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Method for determining liquid water flow in wood during drying using a fluorescent dye tracer

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Abstract A novel procedure for the evaluation of liquid water flow during the drying of wood is described. The method is based on the movement of a fluorescent dye solution in liquid water. The addition of the dye solution produced a clear trace inside the wood, the length and direction of which were directly related to the flow of liquid water. A comparison of the results obtained with image analysis using UV illumination and measurement of the reflectance spectrum of wood showed that image analysis is appropriate and accurate enough for the determination of the trace of the dye solution. The trace of the dye solution can be determined from the images of the cross sectional samples of wood in all three main directions.

Eine Methode zur Bestimmung des Fließverhaltens von flüssigem Wasser im Holz während des Trocknens unter Anwendung eines fluoreszierenden Farbstofftracers

Zusammenfassung Ein neues Verfahren zur Bestimmung des Fließverhaltens von flüssigem Wasser während der Holz Trocknung wird beschrieben. Das Verfahren beruht auf der Bewegung von fluoreszierender Farbstofflösung im Wasser. Die Zugabe der Farbstofflösung erzeugte im Holz eine gut sichtbare Spur, dessen Länge und Richtung direkt von der Bewegung des Wassers abhing. Ein Vergleich der Ergebnisse von Bildanalyse und der Messung des Reflektionsspektrums des Holzes zeigte, dass die Bildanalyse zur Erfassung der Spur der Farbstofflösung ausreichend genau ist. Anhand von Bildern von Querschnittsholzproben ist es möglich, die Farbstoffspur in allen drei Hauptrichtungen zu messen.

1 Introduction

Liquid water flow in wood is an important phenomenon both in living trees and freshly sawn wood raw material. Along with water transportation, nutrients and extractives are also transported in wood. Regarding the drying process of wood above wood fiber saturation point (FSP), a remarkable portion of moisture is removed from the wood by the liquid water flow. The pathways of liquid water flow may differ between wood species, owing to differences in the anatomical structure of wood. In addition, the liquid water flow is affected by moisture content changes, differences in wood density, and abnormalities in wood. Wood cells are typically elongated in the axial direction for support and water transportation. This means that also during the drying process, the resistance for water movement is greater in the radial and tangential directions than in the axial direction. In this study, a novel method to analyze the pathways of liquid water during drying is described.

The three-dimensional behavior of moisture in wood during the drying process can be analyzed nondestructively using modern technology, e.g. by means of X-ray computed tomography scanner (Scheepers et al. 2007) or the NMR technique (Almeida et al. 2008). Mathematical approaches based on physical principles, combined with experimental research have also been developed to model the mechanisms involved in the drying of wood (e.g. Booker 1996; Salin 2006). However, these methods do not unambiguously reveal the pathways of water out of the wood sample during drying. More detailed information on the pathways of water is needed, especially when the movement of extractives along with water is considered.

The importance of knowledge of the pathways of liquid water flow is emphasized, e.g., when the discolouration of wood induced by drying is considered. Artificial drying may involve accumulation of extractives at a certain layer of a sawn timber piece, where the liquid water flow has ceased as a result of decreasing moisture content. The enrichment of wood extractives near the surface of sawn timber during drying has been detected with radiata pine (*Pinus radiata*) (Kreber et al. 1998) and Scots pine (*Pinus sylvestris*) (Terziev 1995). Adequate conditions for the polymerization of extractives at the moisture content around the FSP may lead

to the formation of coloured substances (Wengert 1997). However, there are clear differences in the mechanism of discolouration between wood species. In many light-coloured hardwood species, the darkening occurs in the interior of the sawn timber piece (McMillen 1976; Stenudd 2002). Another type of discolouration is the so-called kiln brown stain, where a darkened layer occurs just below the surface of the sawn timber piece at the depth of about 1 mm (Kreber et al. 1998). Kiln brown stain is found especially in dried sawn timber of radiata pine (*Pinus radiata*). However, the same kind of discolouration can be found in sawn timber of Scots pine and Norway spruce (*Picea abies*) when drying temperatures of 90°C and above are used (Repola et al. 2001).

