EFFECTS OF DIELECTRIC BARRIER DISCHARGE PLASMA MODIFICATION ON SURFACE PROPERTIES OF TROPICAL HARDWOODS AT LOW PRESSURE

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ACDA MN, DEVERA EE, CABANGON RJ, PABELINA KG & RAMOS HJ. 2012. Effects of dielectric barrier discharge plasma modification on surface properties of tropical hardwoods at low pressure. The study investigated the use of dielectric barrier discharge (DBD) for surface modification of *Shorea contorta* (white lauan), *Gmelina arborea* (yemane) and *Acacia mangium*. Wood specimens were exposed to oxygen plasma at various intensities ranging from 5.8 to 46.5 kW-min m^{-2}. Surface free energy was calculated based on contact angle measurements to determine thermodynamic changes on plasma modified wood. Surface characteristics were evaluated using attenuated total reflectance Fourier transform infrared (ATR–FTIR) spectroscopy and atomic force microscopy (AFM). Results of the study showed that plasma modification resulted in significant increase in surface free energy of the three wood species investigated. ATR–FTIR indicated that plasma-treated wood had higher surface polarity compared with untreated controls. AFM 3D image showed that oxygen plasma was capable of cleaning and etching wood surface resulting in the degradation of primary and secondary cell walls.

Keywords: Dieletric barrier discharge, surface modification, surface free energy, atomic force microscopy, *Shorea*, *Gmelina*, *Acacia*

ACDA MN, DEVERA EE, CABANGON RJ, PABELINA KG & RAMOS HJ. 2012. Kesan ubah suai plasma peluahan sawar dielektrik terhadap ciri permukaan kayu keras tropika pada tekanan rendah. Eksperimen ini mengkaji penggunaan peluahan sawar dielektrik (DBD) untuk mengubah suai permukaan *Shorea contorta*, *Gmelina arborea* dan *Acacia mangium*. Spesimen kayu didedahkan kepada plasma oksigen pada keamatan berbeza daripada 5.8 kW-min m^{-2} hingga 46.5 kW-min m^{-2}. Tenaga permukaan bebas dikira berdasarkan ukuran sudut sentuh untuk menentukan perubahan termodinamik kayu yang diubah suai oleh plasma. Ciri permukaan dinilai menggunakan spektroskopi pantulan keseluruhan dikecilkan jelmaan Fourier inframerah (ATR–FTIR) dan mikroskopi daya atom (AFM). Keputusan menunjukkan pengubahsuaian plasma menghasilkan peningkatan signifikan dalam tenaga permukaan bebas bagi ketiga-tiga spesies kayu yang dikaji. ATR–FTIR menunjukkan bahawa kayu yang dirawat dengan plasma mempunyai kekutuban permukaan yang tinggi berbanding dengan kawalan yang tidak dirawat. Imej 3D menunjukkan plasma oksigen berupaya membersihkan serta memunar permukaan kayu lalu menyebabkan penguraian dinding sel primer dan sekunder.

INTRODUCTION

The use of plasma to modify surface properties of wood in order to improve adhesion of glues and coatings has recently attracted research interest in many countries (Kogelschatz 2003, Custodio et al. 2008). Plasma can be described as partially ionised gas consisting of a complex mixture of ions, electrons, atoms, radicals, etc. (Boenig 1982). Plasma particles are usually generated by stripping off electrons from low pressure or atmospheric gases to induce ionisation and fragmentation using thermal or electrical energy. Plasma, characterised by particles with very low degrees of ionisation and little penetrating energy (called cold plasma), is often used for surface modification or activation (Eliezer & Eliezer 2001). Excited particles in cold plasma have energy levels (0.5–3 eV) sufficient to break chemical bonds on the surface of organic

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and inorganic substrates (Lide 1993, Denes et al. 1999). These broken chemical bonds are thermodynamically unstable and combine with gas fragment to molecularly re-engineer the surface of the material (Setoyama 1996). The modification (cleaning, etching or cross linking), however, is limited to the surface, typically to a depth of a few molecular layers.

