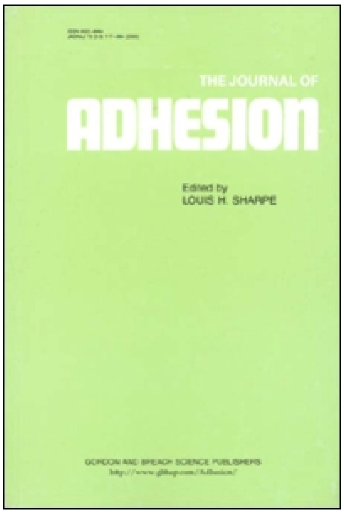

The Journal of Adhesion
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713453635

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To cite this Article Stehr, Micael , Gardner, Douglas J. and Wålinder, Magnus E. P.(2001) 'Dynamic Wettability of Different Machined Wood Surfaces', The Journal of Adhesion, 76: 3, 185 — 200
To link to this Article: DOI: 10.1080/00218460108029625
URL: http://dx.doi.org/10.1080/00218460108029625

J. Adhesion, 2001, Vol. 76, pp. 185–200
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Dynamic Wettability of Different Machined Wood Surfaces

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(Received 8 July 2000; In final form 20 February 2001)

Wetting dynamics on machined wood surfaces is of great interest for the adhesive bonding technology of wood. In this work, the change of apparent contact angles with time of phenolresorcinol-formaldehyde (PRF), polyvinyl-acetate (PVAc), and a series of probe liquids on sawed, planed, sanded, and razor-blade-cut wood surfaces of Southern pine was studied by the sessile drop method using a CCD camera technique. The results indicate that the fastest wetting of the probe liquids occurs on the sanded surfaces because of higher surface roughness and the resulting increase in capillary forces as compared with the sawed, planed and razor blade cut surfaces. The fastest wetting of the PRF and PVAc resins occurred on the comparably smooth planed and razor-blade-cut wood surfaces. A smoother wood surface seems to provide better wetting and penetration properties for high-viscosity liquids such as adhesives, which probably can be attributed to less entrapment of air between the resin and the wood structure.

Keywords: Adhesive wettability; PRF; PVAc; Contact angle; Wood; Machined wood surfaces

INTRODUCTION

The physical quality (or structure) of a surface is of prime importance for adhesion phenomena [1]. The structure of a wood surface is complex and relates mainly to the intrinsic wood anatomy and to the

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*Corresponding author. Tel.: +46 8 790 90 96, Fax: +46 8 21 81 81, e-mail: stehr@woodtech.kth.se mechanical treatment that was used to prepare the surface. Different machining operations, *e.g.*, sawing, sanding and planing, of wood generally result in different levels of mechanical damage of the wood cells [2–10].

The term wetting can be used to describe what happens when a liquid comes in contact with a solid surface [11]. For rough and porous materials, such as wood, several wetting phenomena may occur. For example, Patton [12] used the term wetting to cover the processes of interfacial adhesion (or surface thermodynamics), penetration and spreading. Similarly, Berg [13] defined wetting as "macroscopic manifestations of molecular interaction between liquids and solids in direct contact at the interface between them", involving (1) the formation of a contact angle, θ, at the solid/liquid/fluid interline, (2) the spreading of a liquid over a solid surface, and (3) the wicking (penetration) of a liquid into a porous solid. However, in this definition, wetting does not include dissolution or swelling of the solid by the liquid or any kind of chemical reaction between the materials that changes the system composition.

Of particular importance in the present study is the fact that when a liquid contacts a porous medium such as wood, and the contact angle formed on the material is less then 90° (see Washburn's equation [14]), then spontaneous wicking (penetration) of the liquid into the porous medium will occur because of capillary forces. Similarly, the surface structure of wood's cellular elements, *e.g.*, like tracks in a gramophone record, also promotes wicking of the liquid along the surface.