The knowledge of the mechanisms and pathways of liquid water flow and their dependence on the wood material properties would be essential for the development of new drying processes and techniques in order to control the discolouration of wood. This calls for a method illustrating the pathways of the liquid water flow in different drying conditions and with different wood species. Tracing the liquid water flow with a dye solution had been used earlier especially in soil science to visualize flow paths of water in aquifers (Atkinson 1978; Duwig et al. 2008). In living trees, dye tracers have been used to investigate the sap flow along the trunk (Edwards and Booker 1984; Zimmermann 1983).

The objective of this study was to develop a new method which could demonstrate the liquid water flow in wood during the capillary phase of the drying process. More precisely, the aim was to study how the flow of liquid water in wood during drying can be illustrated by the trace marked by a dye solution. Another aim was to develop a method to illustrate the transportation and enrichment of extractives in wood during drying above FSP.

2 Materials and methods

Fluorescent tracers are substances which strongly absorb light of certain wavelengths and then re-emit the absorbed energy as light of somewhat longer wavelength. The fluorescent tracer pyranine (trisodium 8-hydroxypyrene-1,3,6-trisulfonate, chemical formula $C_{16}H_7Na_3O_{10}S_3$, molecular weight 524 g mol⁻¹) was chosen for the experiments of this study. Pyranine is readily visible with yellow-green fluorescence under UV light. It is soluble in water (water solubility 300 g L⁻¹

¹⁾ and has applications as a coloring agent, biological stain, optical detecting reagent, and pH indicator (chemBLink 2009).

A total of 18 green silver birch (*Betula pendula*) sawn timber boards with dimensions $40 \times 100 \times 1400 \text{ mm}^3$ were used for the experiments. The average moisture content of the undried boards was 58.9%. An injection hole with a diameter of 8 mm and depth of 75 mm was drilled from the edge to the core of the board at different distances (200, 400, or 600 mm) from the board end (Fig. 1). 2.5 ml of the dye solution (150 gL^{-1}) was injected into the hole, and the hole was sealed with a wooden plug (diameter of 8 mm, length 25 mm) and PVAc glue. The boards were not end-sealed during the experiment.

The sample boards were then dried in a laboratory kiln at 75°C for 240 hours. The average final moisture content of the boards after drying was 10.9%. The dried sample boards were divided into two groups for longitudinal and cross sectional analyses. In the longitudinal analyses, the face of the boards was planed, 1.5 mm at a time, until the injection hole was revealed. After each planing, the reflectance spectrum (360-740 nm) of the wood was measured with a spectrophotometer (Minolta CM-2500d) at the location of the injection hole. The actual examination of the reflectance spectrum was focused on the wavelengths between 360 and 550 nm, which is the range where the fluorescence effect of pyranine is seen. The diameter of the spectral measurement area was 8 mm. The boards were then further planed to half of their initial thickness. The main pathways of water flow in the longitudinal direction of the boards were then viewed and photographed under UV light from the planed surface. In cross sectional analyses, three sample boards representing each distance of injection was cut to 25 mm thick slices in the fiber direction. The visibility and usability of the trace of the dye solution were compared between the cutting directions. The average length of the trace was determined both in cross sectional and longitudinal direction.

The experimental set-up of image analysis consisted of a tripod, a 15W UV-A light and a digital camera. The boards split longitudinally and the cross sectional slices of the boards were photographed for longitudinal and cross sectional dye traces, respectively. The digital images were converted to negative images to

better distinguish the dye trace from the wood. In negative images, the dye trace appeared red for colour images and black for black and white images.

In order to assess the effect of diffusion on the migration of dye during dyeing, a theoretical calculation of the maximum diffusion in one direction from the injection point was made. Therefore, the equation for concentration distribution at distance x from the injection point at time t in the case of diffusion across a boundary, where the initial concentration is zero on one side and constant on the other side, was used (Crank 1986):

$$C(x,t) = \frac{1}{2} C_0 \operatorname{erfc} \frac{x}{2\sqrt{Dt}} \quad (1)$$

where C is the concentration of diffusing substance in the material the diffusing substance is immersed in, C_0 is the initial concentration at $x < 0$, erfc is the complimentary error function, and D is the diffusion coefficient.

