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# Effects of surface ageing on wettability, surface chemistry, and adhesion of wood

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Abstract The effects of ageing on the surface chemistry, wettability and the surface free energy were determined according to the acid base theory using contact angle measurements and x-ray photoelectron spectroscopy. Time is a significant variable affecting wettability and adhesion. All measured surface parameters of the wood surface were storage time dependent. It could be demonstrated that the effect of ageing on the adhesion of a coating on the wood surface, but also on the chemical nature of the coating. Therefore, measurements on wettability without considering the time dependence of surface energy and, above all, its components, are not sufficient to assess effects of ageing on adhesion.

## Einfluß der Oberflächenalterung auf die Benetzbarkeit, Oberflächenchemie und Klebeeigenschaften von Holz

Zusammenfassung Die Wirkung der Alterung auf die Oberflächenchemie, die Benetzbarkeit und die freie Oberflächenenergie nach der Säure/Base Theorie wurden mittels Kontaktwinkelmessungen und Photoelektronenspektroskopie untersucht. Die Zeit beeinflusste die Benetzbarkeit und Adhäsion des Holzes deutlich. Mit der Lagerungszeit veränderten sich alle gemessenen Parameter der Holzoberfläche. Es konnte gezeigt werden, daß der Alterungseffekt auf die Adhäsion einer Beschichtung nicht nur von den Eigenschaften der Holzoberfläche, sondern auch von der Chemie der Beschichtung abhängt. Benet-

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zungsmessungen ohne Berücksichtigung der freien Oberflächenenergie und ihrer Komponenten reichen daher nicht aus, um Alterungseffekte auf die Adhäsion abzuschätzen.

# **1** Introduction

Wood is a complex material composed of cellulose, lignin, hemicellulose and extractives, and its wetting properties such as contact angle<sup>1</sup>, surface free energy and interfacial work of adhesion depend on many factors: e.g. wood species, different treatments, sapwood or heartwood, previous history such as exposure to water, light, weathering or biological attacks, cleanliness, method of drying, grain orientation and ageing of exposed surface (Nguyen and Johns 1979; Kalnins and Knaebe 1992). Freshness of the wood surface is one of the most critical factors for the extended durability of painted wood and only a fresh, high energy surface guarantees optimum adhesion conditions (Dougal et al. 1980, Nussbaum 1999). The loss of coating ability and glueability with increasing age of a wood surface is a phenomenon that has attracted the interest of several researchers (Chen 1970; Troughton and Chow 1971, Gardner et al. 1991, Nylund et al. 1998, Nussbaum 1999, Wålinder 2000), who came to the conclusion that the ageing effect is above all due to a migration of wood extractives to the exterior surfaces after their preparation and that the wettability of wood surfaces decreases with the ageing of the surface.

Christiansen (1994) summarised the mechanisms responsible for changes of wood surfaces that may influence the bonding properties of wood: 1) migration of hydrophobic extractives during drying, 2) oxidation, 3) closure of micro-voids in the wood substance which reduces adhesive penetration, 4) acidity or reactivity of extractives affecting the curing time of adhesives, 5)

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<sup>&</sup>lt;sup>1</sup> The contact angle is defined as the angle between the tangent to the liquid surface and the liquid/solid surface at the point of liquid/ solid contact (see also Gindl et al. 2001a).

molecular reorientation of functional groups at the surface.

The aim of this paper is to investigate the effects of ageing on different wood surfaces using contact angle measurements combined with X-ray photoelectron spectroscopy (XPS). From the contact angle data, the surface free energy and its components as well as the interfacial work of adhesion between water and wood, and also between a water soluble coating and wood will be calculated using the acid/base theory. As this method of calculation allows to determine not only polar and unpolar components of the surface free energy, but also permits to split the polar component into an acid and a base part supported by XPS data, it is capable of providing new insights into the chemistry of surface ageing. A subject which is well known but has not been fully explained yet.

