Wettability of Sanded and Aged Fast-growing Poplar Wood Surfaces: II. Dynamic Wetting Models

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The dynamic wettability of adhesive on sanded and aged wood surfaces was measured using the sessile drop method. Four different models were used to evaluate and compare the wetting process. It was shown that the wettability of freshly sanded wood and aged wood both decreased compared to the control wood. There was no evidence of change in wettability with increasing grit number. Aging reduced the wettability of the wood surface. The coefficients of determination (R^2) for all four models were over 90%, and that of the Modified model was 99%. The models can be used to accurately describe the adhesive wetting process. The wettability of water and adhesive on the fresh surface were different, and the wettability of the adhesive increased as grit number increased. On the contrary, the wettability of water decreased as grit number increased, and the same trend was found for the water and the adhesive on the aged wood surface. Advantages and disadvantages were found for each model, but the Modified model needs to be verified by additional experiments.

Keywords: Poplar wood; Sanding; Contact angle; Wettability; Models

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INTRODUCTION

Wettability is a term used to describe the interfacial phenomenon of a liquid contacting a solid surface (Baldan 2012). When a liquid wets a solid surface, three effects can be observed: (1) the formation of an interface between the solid surface and the liquid drop; (2) the spreading of the drop on the solid surface; and (3) the penetration of the liquid into the wood. The formation of an interface (*i.e.*, formation of a contact angle) is related to the interface thermodynamic properties of a liquid-solid contact. Spreading is based on changes to the solid surface free energy, absorption, and kinetics of wetting. Penetration is related to the surface's morphological structure and only occurs on porous solid surfaces.

Wood can be viewed as a porous, heterogeneous, complex composite material of cellulose, hemicellulose, lignin, and extractives. These polymeric compounds are arranged in a cellular structure, resulting in surface roughness on a microscopic scale. The wettability of wood is influenced not only by surface thermodynamics, but also by factors such as surface roughness, wood species (de Meijer *et al.* 2000; Gardner *et al.* 1991), location of wood (sapwood and heartwood), pH value (Gindl and Tschegg 2002), aging time of exposed surface (Gindl *et al.* 2004), machining conditions (Stehr *et al.* 2001; Santoni and Pizzo *et al.* 2011), treatment and drying methods (Wang *et al.* 2007), and amount of extractives (Hakkou *et al.* 2005). In addition to the properties of wood, the properties of the adhesive (*e.g*., viscosity, type, acidity, and surface tension) also influence the wettability.

Wettability can be evaluated by measuring the changes in contact angle as a function of time on a wood surface using the sessile drop method, a direct method for evaluating the wetting process. Many different models have been used to evaluate the wettability of wood. Shupe *et al.* (2001) studied the wettability of sanded and non-sanded hardwoods by measurement of contact angles after 5 s. They also studied the effect of veneer side (tight or loose) and wood grain (earlywood or latewood) on the wettability of loblolly pine veneer. Maldas and Kamdem (1998) and Cao *et al.* (2005) used both the initial contact angle, which is the intercept value of the regression line of the contact angle values over time, and the rate of decrease in contact angle to evaluate the wettability of chromated copper arsenate (CCA) treated wood. Nussbaum (1999) used a differential method for evaluating the contact angle measurement. He considered the “constant wetting rate angle” (CRWA) as the values determined when the wetting rate becomes constant. Liptakova and Kudela (1994) used the contact angle corresponding to an ideally smooth wood surface to study the wettability and then calculated the surface free energy. Buyuksari *et al.* (2011) obtained the contact angle from the average of measurements over a 10-s period to investigate the wettability and surface roughness of natural and plantation-grown narrow-leaved ash wood. Boehme and Hora (1996) observed that the contact angle decreased with the square root of time, while Shi and Gardner (2001) proposed a Shi-Douglas wetting model (S-D model) that is commonly used to evaluate the dynamic wetting process.