3 Results

The trace of the dye solution was not visible in the dried wood to the naked eye in standard light (Fig. 2). However, it could be detected by the spectrophotometer and also with the naked eye upon UV illumination. The flow of the dye solution provided a trace which was easily visible against the wood under UV light. It was found that the perceptivity of the trace in the images could be improved by using the photo-negative effect (Fig. 2). The originally yellow-green trace got a reddish tint in the colour negatives and a black tint in the black and white negatives.

The fluorescence effect of the dye solution on the reflectance spectra of wood is seen as a sharp increase of absorption at 360–420 nm and an increase of emission at 430–550 nm (Fig. 3). Evidently, the increase in the concentration of the dye in wood increased the fluorescence effect in the reflectance spectra. The fluorescence effect was greatest near the injection point of the dye solution, and it gradually decreased with increasing distance from the injection point. The

decrease in the fluorescence effect was in accordance with the change in the saturation of colour of the dye trace in the digital images (Fig.2 and 3).

Image analysis of the longitudinally cut boards showed that at a distance of 200 mm from the board end, the injected dye solution could leave a trace along the entire length of the board as the dye solution flowed along with the water (Fig. 2, Table 1). The maximum trace of the dye solution covered 65 and 71 % of the entire length from the injection point to the board end at distances of 400 and 600 mm, respectively. The average length of the trace covered 89, 39, and 35% of the distance from the injection point to the board end for distances of 200, 400, and 600 mm, respectively (Table 1). However, in the cross sectional samples it was seen that the trace may cover the entire length also at the distances of 400 and 600 mm (Fig. 4).

In the cross sectional direction, the trace of the dye solution was not usually detected on the surface of the sample boards when the reflectance spectra measurements were used at the distance of the injection point (Fig. 5). This was in accordance with the results of image analysis of the cross sectional slices (Fig. 4). The dye solution was moved approximately 6-9 mm from the injection point in the cross sectional direction (Table 2). Direct cross sectional flow from the interior of the board to the surface was not observed.

The cross sectional trace of the dye solution widened slightly as the longitudinal distance from the injection point increased. Mainly, the longitudinal traces of the dye solution followed the grain direction, i.e., not the exact longitudinal direction of the board. This was seen most distinctively around knots, where the trace made a curve and skirted the knot (Fig. 6). Evidently, the effect of the grain direction was also seen in the cross sectional trace in the boards with a distance of injection point of 400 and 600 mm, as it did not precisely follow the longitudinal direction of the boards but turned toward the face of the boards (Fig. 4). Later on, the dye solution had accumulated in a layer where the flow of capillary water ceased.

As result of the accumulation of the dye, a strong and straight layer parallel to the board surface was formed under the board surface (Fig. 4). The accumulated layer

was mainly located at a depth of 2-4 mm from the surface, and the depth decreased with increasing distance from the injection point. Evidently, the accumulation of the dye occurred in the evaporation front developing under the board surface when a dry shell is formed at the very beginning of the drying (see Salin 2008). The appearance of the accumulated layer on one side of the cross sections only indicates the importance of the grain direction in the flow of liquid water.

When the spectral measurements next to the injection point were compared, the effect of the dye fluorescence on the reflectance spectrum differed between cross sectional and longitudinal directions (Fig. 3 and 5). The nearest measuring points of the reflectance spectrum (0 mm in the longitudinal direction and 3 mm in the cross sectional direction) can be considered to represent the same distance from the injection point, as each measurement represented an area of an 8 mm circle. However, the fluorescence effect was seen much clearer at the longitudinal measuring point. The stronger fluorescence can be considered to represent higher concentration of the dye.

Based on Eq. 1, a concentration-distance curve for the diffusion of the dye was plotted (Fig. 7). The diffusion coefficient of pyranine was not known. The diffusion coefficient of raffinose ($D = 0.43 \times 10^{-9} \text{ m}^2 \text{ s}^{-1}$, Longsworth 1953) was chosen, because it has a molecular weight (504.4 g mol^{-1}) close to that of pyranine. The curve shows that 50% of the dye concentration after a drying period of ten days was achieved at a distance of approx. 18.5 mm, and the concentration dropped below 10 % at a distance of approx. 45 mm. The concentration achieved by the diffusion will approach 0 at a distance of approx. 80 mm.