Moreover, it is obvious that liquid wetting on substrates such as wood is complex and influenced by surface thermodynamics and other factors including surface roughness, porosity, heterogeneity, bulk sorption, liquid viscosity (especially regarding systems involving adhesives and coatings), reorientation of functional groups at the wood-liquid interface, and influences related to contamination of the liquid by wood extractives [15–18]. Because of the complexities of wood-liquid relations, when measuring wetting parameters, such as the contact angle, on wood it is preferably to use, *e.g.*, the expression *apparent* contact angle, in contrast to the ideal contact angle as defined by the Young equation.

In recent years, there has been increasing interest [19–24] in measuring the apparent contact angle change on wood as a function of time (the dynamic contact angle; the dynamic liquid wetting process).

Liptáková and Kúdela [19] studied the change of water droplet shape with time on microtomed beech wood, and observed a distinct decrease of the apparent contact angle with time. However, three different stages of the droplet diameter were also observed: an initial stage where the diameter increases, which most probably is due to a gradual spreading over and soaking into the voids of the irregular wood surface; a second stage where the diameter remains constant, presumably starting at the moment when the void spaces are filled; and a third stage where the diameter decreases, presumably starting when the advancing contact angle is changed to the receding one. Based on this, it was argued that an "equilibrium" contact angle could be estimated at the moment when the droplet diameter starts to decrease. Boehme and Hora [21] found a linear decrease of the contact angle *versus* the square root of time, which also was obtained and discussed by Hora [25] on wood topcoats. Shi and Gardner [24] proposed a dynamic wettability model to provide a description and quantification of the wetting and penetration process of adhesives on wood surfaces. Liptáková *et al.* [20] studied the influence of mechanical surface treatments of beech wood on the water wetting process. It was concluded that the mechanical treatment of the wood surfaces changed both their structural and chemical composition, and that sanding and planing increased and decreased the water wettability, respectively. In a study of static apparent contact angles of phenol-formaldehyde (PF) resins on pine veneers [20], it was demonstrated that the angles were lower on the rougher earlywood than on the smoother latewood. Similarly, it was also reported [27] that apparent water contact angles on sawed, planed and sanded beech wood increased with decreased roughness. Based on a dynamic technique to measure apparent contact angles, Scheikl and Dunky [22] also observed lower angles of water on the rougher earlywood than on the smoother latewood of spruce and pine, but no influence of roughness was found regarding high-viscosity urea-formaldehyde (UF) resins. The wetting and penetration rate of the UF resins, however, decreased significantly with an increase in molecular size and viscosity.

The objective of this study was to evaluate the dynamic wettability of four different machined wood surfaces using a series of standard probe liquids and two common adhesive types; polyvinyl-acetate (PVAc) and phenol-resorcinol-formaldehyde (PRF).

Studies of the influence of the surface structure of wood on the adhesive wetting process may generate important information about the adhesive bonding technology of wood. For example, insight about the wood-adhesive wetting process, *e.g.*, the adhesive spreading and penetration properties on wood, may be used to further develop the processing of glued wood products, and to enhance the in-service durability of a cured wood-adhesive bond.