The wettability of wood is a useful parameter that provides information about the physical and chemical affinity between the wood surface and adhesives and has a great influence on the bonding strength of wood composites. Most studies on wettability have been carried out on softwoods, primarily pine and fir, or tropical species. Many authors have investigated the relationship between the wettability and surface roughness of wood panels (Ayrilmis 2010; Buyuksari *et al.* 2010; Unsal *et al.* 2011; Akgul *et al.* 2012; Candan *et al.* 2012). On the contrary, very few studies have been carried out on fast-growing poplar wood. A suitable wettability is an essential factor for good bonding strength or high-strength glue joint. The objective of this study was to investigate and compare the wettability of phenol formaldehyde (PF) adhesive on sanded and aged fast-growing poplar wood samples using different models.

EXPERIMENTAL

Materials

Fast-growing poplar used in the experiment was obtained from a local woodworking factory (Heibei, Wen’an County). The average density of the poplar was 408 kg/m^3, with a standard deviation of 26 kg/m^3.

Samples with 8 to 10% moisture content were cut parallel to the grain and stored under a control condition of a temperature and relative humidity of 20 °C and 65%, respectively. The samples were first planed and then sanded with different dry sanding papers. Each sample was sanded 40 times parallel to the sample with grit numbers of 60, 120, 180, and 240, and the wood dust was cleared with a small brush carefully. The sanded samples were then left to age for 10 days in air (aged sanded wood), while the fresh wood samples were sanded without ageing. Only heartwood was used to avoid experimental error between the sapwood and heartwood. The final dimensions of the samples were 50 mm × 25 mm × 2.5 mm and four replicates were tested.

The PF adhesive was chosen to investigate the dynamic wettability of adhesive on wood. The PF adhesives with formaldehyde/phenol (F:P) molar ratios of 2.25 were prepared in the laboratory. A mixture of phenol (98%), a catalyst (sodium hydroxide solution, 40%), and a formaldehyde aqueous solution (37%) was added to a reactor and stirred uniformly, heated to a temperature of 90 °C, maintained at this temperature for 50 min, then cooled to 80 °C. The second portion of the formaldehyde aqueous solution and catalyst was then added to the reactor, which was heated and maintained at 90 °C for 60 min. The viscosity, solids content, and pH values were 102.6 mPa/s, 44.40%, and 11.8, respectively.

Methods

Contact angle measurement

Contact angle measurements on tangential surfaces of samples were performed with an optical contact angles apparatus (OCA 20, DatapPhysics Instruments GmbH, Filderstadt, Germany) equipped with a video measuring system with a high-resolution CCD camera and a high-performance digitizing adapter that enables instantaneous and frequent registration. SCA 20 software (DatapPhysics Instruments GmbH, Filderstadt, Germany) was used for data acquisition. Sessile droplets (3 μL, measured with a microsyringe) of the PF adhesive were placed on the wood surface, the right and left angles of the drops on the surface were collected at intervals of 0.1 s for a total duration of 120 s, and the average of the angles was calculated. A minimum of 10 droplets was examined for each wood sample. All the contact angles were observed parallel to the macroscopic fiber orientation. The contact angles of fresh wood were detected within 10 min of the sanding treatment, so the possibility of aging on treated surfaces was minimized.

Three drops per sample were captured for the adhesive, four samples were used, and twelve measurements of contact angle were obtained.

Contact angle time characteristics

When a liquid drop of adhesive was placed on the wood surface, a contact angle was formed, but spreading and penetration were also found. Several models were used to evaluate the contact angle measurement by employing differential methods. The wetting rate depends on the contact angle at a particular time, which can be expressed as,

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

where θ is the contact angle, t is the time, and K is the contact angle change rate constant. The K value measures how fast the liquid spreads and penetrates; higher values correspond to a shorter time required for the contact angle to reach relative equilibrium and for the liquid to spread and penetrate.

The wood sample sanded by a grit number of 240 and aged was taken as an example to investigate the models. The values were calculated using the least squares method to fit the equation by Origin 8.0 (Origin Lab, USA). Many studies have improved the basic equation (Eq. 1) to better investigate the change in contact angle with time. The models can be described as follows:

Nussbaum Model (Nussbaum 1999)

A well-defined contact angle is determined when the wetting rate becomes constant, *i.e.*,

$$\frac{d\theta}{dt} = \text{constant} \quad (2)$$

The contact angle obtained under such conditions is called the "constant wetting rate angle" (CWRA). The constant wetting rate angle is used to evaluate the wettability (Fig. 1).