4 Discussion

This study arose from the need to clarify the physical processes between liquid water and wood extractives during the capillary phase of the drying process. This information is essential, e.g., when assessing the reasons for discolouration of wood caused by polymerization of extractives during the drying process.

The liquid solution of dye and water left a dry trace in the wood after a drying process, which was analyzed with spectral and image analysis. The fluorescent dye was not detectable in the dried wood samples by image analysis until UV light was used. Spectral analysis could be used to detect the presence of the dye without UV light illumination. The spectral analysis also indicated variation in the concentration of the dye tracer, which was in accordance with the result of image analysis. The fluorescence effect of the dye shown in spectral measurements weakened with the distance from the injection point of the dye solution. The length of the trace in the longitudinal and cross sectional direction could be determined both with image and spectral analysis.

At low concentrations, the accuracy of image analysis may not be high enough for objective determination of the dye trace. However, according to the results of this study, the image analysis corresponded very well with the measurement of spectral reflectance. Therefore, the image analysis can be considered to be accurate enough for the determination of the flow paths of the liquid water in wood when a dye solution is used. The trace of the dye solution can be determined from the images of the cross sectional samples of wood in all three main directions. In each cross section of the board along the longitudinal direction, the development of the radial and tangential trace was in view. However, the longitudinal flow along the grain direction was better in view in lengthwise-cut samples.

Chemical dye solutions were used by Kawai et al. (2003) to study the liquid water flow at steady state conditions in sawn wood of *Cryptomeria japonica*. They found water flow through the sawn timber first in the longitudinal direction and then in a direction perpendicular to the grain or in radial and tangential directions. According to Edwards and Booker (1984), the farthest penetration of a dye tracer in living trees was found in the longitudinal direction through the outermost growth rings of sapwood. In this study it was found that the movement of the dye solution in the cross sectional direction was less than 1/10 compared to that in the longitudinal direction. The longitudinal flow is possibly even more predominant in hardwood than in softwood. Moreover, it is obvious that longitudinal flow is

more predominant at high moisture contents parallel to the main direction of the wood capillary network.

All the cross sectional flow of water may not be visible due to the fact that the dye tracer and water do not necessarily interact in the same way with the surrounding wood matrix and environment. The most important difference between dye tracers and water is the molecular size (Atkinson 1978). The cross sectional movement of pyranine in wood is possibly more constricted than that of water due to the difference in molecular size. Due to the elongated pattern of wood cells, there are much more barriers for the solute movement in cross sectional than in longitudinal direction, which may retard the cross sectional movement of pyranine more than the movement of water. Compared to other chemical tracers, the solubility of pyranine in water is high (Herbst and Wygoda 2006). Therefore, the movement of pyranine along with the water can be considered to follow the movement of water soluble extractives of wood.

The method developed in this study can be used to determine the pathways of liquid water flow during drying above FSP. The results obtained here are not valid for the transfer of bound water or water vapor during the drying phase below FSP. However, some similarities were found with the study carried out by Mouchot et al. (2006), who determined diffusivities for the transfer of water vapor and bound water in the three orthotropic directions. They found that the axial diffusivities were clearly greater than the diffusivities in the tangential and radial direction. The longitudinal diffusion coefficients vary with moisture content due to the effect of hardwood vessels (Choong et al. 2001).

According to the calculated concentration distribution caused by the diffusion, the effect of diffusion on the migration of the dye is relatively small but not insignificant. The estimated concentration distribution represents the maximal migration which the dye could be moved by diffusion. The migration of the dye in the cross-sectional direction observed with the spectrophotometer was much smaller than one would assume based on the calculations of diffusion, which indicates differences also in diffusion between cross-sectional and fiber directions.

The dye tracer method used in this study indicated a clear connection between the migration of wood extractives and the development of discoloured layers in wood during drying. Although the kiln brown stain is not a typical form of discolouration in birch sawn timber, the location of accumulated layer of the dye, which was formed below the board surface, corresponds with the location of kiln brown stain (cf. Kreber et al. 1998). In addition, the accumulation of dye as a layer parallel to the board surface corresponds to the location of the evaporation front, which is formed at a depth of 1-2 mm below the surface when the damaged surface layer dries and forms a dry shell (Salin 2008).