MATERIALS AND METHODS

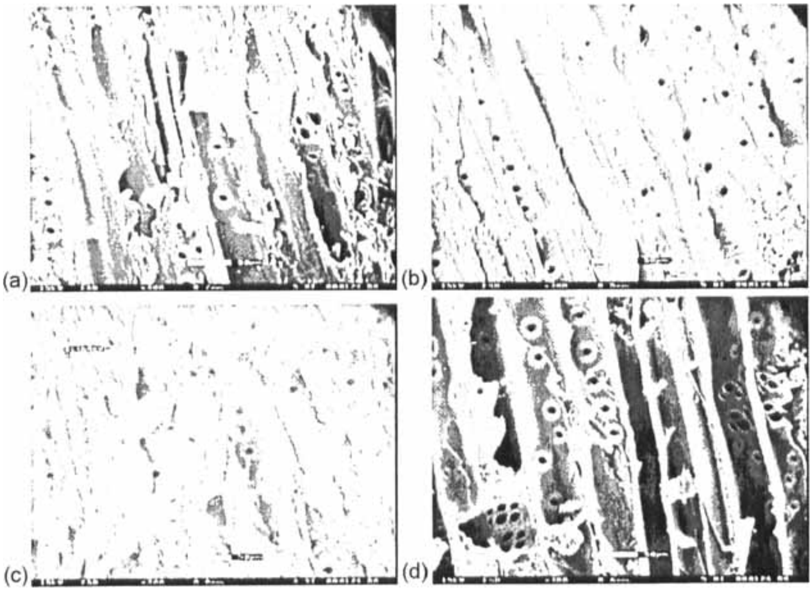
Materials and Sample Preparation

The wood material used was clear sapwood of Southern pine (*Pinus spp*), with a mean density $\rho_8 = 684\,\mathrm{kg/m^3}$ conditioned to moisture content of about 8%. The investigation comprised 28 specimens, which were first prepared by sawing (with horizontal annual rings) using a carbide-tipped circular saw blade, to the approximate dimensions $10 \times 3 \times 1\,\mathrm{cm}$ (L × R × T), where only the radial sections and their earlywood portions were studied. The specimens were divided into four groups depending on the mechanical treatment: sawing, planing, sanding and razor blade cut. Figure 1 shows environmental scanning electron microscope (ESEM)-micrographs which exemplify the studied wood surfaces. In each group, one specimen was used for each of the two adhesives and the five probe liquids.

The equipment used for the wood machining processes was ordinary woodworking machines and a razor blade of steel, respectively. Each machining process began with circular sawing followed (in the last three groups) by additional machining (planing, sanding with a 80-grit paper, and razor blade cutting). All treatments were done along the fibre direction. After each machining process, the wood dust was removed from the specimens with a brush. The razor blade cutting was done on an un-wetted specimen.

Two common adhesives in the wood industry were used in the wetting experiments: (1) a phenol-resorcinol-formaldehyde (PRF), Resorsabond 4242 (Georgia Pacific) without hardener; and (2) a polyvinyl-acetate (PVAc), Elmer's E-701 (Borden). The major specifications of the two adhesives are shown in Table I.

Wetting experiments were also performed using four polar probe liquids: formamide (FM), ethylene glycol (EG), glycerol (GL), water



FIGURES 1a–d ESEM-micrographs of the different earlywood surfaces after (a) sawing (b) planing (c) sanding and (d) razor-blade-cutting, respectively.

TABLE I Specifications of the PRF and PVAc adhesives used in the experiments

Specification	*PRF*	*PVAc*
Solubility in water	partial	dispersible
Specific gravity	1.15–1.16	1.08
Viscosity (25°C) [mPa s]	1200–2000	7000
Surface tension [mN/m]	~60	~53
PH	8.8–8.9	5.0
Non-volatile content [%]	53.5–54.5	52

(WT), and one apolar liquid: α-bromonaphthalene (BN). A specification of their surface tension and viscosity is shown in Table II. All liquids were of HPLC grade and purchased from Sigma-Aldrich (USA).

Measurements of Apparent Contact Angles

To determine the apparent contact angles, drop profile measurements were performed. Drops of the adhesives and the probe liquids were placed on the wood surface with a microsyringe. The apparent contact angle measurements were made within a time frame of 30 minutes and

TABLE II Literature values [28] of surface tension, γ, and of viscosity, η, at 20°C, of the probe liquids used in the experiments

Liquid	*Surface tension,* γ *(mN/m)*	*Viscosity,* η *[mPa s]*
Water	72.8	1.0
Ethylene glycol	48	19.9
Formamide	58	4.55
Glycerol	64	1490
α-Bromonaphthalene	44.4	4.89

4 hours after the particular machining process, in an effort to reduce the influence of surface ageing. The adhesives/liquids were put on the wood surface in the following order: FM, EG, GL, BN, WT, PRF, and PVAc.

Each drop was placed on an earlywood portion of a growth increment on the radial section of the specimens. The drop volume was 0.005 millilitres. However, it should be mentioned that it is difficult to create an exact volume for the viscous adhesives.