Cold plasma is a powerful tool that can be used to improve the wetting and adhesion properties of wood (Evans et al. 2007, Blanchard et al. 2009, Busnel et al. 2009, Acda et al. 2012) and wood composites (De Meijer et al. 2000, Gindl et al. 2004, Wolkenhauer et al. 2008). It is believed that plasma modification results in increased surface polarity brought about by the formation of hydroxyl, carboxyl, aldehyde and other polar functional groups (Odrasková et al. 2008). The increased surface polarity improves wettability and penetration behaviour (hydrophilicity) of wood to liquid (Rehn et al. 2003). Wettability assists in establishing extensive and molecular-scale contact with the wood surface and critical to the development of strong adhesion at the adhesive–wood interface (Frihart 2005). The present paper reports on the effects of plasma modification on surface properties of three species of wood, viz. *Shorea contorta*, *Gmelina arborea* and *Acacia mangium* commonly used in the Philippines and South-East Asia for furniture and wood-based panel manufacture.

MATERIALS AND METHODS

Wood specimen

Kiln dried *S. contorta* (white lauan, density 430 kg m^{-3}) lumber obtained from local furniture manufacturer in Cebu province, Philippines, and *G. arborea* (yemane, density 410 kg m^{-3}) and *A. mangium* (density 470 kg m^{-3}) from the Forest Products Research Development Institute, Philippines were used in this study. These species are grown intensively in industrial plantations and commonly used as raw materials for furniture and various wood products. Wood specimens (5 mm × 25 mm × 25 mm) were cut from the heartwood of straight grained and defect-free boards, and then conditioned for several weeks at 24 °C and 65% relative humidity to bring the moisture content to 8%. All specimens were lightly sanded (150 grit) and dusted off prior to plasma treatment.

Plasma surface modification

Plasma surface modification of wood specimen was performed using a dielectric barrier discharge (DBD) device (Figure 1) developed at the Plasma Physics Laboratory, National Institute of Physics, University of the Philippines, Diliman, Philippines. The DBD system consisted of a stainless steel cylinder with the anode and cathode located in the middle of the chamber. The chamber was initially evacuated to a pressure of 10 Pa for 20–30 min using a rotary pump. Then the operating gas was introduced into the chamber through mass flow controllers and an electronic flow meter. Temperature was monitored through a type K thermocouple connected to a miniature autotune microprocessor controller. High purity oxygen gas was admitted through the anode by a small hollow tube connected to a perforated stainless steel shower cap. However, minor contamination of the plasma gas was possible from wood extractives that migrated to the surface with moisture during the evacuation process.

Wood specimens were exposed to oxygen plasma at varying power output (0.1, 0.2, 0.4 and 0.8 kW) using 3-mm distance between electrodes to determine its effects on contact angle and surface free energy. Power level that resulted in significant improvement in surface free energy was used to vary treatment duration (15, 30, 60 min). All experiments were performed at a constant gas flow rate of 10 cm^3 min^{-1}. Five replicates were used for each treatment. Plasma intensity (E) was calculated as

$$E = \frac{P \times T}{A}$$

where P = electrical output of the power supply (kW), T = duration of treatment (min) and A = cross-sectional area (m^2) of specimen exposed to the plasma gas. All specimens were removed from the DBD device after treatment and sealed in polyethylene bags for surface characterisations using atomic force microscope (AFM) and attenuated total reflectance Fourier transform infrared (ATR–FTIR) spectroscopy. The DBD chamber was thoroughly cleaned with methanol after each treatment to remove deposits and contaminants from previous treatment. Untreated wood specimens were used as control.