## 2 Material and methods

Samples of Norway spruce (*Picea abies [L.) Karst.*) and beech (*Fagus sylvatica L.*) were conditioned to a moisture content of 12%. The determinations of contact angle of the droplets were done in grain direction of the wood with the sessile drop method, using a "Digidrop" (GBX Instruments) measuring device. Two approaches were followed:

- 1. Immediately after surface preparation and after ageing during seven days the components of surface free energy were determined on microtomed surfaces of spruce and beech and also on sanded (P100) spruce surfaces. Considering the low viscosity and molecule size of the appropriate test liquids diiodmethane, formamide, and water, and the hydrophility of wood as a porous material, advancing contact angles as defined in earlier work (Scheikl and Dunky 1998, Gindl et al. 2001a) were measured and used for calculation of surface free energy. Except for sanded spruce, small particles were cut from the surfaces for XPS analysis.
- 2. Immediately after surface preparation and after  $0-4^2$ , 6, 16, 24, 40, 48, 72, 96 and 168 h the surface free energy of a sanded spruce surface (P100) was determined together with the wettability of the surface with water and a water soluble coating. For the same reasons as described above, advancing angles were used with water. Whereas, keeping in mind the rheological properties (especially high viscosity) of the chosen water soluble coating, static contact angles after 20 s as defined in earlier work (Gindl et al. 2001b) were applied to analyse the wetting using the coating liquid.

Additionally, the surface tension of water and the water based coating system were determined using the pendant drop method (Gindl et al. 2001b). The data for the other test liquids used (diiodmethane and formamide) can also be found in this publication.

The interfacial work of adhesion between wood and water soluble coating ( $W_{1,2}$ ) was determined using surface energy data of wood and the hardened water soluble coating.

Here it is important to note that a number of theories (equation of state, geometric mean, harmonic mean, acid/base theory) for the calculation of surface free energy have been applied to wood or to wood composites (Nguyen and Johns 1979, Liptáková et al. 1995, Gardner 1996, Mantanis and Young 1997,Wulf et al. 1997, Scheikl and Dunky 1998, Shen et al. 1998, de Meijer et al. 2000, Wålinder 2000). There is some controversy with respect to the correctness of different thermodynamic approaches to determine the surface energy of solids from contact angle measurement (Good 1992, Wu 1999). Recently we compared the surface free energy of micro-tomed surface of spruce using five different theories (Gindl et al. 2001a) and we concluded that the acid-base approach has the advantage to provide additional information in terms of acid and base components. In an earlier study (Gindl et al. 2000) it was found that the acid and base components of surface free energy of wood react sensitively to varying extractive content and composition and in such a way help to explain coating properties of wood.

New findings on the chemistry of liquids (Morra 1996, dela Volpe and Siboni 1997, Shen 2000) suggest different acid/base ratio of water, causing different acid/base components of test liquids and finally leading to different results for the acid/base components of surface free energy of a solid. The influence of this fact on the components of surface free energy of wood is described by Wålinder (2000) and Shen (2000). For our investigation common values from the literature (Good and van Oss 1993) were used.

Finally, the determination of the surface free energy of wood surface is inherently difficult, because of its intricate nature: i.e. porosity, hygroscopity, anatomic complexity, heterogeneity, extractive content. According to all the aforementioned reasons the values of surface free energy for spruce wood presented here must be regarded with caution.

#### 2.1 Surface free energy of solid

The procedure described in the following was applied to wood surfaces and the surface of the hardened water soluble coating. The steps of calculation of the components of surface free energy applying the acid/base theory will not be described in detail, since descriptions can be found in literature (van Oss 1988, Good and van Oss 1993). It is important to note that the usually used terms are "surface tension" for the liquid state and "surface free energy" for the solid state (Berg 1992). According to the acid/base theory the total surface free energy of a solid (or surface tension for the liquid state) is split into the Lifshitz/van der Waals or disperse (unpolar) component  $\gamma^{LW}$ , which is based on temporary forces of attraction and repellence due to the movement of electrons in all molecules, and the acid/base or polar component  $\gamma^{AB}$ , which is based on dipole-dipole interactions between polarised functional groups (Eq. 1).

$$\gamma_i = \gamma_i^{LW} + \gamma_i^{AB} = \gamma_i^{LW} + 2\sqrt{\gamma_i^+ \gamma_i^-} \tag{1}$$