The drop profile – along the grain of the wood – and its change with time was imaged and recorded by a CCD camera connected to a VCR. The recording was stopped when the apparent contact angle either reached a zero or an "equilibrium" value. The recording was, however, never longer than 120 seconds. The instantaneous (initial) apparent contact angle was measured at the moment when the drop detached from the microsyringe and formed a spherical-shaped drop on the surface.

The apparent contact angles at the times 0, 2, 5, 12, 25, 50, 80 and 120 seconds were determined, using computer software, as the angle between the tangent (manually drawn) to the liquid surface at the solid-liquid-air point of contact. Each contact angle value was averaged from measurements on both ends of a drop. Because the recording was stopped after the "equilibrium" or zero angle had occurred, or after 120 seconds had elapsed, the actual number of data points differed from 2 to 8 depending on the adhesive and liquid evaluated. The number of recorded drops on each surface is given in Table III. The reason for the lower number of drops on the razor-blade-cut surfaces was the difficulties in producing sufficient plane surfaces (since the wood was not wetted before cutting). The results from this group should, therefore, be looked upon with some caution.

TABLE III The number of recorded drops on each surface

Mechanical treatment	*FM*	*EG*	*GL*	*BN*	*WT*	*PRF*	*PVAc*
Sawing	10	10	10	10	10	9	10
Planing	11	10	10	12	11	9	10
Sanding	10	11	10	11	13	12	10
Razor-blade-cut	5	7	8	9	7	9	8

Where FM = formamide, EG = ethylene glycol, GL = glycerol, BN = α-bromonapthalene, WT = water, PRF = phenol-resorcinol-formaldehyde resin, and PVAc = polyvinyl-acetateglue.

Dynamic Wettability Model

In the present study, a dynamic wetting model as suggested by Shi and Gardner [24] was applied. The model was specifically developed for measuring adhesive wettability. This model is described as follows [Eqs. (1)–(4)]. When a liquid drop is placed on a porous surface, in addition to forming a contact angle at the liquid/solid surface, liquid penetration together with liquid spreading also occurs. Under such conditions, the apparent contact angle changes relative to the rate of liquid penetration and spreading. At the initial stage of the wetting process, the apparent contact angle of a liquid drop decreases quickly. As time elapses, this angle decreases more slowly and may finally also attain a relative equilibrium. For an ideal liquid and solid system, the penetration and spreading rate depends on the drop shape at a particular moment in time which can be expressed as:

$$\frac{d\theta}{dt} = -K\theta \tag{1}$$

in which K is a constant referring to the relative decrease rate of the apparent contact angle (the penetration/spreading constant). The physical meaning of K represents how fast the liquid spreads and penetrates into the porous structure of wood. By knowing the K value, spreading and penetration in adhesive/solid systems can be quantified.

As the contact angle change rate decreases because of less spreading and penetration and the change rate tends to be zero at infinity, a limitation term is added to Eq. (1):

$$\frac{d\theta}{dt} = -K\theta \cdot \left(1 - \frac{\theta_i - \theta}{\theta_i - \theta_e}\right) \tag{2}$$

in which θ_i represents the instantaneous (initial) contact angle, and θ_e represents the equilibrium contact angle. Reorganizing Eq. (2) leads to the expression:

$$\frac{d\theta}{dt} = K\theta \cdot \left(\frac{\theta_e - \theta}{\theta_i - \theta_e} \right) \tag{3}$$

After integration, the final expression of the wetting model is:

$$\theta = \frac{\theta_i \theta_e}{\theta_i + (\theta_e - \theta_i)e^{K(\theta_e/(\theta_e - \theta_i))t}} \tag{4}$$