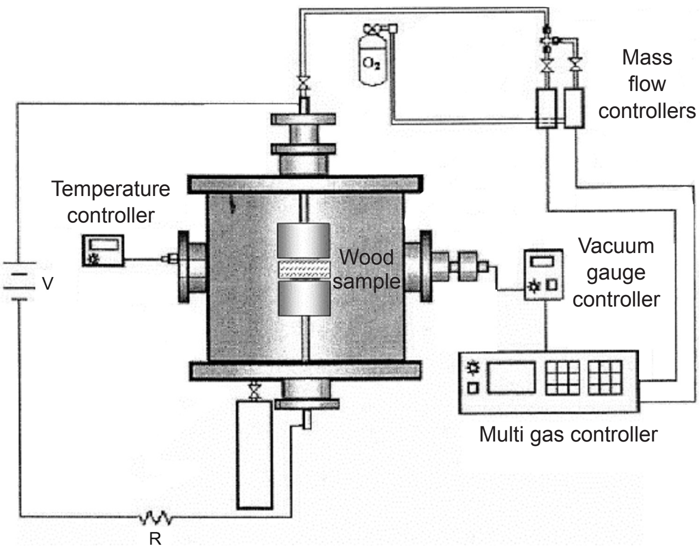

Figure 1 Schematic diagram of the dielectric barrier discharge (DBD) device for plasma surface modification of wood

Contact angle and surface free energy

Contact angle and surface free energy were used to investigate thermodynamic changes in surface properties of all three wood species after exposure to oxygen plasma. Contact angles were measured using the static sessile drop method. Five droplets (5 µL) of the test liquid were randomly deposited on the tangential surface of each specimen using a microsyringe. A digital microscope was used to capture high resolution images of droplet profile on wood surface after it had established equilibrium. Contact angles were measured directly by drawing a tangent line from the droplet profile to the wood surface using image analysis software. All contact angle measurements were made immediately after droplet deposition at room temperature (25 °C) and within 10 min of plasma treatment.

Surface free energies of plasma-treated wood specimens were calculated based on the Owens, Wendt, Rabel and Kaeble method (Kaeble 1970). The equation for surface tension of solid (Owens & Wendt 1969)

$$\gamma_{SL} = \gamma_S + \gamma_L - 2\sqrt{\gamma_S^D \gamma_L^D} - 2\sqrt{\gamma_S^P \gamma_L^P} \quad (1)$$

was combined with Young's equation

$$\gamma_S = \gamma_{SL} + \gamma_L \cos\theta \quad (2)$$

and transposed to the general equation of a straight line (Berg 1993, Pocius 2002)

$$\frac{(1+\cos\theta)\gamma_L}{2\sqrt{\gamma_L^D}} = \sqrt{\gamma_S^P}\sqrt{\frac{\gamma_L^P}{\gamma_L^D}} + \sqrt{\gamma_S^D} \quad (3)$$

where γ_S = total surface energy, γ_{SL} = interfacial energy between wood and liquid, γ_L = surface tension of the liquid and θ = contact angle. The model (3) divided the surface energy into non-polar (γ^D) and polar (γ^P) components. The non-polar component is associated with London dispersive forces of interaction and the polar component is associated with acid–base interactions. From the linear regression equation, γ_S^P was obtained from the square of the slope of the curve and γ_S^D, from the square of the ordinate intercept. Three test liquids of known disperse and polar surface tension components were used to determine the total surface energy of wood specimen, namely, deionised water (γ^P = 51 mN m^{-1}, γ^D = 21.8 mN m^{-1}), glycerol (γ^P = 30 mN m^{-1}, γ^D =34 mN m^{-1}) and ethylene glycol (γ^P = 19 mN m^{-1}, γ^D = 29 mN m^{-1}). Data for surface free energy and plasma intensity were fitted in a completely randomised design and evaluated using analysis of variance (ANOVA) with Statgraphics Plus for Windows 4 software (1999). Treatment means were separated by Tukey's honest significance difference (HSD) test (α = 0.05).