As seen in Eq. 1, the acid/base component is further split into an electron acceptor  $(\gamma^+)$  and an electron donor  $(\gamma^-)$  component. According to the theory, the mathematical approach for the solid/liquid interfacial tension is given by Eq. 2, where the subscripts *S* and *L* represent the solid and liquid, respectively.

$$\gamma_{SL} = \gamma_S + \gamma_L - 2\left[\left(\sqrt{\gamma_S^{LW}\gamma_L^{UW}}\right) + \left(\sqrt{\gamma_S^+\gamma_L^-}\right) + \left(\sqrt{\gamma_S^-\gamma_L^+}\right)\right]$$
(2)

#### 2.2 Interfacial work of adhesion

Since the process of adhesion is essentially completed after transition of the coating from liquid to solid state (Vick 1999, de Meier and Militz 2000), the interfacial work of adhesion ( $W_{1,2}$ ) between two phases (hardened water soluble coating and wood) was calculated from the surface free energies of both according to the following equation using Lifshitz-van der Waals acid/base parameters:

$$W_{1,2} = 2\sqrt{\gamma_1^{LW}\gamma_2^{LW}} + 2\sqrt{\gamma_1^+\gamma_2^-} + 2\sqrt{\gamma_1^-\gamma_2^+}$$
(3)

Subscript 1 stands for the hardened water soluble coating, subscript 2 for the wood surface.

 $<sup>^2</sup>$  "0–4 h" indicates the mean value of surface free energy (and wettability) on sanded surface of spruce immediately after treatment and after 1; 2 and 4 h, as no significant difference between these four measurements was found.

Detailed descriptions of the XPS method will not be given here, as they can be found in the literature (Sinn et al. 2001). For the XPS investigations a PERKIN ELMER 5500 ESCA System, with Mg Anode (X-ray-voltage 15 kV, X-ray energy 1,253.6 eV) was used. The acquisition time was chosen to 10 min in order to obtain sufficient counting statistic. For each sample two spectra in a fixed analyser mode were measured, and from these survey spectra the percentage of oxygene (O) and of carbon (C) were determined and the ratio of atomic concentration of oxygen to carbon was calculated.

# **3 Results**

The data of the surface tension properties of water and the applied water soluble coating, and the surface free energy of water soluble coating are depicted in Table 1. It may be seen that water has a clearly polar character (the acid/base component dominates), whereas the water soluble coating is only slightly polar in the liquid state, but shows an unpolar character in the solid state (the Lifshitz/van der Waals component is highest).

Figures 1 and 2 depict the effect of ageing on the surface free energy and its components on sanded and microtomed spruce wood and also on microtomed beech wood.

Seven days after sanding or microtoming the surface free energy decreased significantly. On sanded surfaces, the change is mainly due to a reduction of the disperse component, whereas the polar component is responsible for the reduction of the surface free energy of the microtomed surfaces (Fig. 1). Also regarding the acid/base components, differences between sanded and microtomed surfaces were observed (Fig. 2a and Fig. 2b). On sanded surfaces the acid component increased and the base component decreased. On microtomed surfaces only the base component changed significantly with increasing surface age (Fig. 2b).

The results of the XPS analysis of fresh and aged wood surfaces are shown in Table 2. A consistent decrease of the O/C ratio after ageing was found on spruce as well as

Table 1 Experimental data for the surface tension of water and water soluble coating and the surface free energy of water soluble coating Tabelle 1 Experimentelle Daten für die Oberflächenspannung von Wasser und wasserlöslicher Beschichtung und der Oberflächenergie der wasserlöslichen Beschichtung

		γ	$\gamma^{LW}$	$\frac{\gamma^{AB}}{[\mathrm{mJm}^{-2}]}$	$\gamma^+$	γ <sup>-</sup>
Distilled water <sup>a</sup> Water soluble coating <sup>a</sup> Water soluble coating <sup>b</sup>	liquid solid	72.8 24.5 50.9	21.8 11.0 45.9	51.0 13.5 5.6	25.5 <sup>c</sup> - 0.9	25.5° - 8.7

Pendant drop method

b Sessile drop method

Good and van Oss (1993)

 $\gamma = \text{total surface free energy}, \gamma^{LW} = \text{disperse comp. of } \gamma, \gamma^+ = \text{acid comp. of } \gamma, \gamma^- = \text{base comp. of } \gamma, \gamma^{AB} = \text{polar comp. of } \gamma$ 