5 Conclusion

The method developed in this study can be utilized for the evaluation of liquid water flow in wood during drying. It also demonstrates the flow of water soluble extractives towards the surface along with the liquid water and their accumulation at the layers near the surface. Therefore, the method supports the discolouration phenomenon caused by the migration of extractives during the drying of wood.

The results of this study show that the moisture content of wood in the core of sawn birch timber may remain high for a relatively long time, as a result of longitudinal flow of liquid water. This, in turn, may be the reason for differences in discolouration between the surface layer and the core of birch sawn timber.

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Table 1 Length of the trace of the dye solution (mm) measured from the longitudinal images for each distance of injection from the board end

Tabelle 1 Länge der Spur der Farblösung (mm), gemessen von den längsgerichteten Aufnahmen für jede Distanz der Injektion vom Brettende

Distance of injection	200 mm	400 mm	600 mm
Average (N=9)	178	155	210
Min	95	28	75
Max	200	260	425

Table 2 Length of the trace of the dye solution in cross sectional direction based on the spectral measurements of wood

Tabelle 2 Länge der Spur der Farblösung in Querschnittsrichtung, basierend auf den Spektralmessungen des Holzes

Distance of injection	200 mm	400 mm	600 mm
Average (N=9)	6.0	9.0	7.0
Min	0	3.0	0
Max	10.5	15.0	15.0

Figure Captions

Fig. 1 Schematic presentation of the injection of dye solution at the beginning of the experiment

Fig. 2 Longitudinal trace of the dye solution in dried and split board for 200 mm distance of injection. Images: a) standard, b) UV-illuminated, c) UV-illuminated color negative, and d) UV-illuminated black and white negative

Fig. 3 Change in the reflectance spectrum of wood by the distance from the injection point of the dye solution in the longitudinal direction

Fig. 4 Cross sectional trace of the dye solution at 25 mm steps along the board. Distances of the injection point of dye solution from the board end: 200 mm (above), 400 mm (in the middle) and 600 mm (below)

Fig. 5 Change in the reflectance spectrum of wood by the distance from the injection point of the dye solution in the cross sectional direction

Fig. 6 Longitudinal trace of the dye solution in dried and split boards for a 400 mm (above) and 600 mm (below) distance of injection

Fig. 7 Concentration-distance curve for calculated migration of pyranine by diffusion during the 10 day drying period

Abb. 1 Schematische Darstellung der Injektion der Farblösung zu Experimentbeginn

Abb. 2 Längsgerichtete Spur der Farblösung im getrockneten und gespaltenen Brett bei 200 mm Distanz der Injektion. Aufnahmen: a) Standard, b) UV-beleuchtet, c) UV-beleuchtet, Farbnegativ, d) UV-beleuchtet, Schwarzweissnegativ

Abb. 3 Veränderung im Reflektionsspektrum des Holzes in Abhängigkeit von der Distanz zum Injektionspunkt der Farblösung in Längsrichtung

Abb. 4 Querschnittsgerichtete Spur der Farblösung in 25 mm-Schritten entlang des Bretts. Distanzen des Injektionspunktes der Farblösung vom Brettende: 200 mm (oben), 400 mm (Mitte) und 600 mm (unten)

Abb. 5 Veränderung des Reflektionsspektrums des Holzes in Abhängigkeit vom Injektionspunkt der Farblösung in Querschnittsrichtung

Abb. 6 Längsgerichtete Spur der Farblösung im getrockneten und gespaltenen Brett für eine Injektionsdistanz von 400 mm (oben) und 600 mm (unten)

Abb. 7 Konzentrations-Distanz Kurve für die berechnete Migration des Pyranins durch Diffusion während der 10-tägigen Trocknungsphase