Surface characterisation

Specimens from plasma treatment that showed significant increase in surface free energy were characterised for surface topography using an atomic force microscope running on a Nova 1.0.26C software. All measurements were performed at atmospheric pressure and room temperature in non-contact tapping mode (scan size 1.0–2.5 μm^2, scan rate 0.3–1 Hz with a silicon cantilever tip, 256 × 256 pixels resolution). Topographic 3D-images of plasma-treated specimens were obtained and compared with untreated controls. ATR–FTIR spectroscopy was carried out using a spectrometer equipped with Miracle ATR to investigate any chemical changes on the wood surface due to plasma treatment. Scans were made using a diamond crystal (45° incidence angle, single bounce) from 600–4000 cm^{-1} wavelength with 10 scans for each sample at a resolution of 4 cm^{-1}.

RESULTS AND DISCUSSION

Effects of plasma treatment on surface free energy

Plasma surface modification of *S. contorta, G. arborea* and *A. mangium* at conditions used in this study resulted in no apparent surface defects or change in bulk properties. Dielectric barrier discharge treatments resulted in plasma intensities of 5.8–46.5 kW-min m^{-2} with chamber temperature ranging from 18–71 °C and electric potential of 700–1200 V. The total surface free energy for all three species increased significantly ($p < 0.001$) by five to six times compared with untreated controls (Figure 2). Similar trends were reported for softwood and hardwood treated with corona plasma (Podgorski et al. 2000). Untreated wood specimens for all three species investigated showed predominantly non-polar characteristics (Figure 2). However, both polar and disperse components of each species showed significant changes ($p < 0.001$) after plasma treatment. Polar component for *S. contorta* increased by more than 50 times while disperse component decreased by about 8 to 11 times with increasing plasma intensity (Figure 2). Both polar and disperse components of *G. arborea* and *A. mangium* increased significantly by about 3 to 15 times compared with that of untreated materials (Figure 2). The change in polar and dispersive components after treatment may be related to the interaction of plasma particles and chemical constituents on the surface of the wood (Blanchard et al 2009). Major constituents of wood are combined into a number of organic polymers (cellulose, hemicellulose and lignin) and a minor proportion of extraneous materials called extractives (Sjostrom 1993). The proportion of each component vary among species and between softwoods and hardwoods. Proximate chemical analyses of *S. contorta, G. arborea* and *A. mangium* are shown in Table 1. Earlier studies reported the formation of polar functional groups such as hydroxyls (O–H), carbonyl (C–O) or aldehydes upon exposure of wood to reactive plasma gases (Blanchard et al. 2009). The interaction results in increased surface free energy as indicated by improved wettability and penetration behaviour of wood to liquid (Klarhofer et al. 2005, Odrasková et al. 2008). Degradation and removal of contaminants (extractives) on wood surface by oxidative reactions resulted in improved wettability (Vander Wielen et al. 2005, Wolf & Sparavigna 2010, Jamali & Evans 2011). Wettability assists in establishing an extensive and molecular scale contact with wood surface and is critical for the development of strong adhesion at adhesive–wood interface (Frihart 2005). The increased surface free energies of plasma-treated *S. contorta, G. arborea* and *A. mangium* could be positively correlated in achieving good wettability of polar adhesives and coating systems on the three species investigated in this study.

Effects of plasma treatment on surface characteristics

FTIR spectra of treated and untreated specimen of *S. contorta, G. arborea* and *A. mangium* showed the same basic structure (Figure 3). There was strong broad O–H stretching at 3300–3400 cm^{-1} derived from adsorbed water, C–H stretching vibration around 2900 cm^{-1} and several distinct peaks in the fingeprint region between 600 and 1750 cm^{-1}. These well defined peaks have been attributed to functional groups from both carbohydrates (cellulose and hemicellulose) and lignin (Pandey 1999). No other absorbance peaks besides those already reported in the literature for wood were detected. *Shorea contorta* and *G. arborea* wood specimens exposed to oxygen plasma showed slightly higher absorbance with increasing plasma intensity compared with untreated controls (Figure 3). This could be explained by the formation of hydroxyl and carbonyl groups on the wood surface during

