

were covered, one with uncoated glass and the other with a coating without zinc oxide particles. As expected, these areas showed browning similar to uncovered areas.

Lightness values (Table 1) confirmed the observations. Areas covered with zinc oxide containing coatings had darkened less compared to the areas without such a coating. Although, compared to the areas covered with steel a slight darkening was observed.

Covering	L	a	b	C
Initial values	83.6	5.7	21.0	21.8
Steel plate	78.4	9.3	26.8	28.4
Glass without coating	57.0	18.9	33.7	38.6
Coating without Zinc oxide nanoparticles	56.9	17.8	31.2	35.9
Coating including zinc oxide nanoparticles	67.7	13.9	32.0	34.9

Table 1: Colour values after 8 weeks of UV irradiation in sun test chamber.

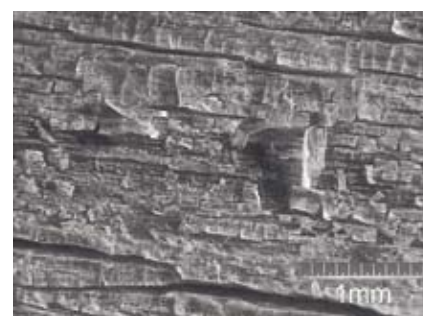


Figure 3: Light microscopic image of an inflexible coating after four weeks of artificial weathering.

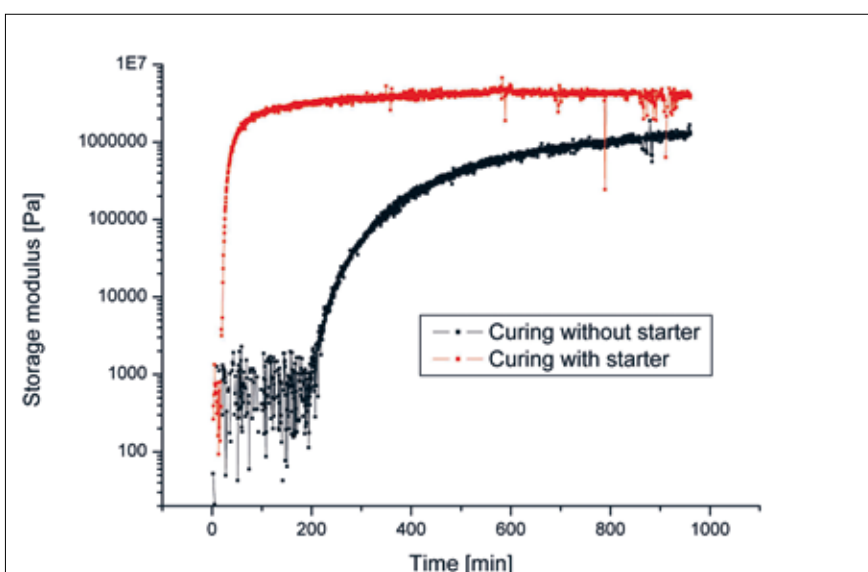


Figure 4: Dynamic mechanical thermal analysis of the coating sol with and without the addition of catalyst.

The investigation of treated and untreated wood specimens in a climate chamber using standard climate followed by humid climate showed a remarkable volume change even when using impregnated specimens (Table 2). The observed values scattered over a relatively wide range what can be attributed to the fact that the arboreal growth and the density have remarkable influence on the characteristics of wood.

	Volume gain [%]	Water uptake [mg/mm <sup>3</sup> ]
Untreated wood	5.0 – 6.7	41.5 – 46.1
Impregnated wood	2.9 – 3.1	27.9 – 34.3

Table 2: Volume gain and water uptake of wood caused by the change from standard climate conditions to humid climate conditions; all values are related to the volume at standard climate.

Therefore, it was necessary to develop flexible coatings to avoid crack formation and flaking of the coating as it was observed for coatings with a pure inorganic network (Figure 3).

Silanes with reactive organic groups in their side chains should allow organic crosslinking to improve flexibility. To this end, (3-glycidoxypropyl)-trialkoxysilanes and bisphenol A were used to crosslink the silanes via polymerisation of epoxide rings.

The reaction was initiated with a catalyst. In Figure 4, the curing behaviour with and without the addition of starter was measured by isothermal dynamic mechanical thermal analysis (DMTA) at 120 °C. The DMTA allows the characterization of the curing progress and the estimation of the necessary curing time.

Without the catalyst the reaction did not start within three hours whereas the reaction with the catalyst was initiated immediately and reached a stable state after a short time indicating the completion of the reaction.

The coatings were manufactured by dip-, roll- or spray-techniques. To receive homogeneous and plane layers, the coatings were slightly abraded after curing followed by another coating step. The procedure was repeated several times if necessary.

The coatings showed excellent abrasion resistance as shown in Table 3.

	Weight loss [mg]
Coating without boehmite particles	21
Coating including boehmite particles	8

Table 3: Average values of weight loss as consequence of taber abrader tests.