Fig1.doc

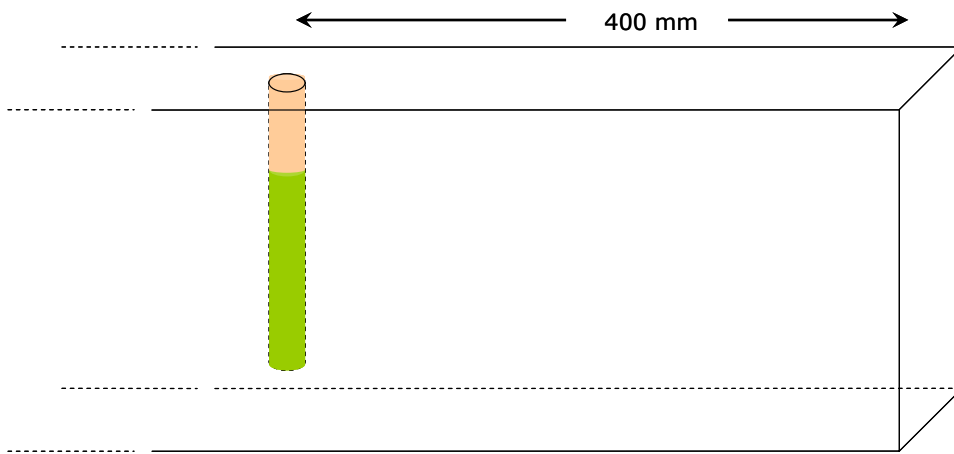


Fig2.doc

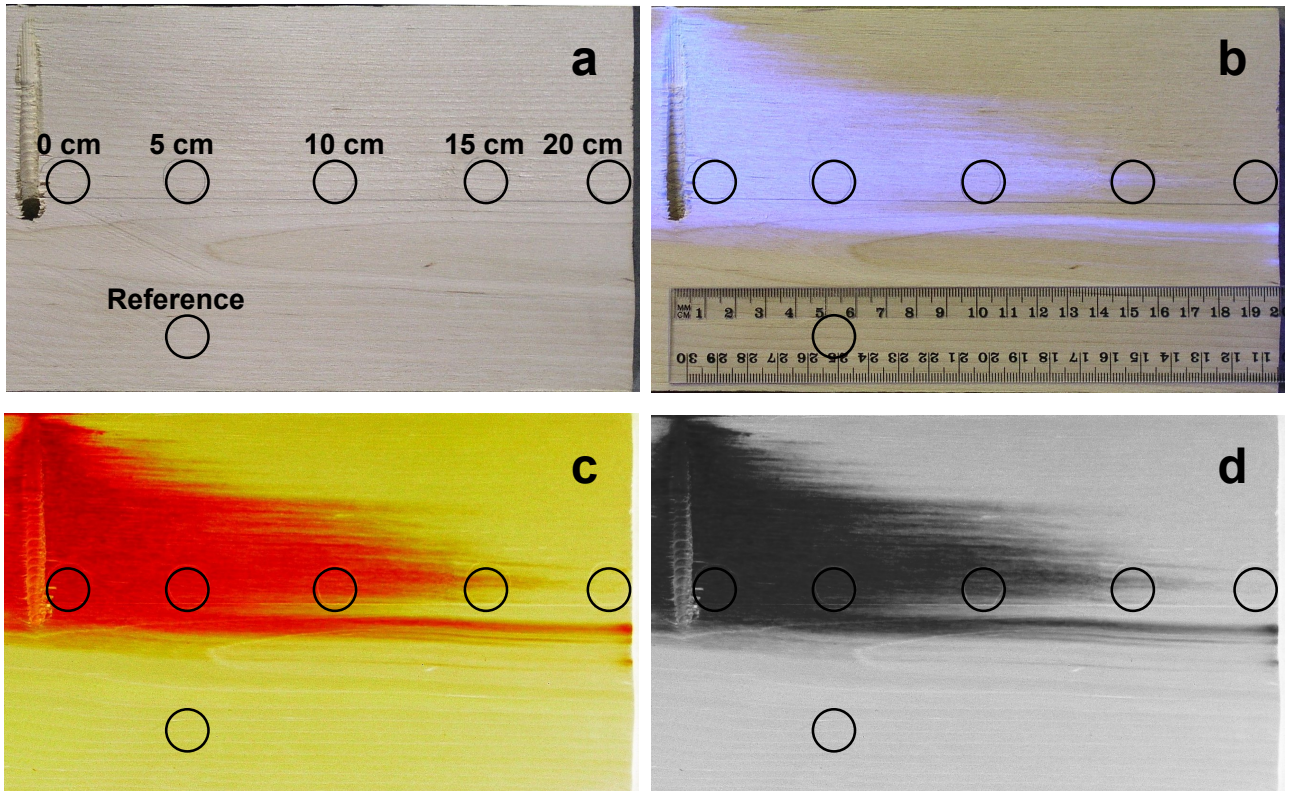


Fig3.doc

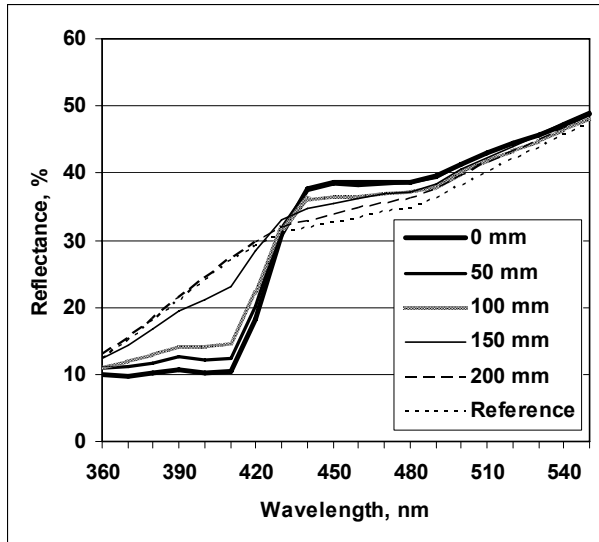