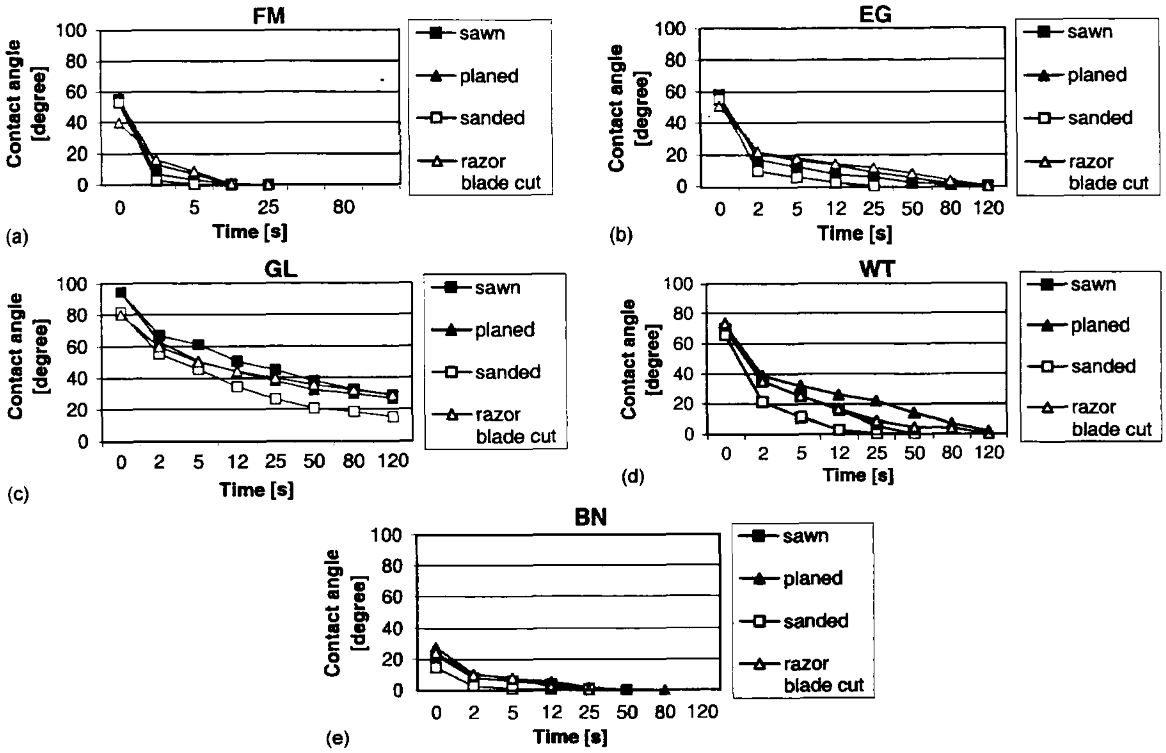
The Marquardt-Levenberg algorithm was used to obtain the K value that provides the best fit between Eq. (4) and the data. This algorithm seeks K values that minimize the sum of the squared differences, SS, between the observed and predicted values of the dependent variable:

$$SS = \sum_{i=1}^{n} (y_i - \hat{y}_i) \tag{5}$$

where y_i and $\hat{y}_i$ are the observed and predicted values of the dependent variable, respectively. The coefficients of variation (CV) of the K value and R^2 were calculated based on the curve-fitting results. It should be noted that when the measured apparent equilibrium contact angle is zero, a contact angle of 0.01 degree must be input into Eq. (1) in order to solve for the K value.

RESULTS AND DISCUSSION

Figure 2 shows mean values of the measured apparent contact angles as a function of time for each probe liquid on the different machined wood surfaces. As shown in this figure, the apparent contact angle changes rapidly as function of time for all probe liquids on all wood samples. It is obvious that the decrease is more rapid between the two first measurement points (0 and 2 seconds) compared with the succeeding measurements. From Figure 2 it is also evident that no equilibrium state is achieved for the apparent contact angles and that they, in all cases, except for glycerol due to its high viscosity, reach zero values within the 120-second time frame of the experiment.



FIGURES 2a–e Mean values of the apparent contact angle as a function of time for the five probe liquids on different mechanical prepared wood surfaces. Probe liquids: Formamide (FM), ethylene glycol (EG), glycerol (GL), water (WT), and one apolar liquid: α-bromonaphthalene (BN).