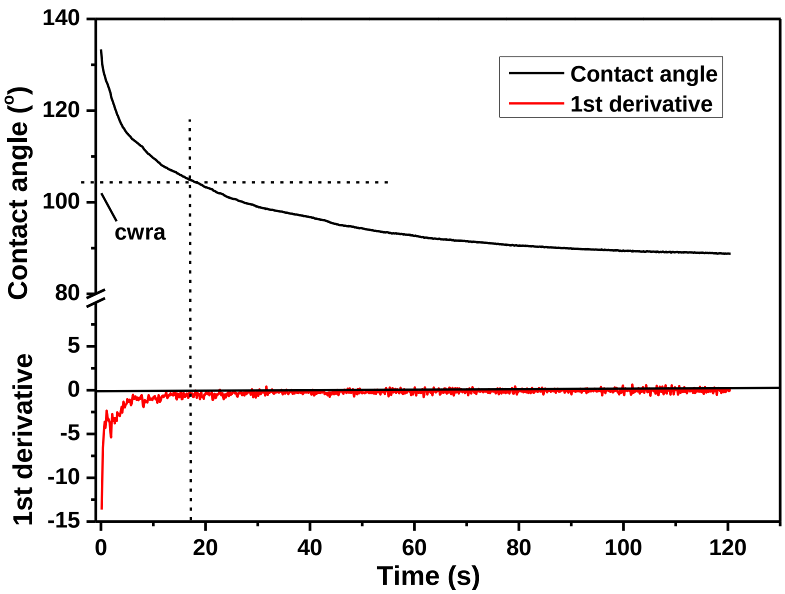

Fig. 1. Determination of the constant wetting rate angle (CWRA) from a plot of the contact angle as a function of time, and a plot of the wetting rate *versus* time

S-D Model (Shi and Gardner 2001)

As the contact angle change rate decreases due to less spreading and penetration and tends to be zero at infinity, a limitation term was added to Eq. 1,

$$\frac{d\theta}{dt} = -K\theta \cdot \left(1 - \frac{\theta_i - \theta}{\theta_i - \theta_e}\right) \quad (3)$$

where θ_i represents the initial contact angle, θ_e is the equilibrium contact angle, θ is the contact angle at a certain time, t is the wetting time, and K is a constant referring to the intrinsic relative contact angle decrease rate.

After integration, the final expression of the S-D wetting model (Shi and Gardner 2001) can be expressed as Eq. (4):

$$\theta = \frac{\theta_i \times \theta_e}{\theta_i + (\theta_e - \theta_i)\exp\left[K\left(\frac{\theta_e}{\theta_e - \theta_i}\right)t\right]} \quad (4)$$

The wetting process for a wood sample using the S-D model (Shi and Gardner 2001) is shown in Fig. 2.

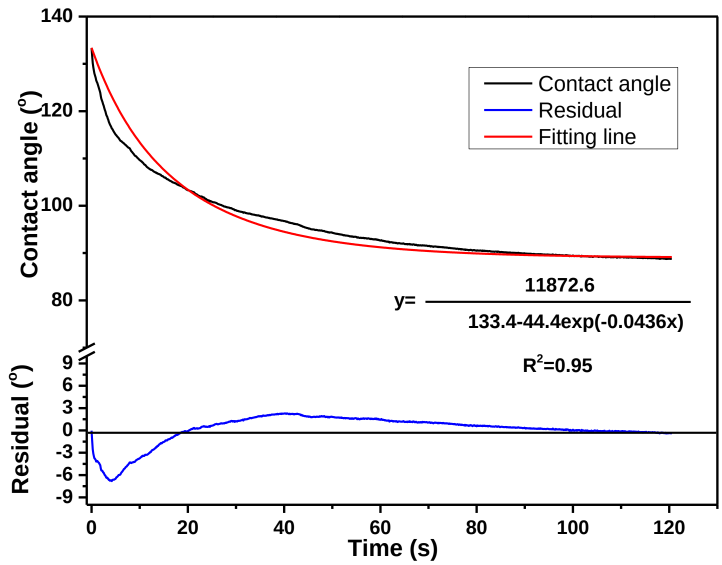

Fig. 2. Contact angle changes as a function of time on a poplar wood sample according to the S-D model; the residual contact angle is shown at the bottom of the graph.