Fig4.doc

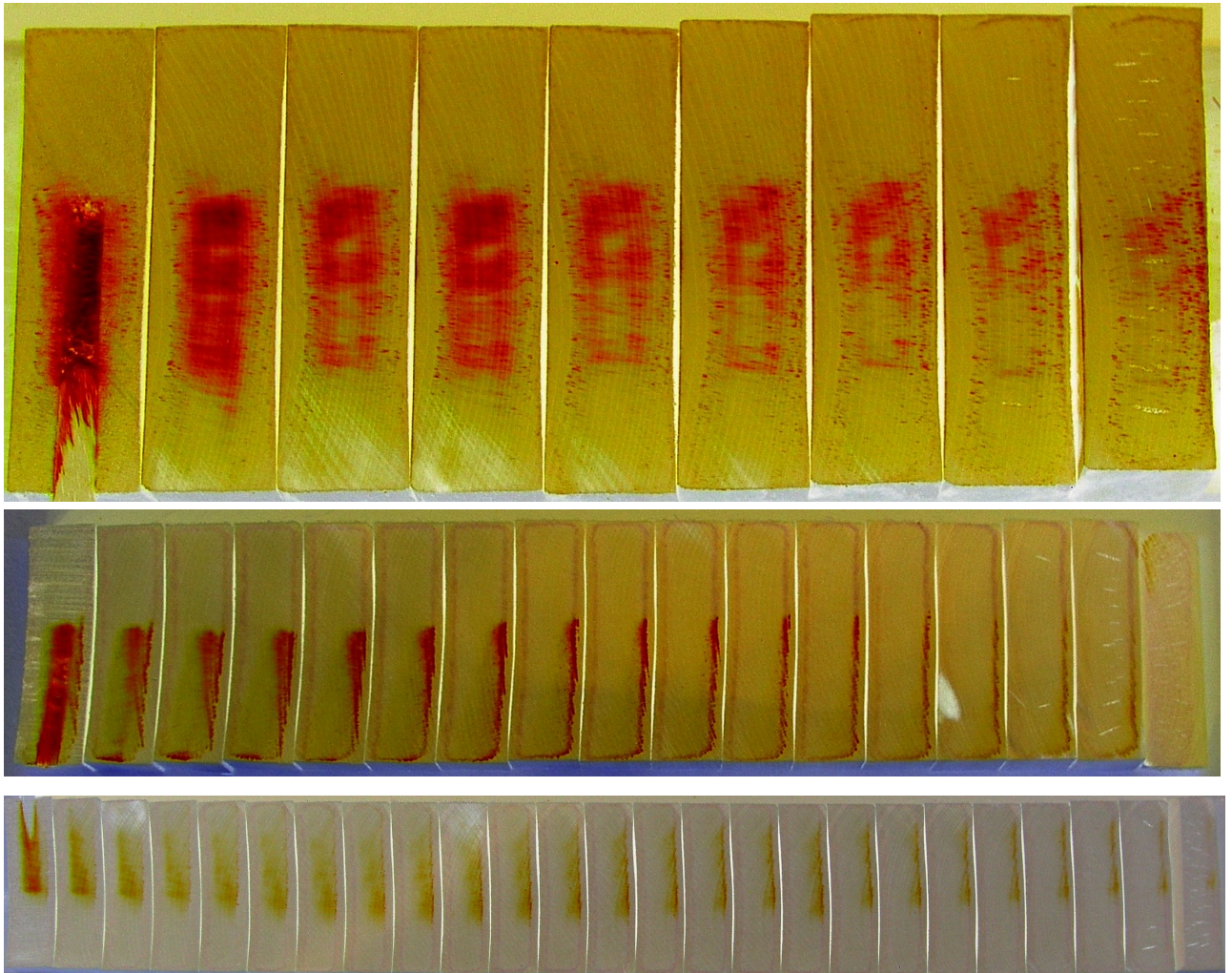


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