The measured average "initial" apparent contact angle, θ_i, and the calculated K values for the probe liquid wetting process on the different machined wood surfaces are shown in Table IV. As can be seen in this table, sanding generates the highest K value for all the probe liquids except for glycerol. As indicated in Figure 1, it seems as sanding, compared with the other machining processes, produces the most damaged and roughened surface structure. This is also in accordance with the study performed by Stewart and Crist [3]. Consequently, such roughness, and the resulting increase in surface area, may cause a liquid to move more easily due to capillary forces. This could explain why a higher K value (and lower contact angles) is observed for the wetting process of the low-viscosity liquids on the sanded surfaces, which also agrees well with observations in other studies [20, 22, 27]. However, capillary movement and penetration of liquids into porous

TABLE IV Measured average "initial" (instant) θ_i and apparent contact angles (in degrees) and the calculated K values for the different machining processes and the probe liquids. Standard deviations are given in parentheses

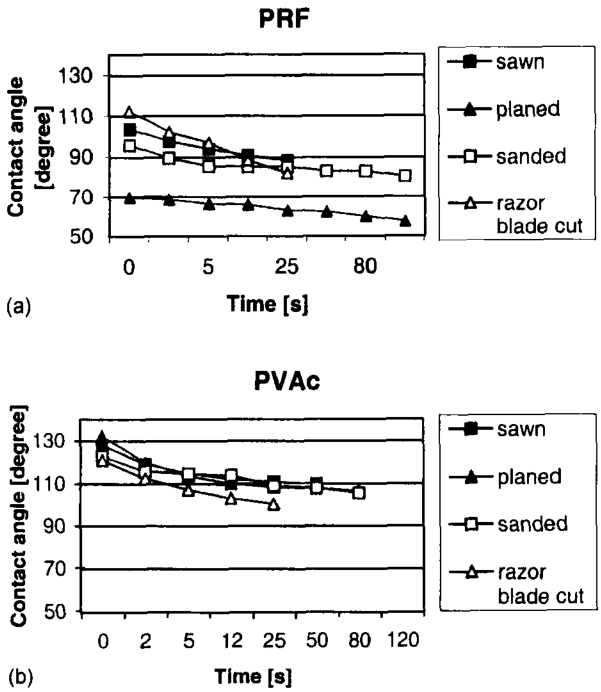
Probe liquid	*Machining process*	θ_i	K [1/*sec.*]	*CV* [%]	R^2
FM	Sawing	55(9)	3.4	7.6	0.99
	Planing	56(6)	1.7	12.1	0.99
	Sanding	53(4)	10.4	20.3	0.99
	Razor-blade-cut	40(8)	0.8	15.6	0.99
EG	Sawing	58(8)	0.9	12.7	0.99
	Planing	54(6)	0.5	19.4	0.96
	Sanding	55(6)	2.3	7.7	0.99
	Razor-blade-cut	51(6)	0.4	24.2	0.91
GL	Sawing	95(11)	0.1	20.3	0.91
	Planing	95(4)	0.2	17.1	0.95
	Sanding	82(7)	0.2	12.1	0.98
	Razor-blade-cut	80(10)	0.1	19.2	0.92
BN	Sawing	23(3)	0.7	13.4	0.99
	Planing	28(4)	0.7	13.9	0.98
	Sanding	15(4)	2.6	8.7	0.99
	Razor-blade-cut	24(2)	0.6	18.8	0.98
WT	Sawing	70(4)	0.4	11.4	0.99
	Planing	72(7)	0.2	22.9	0.91
	Sanding	66(8)	1.1	8.5	0.99
	Razor-blade-cut	74(16)	0.4	10.1	0.99

Where FM = formamide, EG = ethylene glycol, GL = glycerol, BN = α-bromonapthalene, WT = water, CV = coefficient of variation, and R^2 = model fit.

media is also strongly related to the viscosity of the liquid, *i.e.*, the higher the viscosity the slower the movement (see Washburn equation [14]) which could account for the distinctly different behaviour of glycerol compared with the other probe liquids. It is also notable that the wetting of glycerol generally results in the highest θ_i and the lowest K values, which validates the strong influence of the viscosity on the dynamic wettability on rough and porous surfaces like wood, as reported by Scheikl and Dunky [22]. It is, however, evident that the wetting process model used in the present study does not account for influences related to the viscosity of the liquid. Further development of the model should, therefore, be performed.