Modified Model

Integrating Eq. 1 directly, the following is obtained,

$$\theta = A \cdot e^{-Kt} \qquad (5)$$

where A represents the integration constant.

A natural decay model in nuclear physics (Halliday *et al.* 1997) was used, and, similar to the S-D model, a limitation term of the initial contact angle, θ_i, was added to Eq. 5. The model has the following form:

$$\theta = \theta_i + A \cdot e^{-Kt} \qquad (6)$$

Zhou *et al.* (2007) added a limitation term of the equilibrium contact angle θ_e to Eq. 5:

$$\theta = \theta_e + A \cdot e^{-Kt} \qquad (7)$$

Topala and Dumitrascu (2007) introduced the following equation to describe the dynamics of the wetting process on dielectric barrier discharge (DBD) from treated wood surfaces,

$$\theta = \theta_i + A_1 \cdot e^{-K_1 t} + A_2 \cdot e^{-K_2 t} \qquad (8)$$

where θ_i is the asymptotic value of the contact angle at long times, A_1 and A_2 are the amplitudes, and K_1 and K_2 are the rates of spreading and penetration, respectively.

It is difficult to determine the initial contact angle or equilibrium contact angle because of the strong time-dependence of the contact values, so Eqs. 6 and 7 can be expressed as,

$$\theta = B + A \cdot e^{-Kt} \tag{9}$$

where B represents the limitation terms of the initial contact angle and equilibrium contact angle; when the time t is zero, $A+B$ is the initial contact angle, and when the time t is infinity, B can be defined as the relative equilibrium contact angle. This model was named the Modified model (Fig. 3).

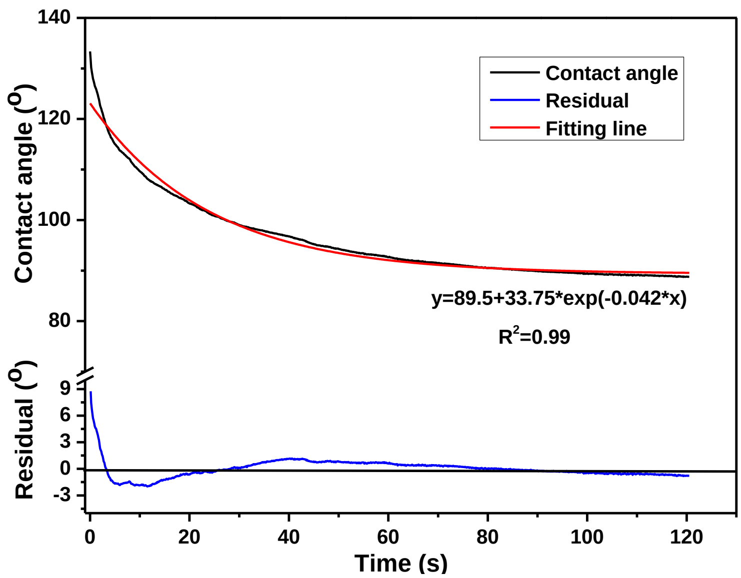

Fig. 3. Contact angle change as a function of time for a poplar wood sample according to the Modified model; the residual contact angle is shown at the bottom of the graph

Santoni Model (Santoni and Pizzo 2011)

A curve for the contact angle of a sanded and aged wood sample over time was obtained during measurements, as shown in Fig. 4. The curve of the contact angle as time elapsed could be divided into three temporal phases: (1) in the first phase, lasting a few seconds, the variation of the contact angle over time (dθ/dt) was rapid. In this phase, the liquid spread and filled the spaces provided by surface roughness; (2) in an intermediate phase, the drop settled and the angle decreased over time with variable speed; (3) in the final phase, the change in the contact angle *versus* time (dθ/dt) was considerably slower than that in the first phase, and it was constant until nearly complete absorption of the drop.