Without the use of boehmite particles the coating was partly worn after 1000 test cycles (Figure 5, top), whereas boehmite containing coatings still showed complete surfaces without crack formation (Figure 5, bottom).

The coated specimens as well as some uncoated and non-impregnated samples were tested by artificial weathering using a Quick-UV-test (QUV) cabinet.

As a consequence of the treatment with silanes and adjacent heat curing, lightening values of the impregnated wood displayed a darker surface colour than untreated specimens before the weathering test. After one week of artificial weather-

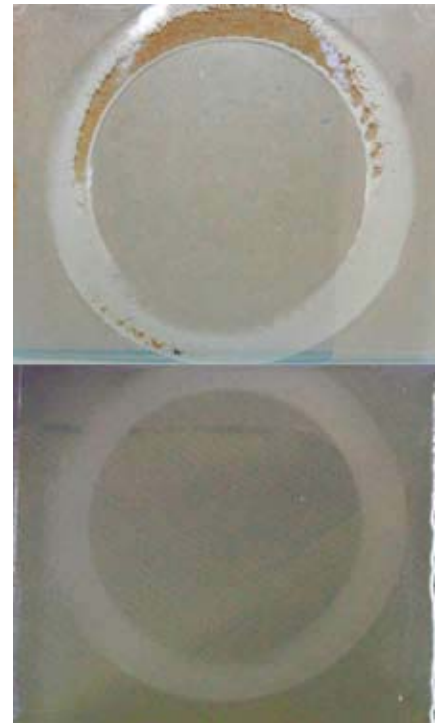


Figure 5: Coating without boehmite particles (top) and with boehmite particles (bottom) after 1000 cycles taber abrader test.



ing, completely untreated controls had darkened due to lignin oxidation and formation of chromophoric degradation products. Continuation of the experiment led to colour lightening caused by the wash-out of these degradation products (Figure 6 left).

After four weeks of artificial weathering impregnated wood without any coating showed internal cracks due to water infiltration. The colour of the wood lightened according to the wash-out of degraded lignin as already observed in the test of untreated wood when continuing the test with uncoated wood for up to 12 weeks.

Wood with inflexible coatings without organic crosslinking showed cracks and flaking after just 4 weeks of weathering as shown in Figure 3. However, no surface crack formation could be observed after 12 weeks using flexible coatings containing zinc oxide nanoparticles. Furthermore, enhanced colour stability was observed as no lignin had been washed out (Figure 6 right). An impregnated and coated window has been built and outdoor weathering experiments are currently underway.

## Conclusions

In summary, a translucent coating which provides permanent protection against weathering and UV light degradation was developed. Integrated nano zinc oxide particles provided nearly complete UV protection.

The coating showed excellent adhesion on untreated and on impregnated wood,

optical appearance, high abrasion resistance, UV light absorption and no crack formation due to weathering.

To compensate for the dimensional instabilities of weather exposed wood, it was necessary to build up a more flexible organic-inorganic hybrid matrix using (3-glycidoxypropyl)-trialkoxysilanes. This flexibility was adequate to equalize the dimensional fluctuations due to moisture absorption and to avoid crack formation. No lightening of the surface colour caused by the wash-out of degraded lignin could be observed after 12 weeks of artificial weathering.

## Acknowledgments

The German Federal Ministry of Economics and Technology (BMWi) and the "Arbeitsgemeinschaft industrieller Forschungsvereinigungen 'Otto von Guericke' e.V. (AiF)" is gratefully acknowledged for financial support within the program for the promotion of "Industrial Joint Research (IGF)" (Research project V-Nr. 14897 N of the German Society for Wood Research (DGfH e. V.)).

We express our gratitude to Bühler Partec GmbH, Saarbrücken, for providing suspensions with ZnO nanoparticles.

## References

- [1] S. Donath, H. Militz, C. Mai, Holz Roh. Werkst., 2007, 65, 35-42.
- [2] C. Mai, H. Militz, Wood Sci. Technol., 2004, 37, 339-348.

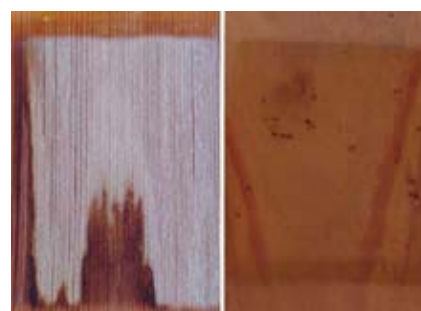


Figure 6: Untreated wood (left side) and impregnated and coated wood (right side) after 12 weeks of artificial weathering.

---

[3] C. Mai, H. Miltz, Wood Sci. Technol., 2004, 37, 453-461.

[4] modified DIN EN 927-6: Exposure of wood coatings to artificial weathering using fluorescent UV lamps and water.

[5] DIN 6174: Colorimetric evaluation of colour coordinates and colour differences according to the approximately CIELAB colour space.

[6] DIN 52184: Determination of swelling and shrinkage.

[7] DIN 53754: Determination of abrasion, abrasive disk method.