Additionally, from Table IV, it is clear that the wetting of formamide on all samples results in the highest K values. Formamide is a strong hydrogen-bonding liquid, and it has been demonstrated [17, 18] that the formamide wettability of wood is strongly influenced by sorption and contamination effects. The first effect is related to bulk sorption of the liquid with resulting swelling of the wood substance, and the latter effect is due to dissolution or presence of wood extractives at the wood-liquid interface, which radically reduces the liquid surface tension. Accordingly, a reduction in surface tension results in an increase in the spreading and penetration rate, which probably is the cause of the overall higher wetting rates of formamide compared with those of water, ethylene glycol and α-bromonaphthalene. It is also interesting to note that the wetting rates of ethylene glycol and water are approximately in the same range as those of α-bromonaphthalene, despite the fact that the latter liquid has lower surface tension and exhibits significantly lower θ_i values on the wood surfaces than the two former. Both water and ethylene glycol are polar liquids, and their comparatively fast wetting rates may, therefore, be attributed to their ability to hydrogen bond to the polar wood substance.

Figure 3 shows mean values of the apparent contact angle as a function of time for both of the adhesive systems. As can be seen, the change of the angles over the whole measurement range is less compared with the low-viscosity probe liquids. However, similar to the wetting process of the probe liquids, no evident equilibrium stage of the angles was observed (see especially the curves for PRF and PVAc for sawn, planed and razor-blade-cut surfaces). Shi and Gardner [24], on the other hand, observed a more pronounced final equilibrium



FIGURES 3a - b Mean values of the apparent contact angle as a function of time for the phenol-resorcinol-formaldehyde (PRF) and polyvinyl-acetate (PVAc) adhesives on different mechanically prepared wood surfaces.

state of the dynamic wetting process of polymeric diphenylmethane diisocyanate (PMDI) and phenol-formaldehyde (PF) resins on razor-blade-smoothened Southern pine and Douglas-fir. However, it should be noted that the viscosity of these two adhesives are an order of magnitude lower than those of the PRF and PVAc adhesives used in the present study. It is also important to mention that during the 120-second time frame of the experiment, the PRF and PVAc drops appeared to be coagulated as a result of the water (solvent) penetrating into the wood structure.

PRF exhibits lower apparent contact angles, on the corresponding surfaces, than the PVAc. This is not surprising as PRF has distinctly lower viscosity than PVAc. In the PRF case, significantly lower apparent contact angles are observed on the planed surface compared with the other surfaces. However, it must be remembered that an apparent contact angle on wood, in addition to surface thermodynamics, is influenced by many factors such as surface roughness, porosity and liquid contamination [15, 17, 18]. The absolute values of the measured angles should, therefore, be considered with some caution.

The measured average "initial" and "equilibrium" apparent contact angles and the calculated *K* values for the adhesive wetting process on the different machined wood surfaces are shown in Table V. As can be seen, the *K* values are, as expected, several orders of magnitude lower for the adhesive compared with those for the probe liquid wetting process. It is also indicated that the adhesive wetting on the planed surfaces results in the highest *K* values, which implies that planing in this case provides the surface with the fastest adhesive spreading and/or penetration. Additionally, it is interesting to note that, in contrast to the wetting processes of the low-viscosity probe liquids, the adhesive wetting on the sanded surfaces results in the lowest *K* values, which implies that this process provides the surfaces with the slowest adhesive spreading and/or penetration. As discussed above and as indicated in Figure 1 sanding produces the most damaged and rough surface. In the case of spontaneous spreading and penetration of high-viscosity liquids, such as the PRF and PVAc resins, on substrates like wood, it is probable that increased surface roughness could cause increased entrapment of air between the resin and the wood structure, thus resulting in a slower wetting rate, compared with that on smoother surfaces. This argument could also be in agreement with the comparably high *K* values observed for the comparably smooth planed