During the first few seconds, the wetting model can be expressed as:

$$\theta = -K_1 t + \theta_1 \tag{10}$$

Santoni and Pizzo (2011) evaluated the dynamic process of water on a wood surface during the first 5 s, and we studied the process during the first 3 s.

Meanwhile, the wetting model can be expressed between 20 and 120 s (while Santoni and Pizzo (2011) evaluated the process between 100 and 150 s) as follows,

$$\theta = -K_2 t + \theta_2 \tag{11}$$

where $K_1, \theta_1, K_2, \theta_2$ are the spreading wetting rate (also with some penetration), spreading initial contact angle, penetration wetting rate, and penetration initial contact angle, respectively.

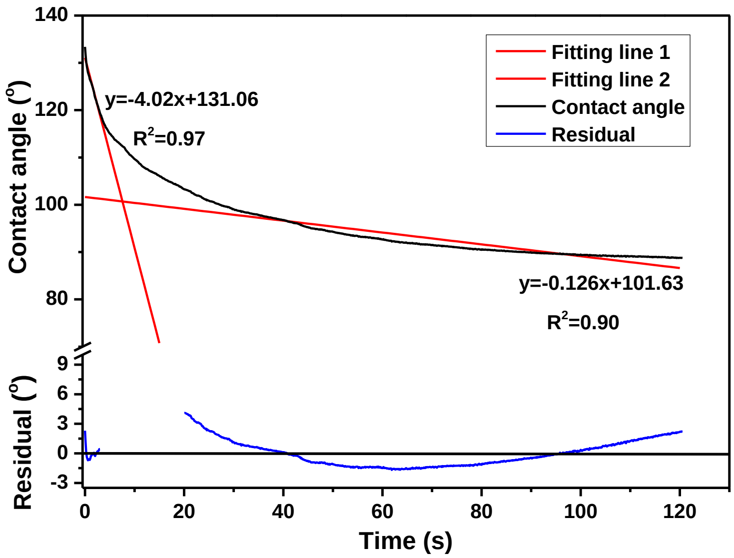

Fig. 4. Contact angle change as a function of time for a poplar wood sample according to the Santoni model; the residual contact angle is shown at the bottom of the graph

It can be seen from Figs. 2, 3, and 4 that the regression equations had high fitting degrees (all of the coefficient of determination R-squared values were over 90%), and the residuals of the model were between 0 and 9°. The four wetting models can provide an excellent fit to the experimental data. Therefore, they can accurately describe the adhesive wetting process on a wood surface.

RESULTS AND DISCUSSION

Nussbaum Model

The constant wetting rate angles of sanded and aged wood determined through the Nussbaum model (Nussbaum 1999) are shown in Fig. 5.

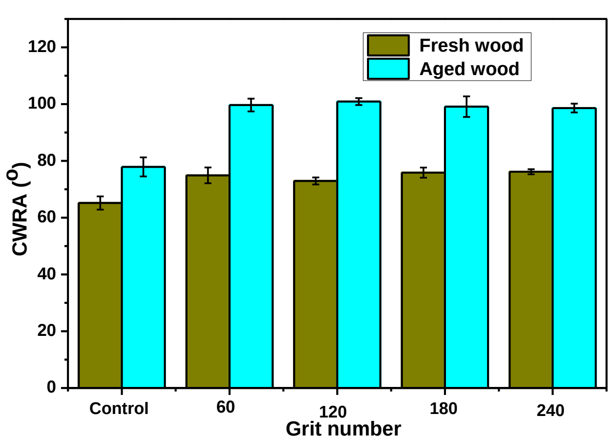

Fig. 5. Determination of the constant wetting rate angle (CWRA) from the Nussbaum model (Nussbaum 1999)