Fig. 1 Polar and disperse components of surface free energy of spruce and beech wood surfaces (mean values and standard deviation of 5 identically prepared samples; always 3 drops per test liquid on each samples): LW-for Lifshitz van der Waals component of  $\gamma_S$ ; AB—for acid/base component of  $\gamma_S$ 

Abb. 1 Polare und disperse Anteile der Oberflächenenergie von Fichten- und Buchenholzoberflächen (Mittelwerte und Standardabweichung von 5 gleich vorbereiteten Proben: jeweils 3 Tropfen pro Testflüssigkeit an jeder Probe): LW-für Lifshitz van der Waals Anteile der  $\gamma_S$ ; AB—für Säure/Base Anteile der  $\gamma_S$ 

Fig. 2 a Acid components of surface free energy of spruce and beech wood surfaces; b Base components of surface free energy of spruce and beech wood surfaces

Abb. 2 a Säure Anteile der Oberflächenenergie von Fichten- und Buchenholzoberflächen; b Base Anteile der Oberflächenenergie von Fichten- und Buchenholzoberflächen



Table 2XPS analysis of fresh and aged wood samplesTabelle 2XPS Analyse von frischem und gealtertem Holz

Sample	% Atom com	O/C	
	Oxygen (O)	Carbon (C)	ratio
Fresh sanded spruce	29	71	0.42
Aged sanded spruce	-	-	_
Fresh microtomed spruce	34	66	0.52
Aged microtomed spruce	26	74	0.35
Fresh microtomed beech	33	67	0.49
Aged microtomed beech	27	73	0.38

on beech surfaces. The change of the O/C ratio was more pronounced on microtomed spruce than on microtomed beech.

The dynamics of the ageing process on sanded spruce surfaces are described in Figs. 3 and 4. The decrease of the surface free energy with time is best fitted by a logarithmic trend line (Fig. 3). While the change of the disperse component with time is comparably small but significant, the composition of the polar component, i.e. the acid and base component, exhibit drastic alterations within the investigated seven day period (Fig. 4). By comparing the time dependent behaviour of water in terms of changing contact angle to a water soluble coating system, a clearly different trend becomes visible (Fig. 5). The contact angle of the coating system does not increase, but decreases, though highly scattering. However, if the interfacial work of adhesion is calculated, water and also the water soluble coating show a decrease over time (Fig. 6 and 7). The disperse (Lifschitz/van der Waals) component is responsible for the reduction of the interfacial work of adhesion between the water soluble coating and the wood surface (Fig. 6). In contrast, the interfacial work of adhesion between the water and the wood surface decreases with time because of a diminishing polar (acid/base) component (Fig. 7)

# **4** Discussion

In this study surface parameters like surface free energy and its components (disperse, acid and base components), wettability and adhesion properties of aged and freshly produced surfaces of wood samples, and their surface elemental composition were determined and applied to describe ageing effects as a function of alteration of wood Fig. 3 Time dependence of total surface free energy of sanded spruce wood over 7 days after treatment Abb. 3 Zeitabhängigkeit der gesamten Oberflächenenergie von geschliffenen Fichtenholzoberflächen ermittelt über 7 Tage nach der Bearbeitung

**Fig. 4** Time dependence of disperse, acid and base components of surface free energy of sanded spruce wood: LW—for Lifshitz van der Waals component of  $\gamma_S$ 

Abb. 4 Zeitabhängigkeit der Disperse, Säure und Base Komponenten der Oberflächenenergie von geschliffenen Fichtenholzoberflächen: LW für Lifshitz van der Waals Anteile der  $\gamma_S$ 



Time [h]

days

 $\sim$ 

day

60

days

days

components on the surface. Results for the change of surface free energy over time determined in this study, i.e. the total surface free energy diminishes with surface age (Fig. 1 and 3), are in good agreement with results from the literature obtained using different mathematical approaches (Nguyen and Johns 1979, Dougal et al. 1980, Hse and Kuo 1988, Gardner et al. 1991, Back 1991, Christiansen 1994, Wålinder 2000). The different ageing behaviour of sanded and microtomed surfaces of spruce wood (Fig. 1), which is due to the influence that the conditions and methods of wood surface formation exert on surface chemistry, has been discussed by several authors (Zavarin 1984, Liptáková et al. 1995, Jaić et al. 1996, Mantanis and Young 1997, Gindl et al. 2001b). However, the study of the acid, base and disperse components of surface free energy according to the acid/base

theory, in combination with XPS analysis, as performed in the present investigation, allows deeper insights into the mechanisms of surface ageing.