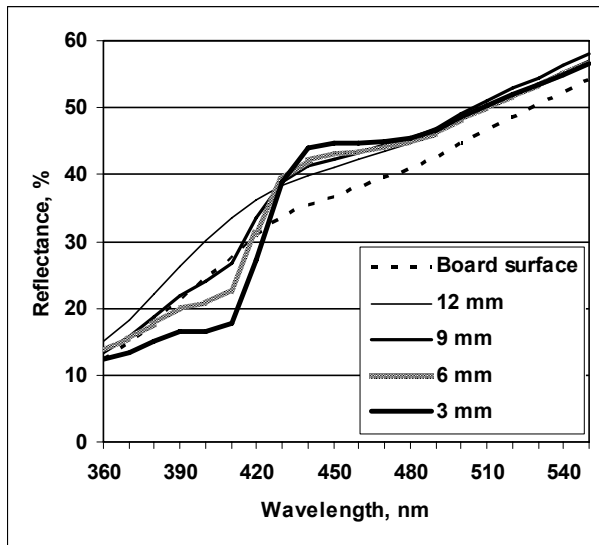


Fig6.doc

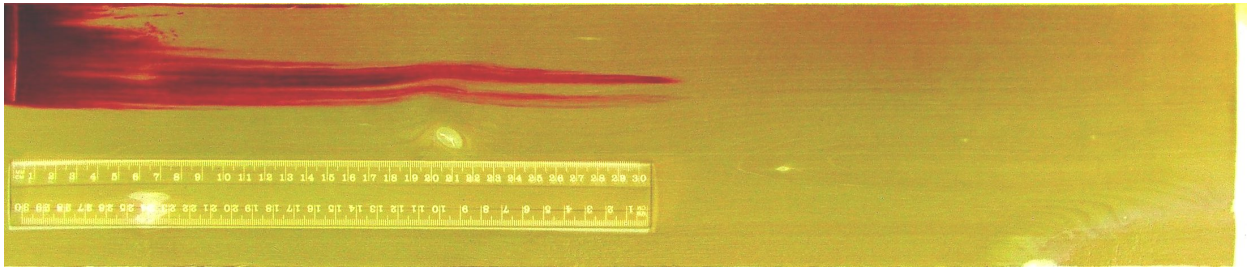
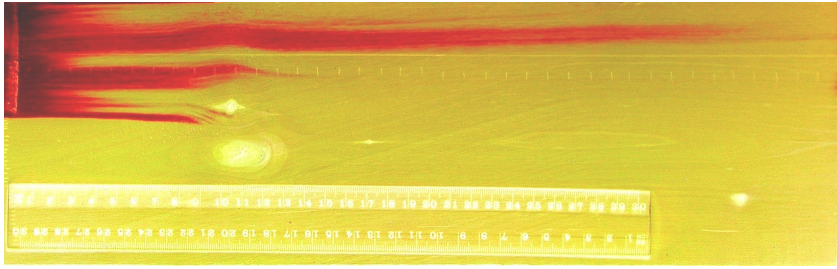


Fig7.doc