For the fresh wood, the CWRA increased after sanding; the range was between 65.17° and 76.18°, and the change in CWRA with increasing grit number was not significant for grit numbers of 120 and 240 (72.92° and 76.18°, respectively). For the aged wood, similar to the fresh wood, the CWRA increased, and the change was also insignificant. We have described before that the surface roughness decreased as grit number increased, and the CWRA increased as the roughness decreased. A similar occurrence was observed between the adhesive and wood surface roughness by Arnold (2011), who observed that the contact angle is lower when the surface roughness is rougher. Ayrilmis *et al.* (2010) found that a rougher surface is more wettable compared to a smoother surface in the case of medium density fiberboard. The CWRA means that the initial contact angle is spreading when the wetting rate ($d\theta/dt$) is a constant (*i.e.*, the decay of the contact angle as a function of time is linear). Therefore, we can conclude that the effect of grit number on the penetration for the sanded wood was not remarkable by looking at the Nussbaum model (Nussbaum 1999). However, the contact angle of water on the fresh wood surface decreased (see part I) as the grit number increased. It is concluded that the wettability of a wood surface is influenced not only by the surface free energy of wood but also by the viscosity, liquid surface tension, and the format employed for determination of the contact angle. An accurate explanation for this requires further study.

There was a significant difference in the CWRA of the fresh wood and that of aged wood, most likely caused by the natural migration of wood extractives to the surface. A similar occurrence was observed by Nussbaum (1999), who found a decrease in the wettability of a wood surface after sawing and aging.

S/D Model

The S-D model (Shi and Gardner 2001) is the most commonly used model (Hernández and Cool 2008; Huang *et al.* 2012; Stehr *et al.* 2001; Wei *et al.* 2012; Xu *et al.* 2010). By applying this model, the adhesive or coating wettability on different wood surfaces can be quantitatively evaluated. As shown in Fig. 6, for the fresh wood, the control wood sample had the highest K -value and the value of sanded wood decreased

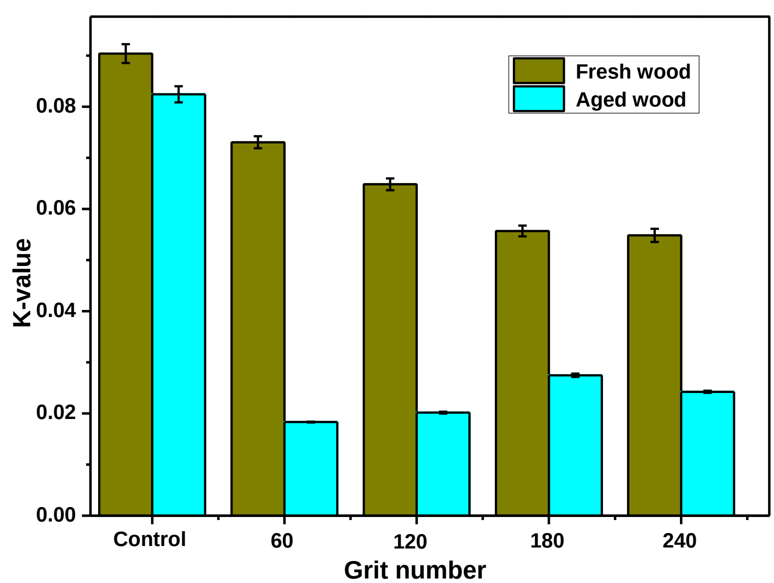

Fig. 6. K-value of wood samples according to the S-D Model

with increasing grit number, which indicated that a higher wood roughness resulted in a higher K -value, *i.e.*, surfaces sanded with a higher grit number had a lower wettability. A contrary result was found by Stehr *et al.* (2001) in the case of southern pine; they found that a smoother wood surface provides improved wetting and penetration properties for high-viscosity liquids such as adhesives, the reasons may be due to the different of the machined process of wood or the type of adhesives used in the experiment. However, the K -value of the sanded aged wood surface increased with increasing grit number, the sanding process decreased the wettability of the aged wood surface, and the aged wood sanded with a grit number of 180 had the highest K -value. Compared to the aged samples, the fresh wood surface was more wettable, and the change compared to the control wood was not evidence. As shown in Fig. 8, the Modified model can provide an excellent fit to the experimental data (R^2 = 0.99); therefore, this model can accurately describe the adhesive wetting process on a wood surface.