Figures 2 and 4 depict age-dependent values for the acid and the base component. It is believed that oxidation of extractives over the time tends to increase the acidity of wood (Hse and Kuo 1988, Back 1991) a trend which is excellently reflected by the general increase of the acid component and the decrease of the base component (Wålinder 2000).

Concerning the wettability properties of the aged wood surface, it is not surprising that the fresh wood surface is more easily wetted by water then the aged surface, as consistently found in the literature (Nguyen and Johns 1979, Gardner et al. 1991, Liu et al. 1995, Nussbaum 1999). Also our XPS data indicates a decrease of the polar

7 days

Fig. 5 Time dependence of the contact angle of water and water soluble coating Abb. 5 Kontaktwinkel von Wasser und wasserlöslicher Beschichtung in Abhängigkeit von der Zeit

Fig. 6 Time dependence of total interfacial work of adhesion and its components for water soluble coating/wood system: LW-for Lifshitz van der Waals component of total interfacial work of adhesion; AB-for acid/base component of total interfacial work of adhesion Abb. 6 Zeitabhängigkeit der gesamten Adhäsionsarbeit und ihrer Komponenten für das System wasserlösliche Beschichtung/Holz: LW-für Lifshitz van der Waals Anteile der Adhäsionsarbeit; AB-für Säure/Base Anteile der Adhäsionsarbeit



character of the wood surface, since the percentage of carbon increases with time and the oxygen percentage decreases simultaneously, pointing towards a greater hydrophobicity (Gardner et al. 1991).

The contact angle of the water soluble coating shows an opposite trend; it decreases with time, indicating a better wettability of aged surfaces (Fig. 5). Considering the fact that the water soluble coating has a much less polar character than water (Table 1) and hydrophobicity of the wood surface increases with time, this surprising behaviour can be explained. Results for the age dependent interfacial work of adhesion (Fig. 6, Fig. 7) demonstrate that a good wetting, as it is the case with the water soluble coating on aged surfaces, does not automatically ensure good adhesion. The process of adhesion is essentially completed after transition of the adhesive from liquid to solid form. So far, the effect of surface ageing on wettability has almost been exclusively studied by measurement of contact angle of water or other lower viscosity liquids on ageing wood surface (Nguyen and Johns 1979, Nussbaum 1999). Based on these results, the adhesion between the ageing wood surface and coating or adhesive materials was assessed, but the wettability and the interfacial work of adhesion between water and wood are the parameters which are not suitable for an exact description of the system wood-coating. Only the knowledge of surface free energy and its components of a coating material after hardening allows a meaningful calculation of interfacial work of adhesion in the wood-coating system. The chemical nature (dominance of polar or unpolar charac-



Fig. 7 Time dependence of total interfacial interfacial work of adhesion and its components for water/wood system: LW—for Lifshitz van der Waals component of total interfacial work of adhesion; AB—for acid/base component of total interfacial work of adhesion

ter) of the coating has to bee taken into account to be able to make conclusions about the time-dependence of adhesion from wetting experiments, as any coating system will show its own characteristic behaviour according to its chemical and physical properties. It has to be considered, however, that the distribution of components within a hardened coating layer is not always perfectly homogeneous (Pecina and Paprzycki 1995).

In future, more attention should bee given to the relation between adhesion, surface free energy or interfacial work of adhesion and chemical structure of the system wood-coating.

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Abb. 7 Zeitabhängigkeit der gesamten Adhäsionsarbeit und ihrer Komponenten für das System Wasser/Holz: *LW*—für Lifshitz van der Waals Anteile der Adhäsionsarbeit; *AB*—für Säure/Base Anteile der Adhäsionsarbeit

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