TABLE V Measured average "initial" (instant) θ_i and "equilibrium" θ_e apparent contact angles (in degrees) and the calculated *K* values for the different machining processes and the two adhesives. Standard deviations are given in parentheses

Adhesive	*Machining process*	θ_i	θ_e	$\theta_i - \theta_e$	*K* [1/*sec.*]	*CV* [%]	R^2
PRF	Sawing	104(10)	88(4)	16	0.031	7.6	0.99
	Planing	70(16)	58(0)	12	0.050	12.8	0.95
	Sanding	96(17)	80(12)	16	0.026	32.3	0.71
	Razor-blade-cut	113(11)	82(9)	30	0.045	8.1	0.99
PVAc	Sawing	128(10)	110(4)	18	0.041	11.0	0.98
	Planing	133(8)	107(8)	26	0.057	11.7	0.98
	Sanding	123(23)	106(10)	18	0.012	25.2	0.84
	Razor-blade-cut	121(10)	101(3)	21	0.042	6.9	0.99

Where CV = coefficient of variation, and R^2 = model fit.

TABLE VI A qualitative ranking among the different machining processes and adhesive respectively [points]

Adhesive	*Machining process*	*K*	θ_i	θ_e	$\theta_i - \theta_e$	Σ
PRF	sawing	3	3	4	2	12
	planing	1	1	1	3	**6**
	sanding	4	2	2	2	10
	razor-blade-cut	2	4	3	1	10
PVAc	sawing	2	3	4	3	12
	planing	1	4	3	1	**9**
	sanding	3	2	2	4	11
	razor-blade-cut	2	1	1	2	**6**

Where θ_i = the initial apparent contact angle, θ_e = the apparent equilibrium contact angle.

and razor-blade-cut surfaces (Fig. 1), regardless of adhesive type, as compared with the sanded surfaces. Except for the sanded surfaces, the R^2 for the wetting-model-fit to the experimental data are at least 0.95. It should also be noted that the sanded surfaces exhibit the highest coefficient of variation.

In Table VI the results from the investigation are summarised in a qualitative ranking among the different machining processes. The rankings are based on *K* value, initial and equilibrium contact angles, and decrease in contact angle. In this case, the lower *K* values, the higher initial and apparent equilibrium contact angles and the smaller difference in apparent contact angles contribute to the larger ranking points. A low total number of summed points indicates better adhesive wettability. According to the qualitative ranking, the planed surface provides the fastest wetting with regard to the PRF resin. In the PVAc case, the razor-blade-cut surface, followed by the planed surface, provides the fastest wetting.

CONCLUSIONS

The wetting dynamics of phenol-resorcinol-formaldehyde (PRF), polyvinyl acetate (PVAc) and a series of probe liquids on sawed, planed, sanded and razor-blade-cut wood surfaces of Southern pine was studied. The results indicate that the fastest wetting of the probe liquids occurs on the sanded surfaces because of the greater surface roughness and the resulting increase in capillary forces as compared with the smoother surfaces. In contrast to this, the fastest wetting of the PRF and PVAc resins occurred on the planed wood surfaces and on the razor-blade-cut surfaces. A smoother wood surface provides improved wetting and penetration properties for high-viscosity liquids such as adhesives. The evaluated wetting process model does not account for influences related to the viscosity of the liquid. Further development of the model should, therefore, be performed.

Acknowledgements

The authors thank Dr. Sheldon Shi and William Tze M.Sc. for fruitful discussions. The Swedish Foundation for Strategic Research–Wood Technology, Träforsk (Association for Swedish Wood Products Research), Stiftelsen Gunnar Sundblads Forskningsfond, and the Swedish National Board for Industrial and Technical Development (NUTEK) are acknowledged for financial support.

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