Modified Model

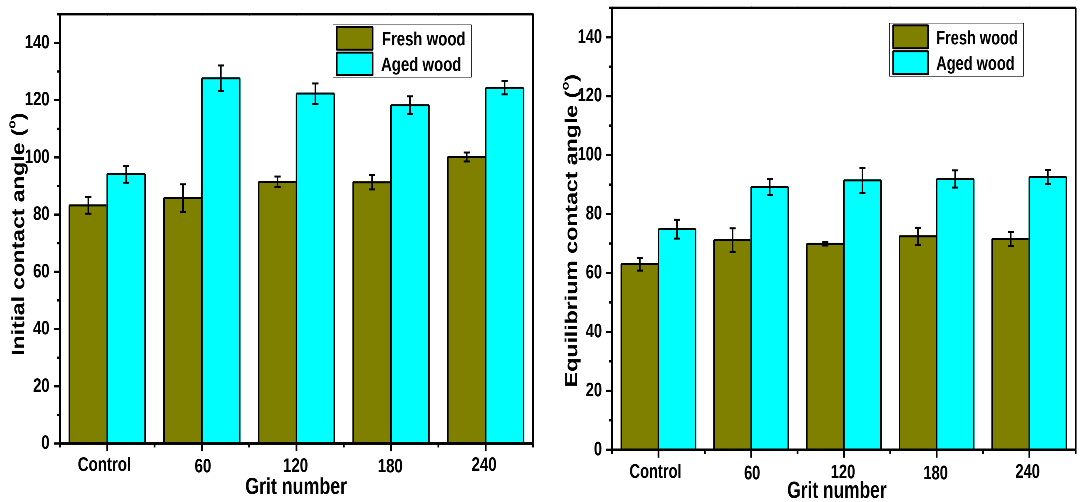

Fig. 7. Contact angle for fresh and aged wood surfaces according to the Modified model

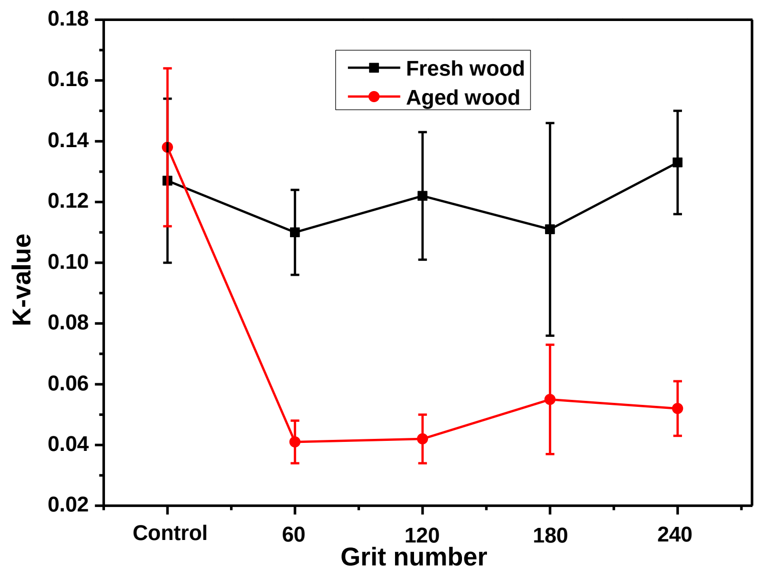

Fig. 8. K-value of wood samples according to the Modified model

All the contact angles (*i.e.*, initial and equilibrium contact angles) of the fresh and aged wood determined by the model increased with increasing grit number, and the contact angle of aged wood was higher than that of fresh wood. These results are similar to the trend obtained by the (Nussbaum 1999).

For the K -value of the fresh wood samples, no clear trend was found when the grit number was increased. However, the K -value of the sanded aged wood was much lower than that of the control aged wood, the K -value slightly increased with increase in grit number, and the wood sample sanded with a grit number of 180 had the highest K -value; this is the same data obtained by the S-D model (Shi and Gardner 2001). There was an inverse trend with the K -value (*i.e.*, higher K-value with lower contact angle) in most cases.

Santoni Model

Santoni and Pizzo (2011) used their model to investigate the effects of the machining process and air exposure on the wettability with water of six different Mediterranean wood species. The parameters and standard deviations obtained with the Santoni model (Santoni and Pizzo 2011) for sanded and aged wood samples are shown in Table 1. θ_1 can be considered the spreading initial contact angle caused by the surface morphology and capillary action, which increased from 88.27° to 106.71° with increasing grit number for the fresh wood samples. For the aged wood, the contact angle, θ_1, ranged from 100.16° to 133.54°, but the change with different grit numbers was insignificant. θ_2 can be defined as the contact angle when the penetration starts, the changes in which may be caused by the internal cell structure. The contact angle of the sanded wood (fresh wood and aged wood) was lower than that of the unsanded wood, which may be due to the cell lumen being blocked by the sand wood ash. An accurate explanation for this result requires further investigation. However, there is no doubt that both contact angles, θ_1 and θ_2, of the aged wood were higher than those of the fresh wood.

Table 1. Values of Parameters for Contact Angle Measurement on Sanded and Aged Wood Surface

Species	Surface	θ_1 (°)	θ_2 (°)	K_1	K_2
Control	Fresh wood	88.27(3.32)	64.08(2.46)	5.04(0.64)	0.030(0.004)
	Aged wood	100.16(3.14)	77.02(3.53)	5.82(0.59)	0.028(0.004)
60	Fresh wood	93.74(2.80)	74.28(3.25)	3.80(0.23)	0.038(0.007)
	Aged wood	133.54(3.37)	104.11(2.87)	3.53(1.14)	0.147(0.038)
120	Fresh wood	97.94(4.59)	72.82(1.09)	5.78(0.87)	0.039(0.006)
	Aged wood	131.17(1.42)	103.54(0.99)	4.11(0.81)	0.120(0.030)
180	Fresh wood	97.06(3.62)	75.44(2.25)	4.91(1.03)	0.036(0.006)
	Aged wood	130.34(5.07)	98.63(4.72)	5.71(1.63)	0.074(0.014)
240	Fresh wood	106.71(2.54)	74.62(2.33)	7.11(0.72)	0.042(0.005)
	Aged wood	132.09(4.05)	102.10(0.74)	4.41(1.10)	0.076(0.042)

Standard deviation denoted in brackets

In Table 1, the variable K_1 is defined as the spreading rate. The change in K_1 was irregular because of the complex morphology of the wood surface. K_2 is considered to be the rate of adhesive penetration into the wood. The K_2 value of the aged wood was higher than that of the fresh wood. From this observation, it can be concluded that the rate of spreading was much faster than that of penetrating.

The calculation of the Nussbaum model (Nussbaum 1999) is simple, but it cannot describe the dynamic process of spreading and penetration. The S-D model (Shi and Gardner 2001) is widely used to quantitatively evaluate the wettability; however, the initial and equilibrium contact angles are difficult to determine for wood because it is a porous material, and the calculation is complex. The Modified model can evaluate the dynamic wettability without determining the initial or equilibrium contact angle, and the theoretical initial and equilibrium contact angle can be also acquired through the model. The correlation coefficients' R-squared values for all four models were over 90%, but the accuracy of the models needs to be further verified. The Santoni model (Santoni and Pizzo 2011) can accurately describe the wettability, including the spreading and penetration process; the problem is that the time of each stage is hard to determine, and the data may be influenced by human factors.

The wettability is an important factor in determining adhesion strength. Liquid penetration that is too deep will lead to a reduction in adhesion. The relationship between wood surface wettability and bonding strength should also be further investigated.

CONCLUSIONS

1. The wettability of PF adhesive on freshly sanded wood and aged wood both decreased compared to the control wood, and the change in wettability with increased grit number was not evident.
2. Aging reduced the wettability of the wood surface.
3. All coefficients of determination (R^2) values were over 90%, and the value of the Modified model was 99%. The models can be used to accurately describe the adhesive wetting process.
4. The wettability of adhesive and water on the fresh wood surface had evident differences. The wettability of the adhesive increased with increasing grit number, while the wettability of water decreased. The same trend was found for the water and adhesive on the aged surface.
5. The calculation of the Nussbaum model is simple, but it cannot describe the dynamic process of spreading and penetration. The S-D model is widely used to quantitatively evaluate the wettability, but the calculation is complex. The Modified model is sample to evaluate the wettability but needs further improvement. The Santoni model can accurately describe the wettability of spreading and penetration process, but the data may be influenced by human factors.

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