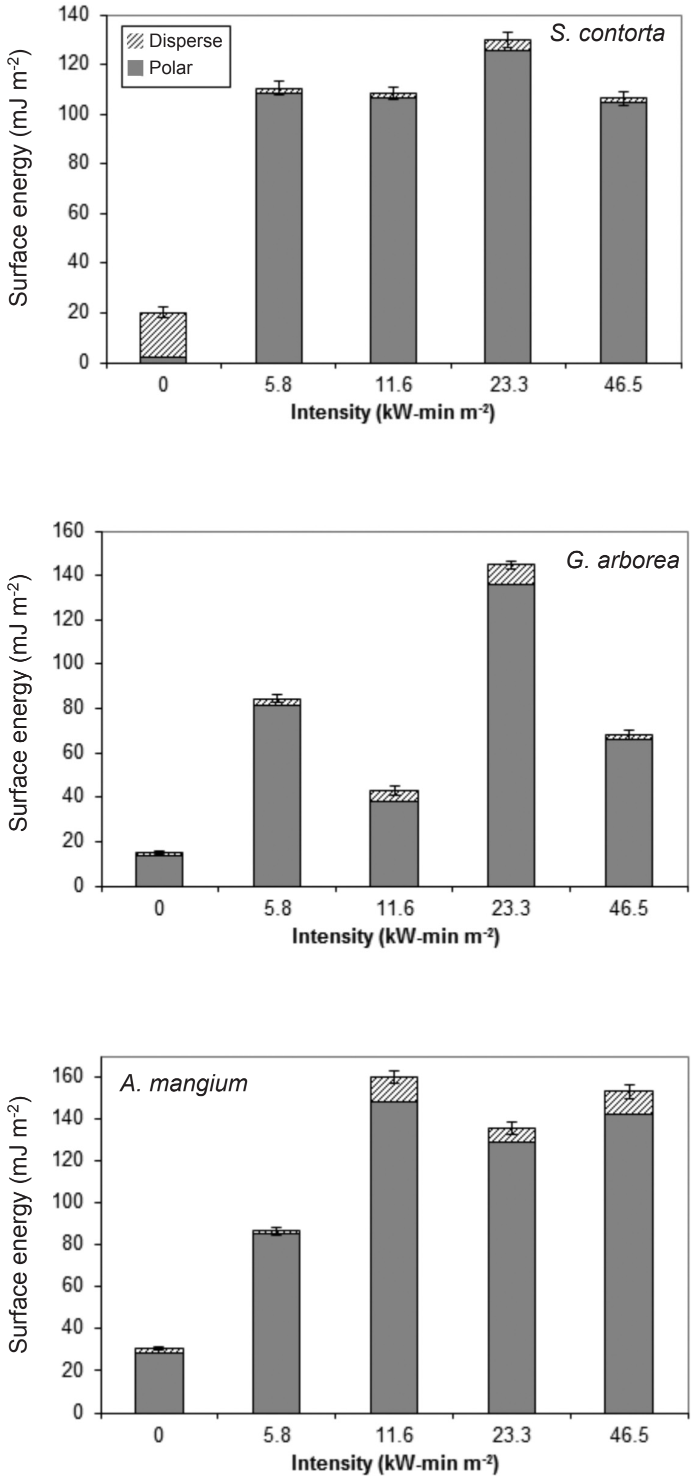

Figure 2 Surface free energy of plasma-treated and untreated specimens of *Shorea contorta, Gmelina arborea* and *Acacia mangium*

Table 1 Proximate chemical analyses of *Shorea contorta*, *Gmelina arborea* and *Acacia mangium*

Species	Hollocellulose (%)	Lignin (%)	Extractives (%)
S. contorta	67.9	27.2	3.8
G. arborea	72.3	22.3	4.6
A. mangium	72.4	21.4	5.6

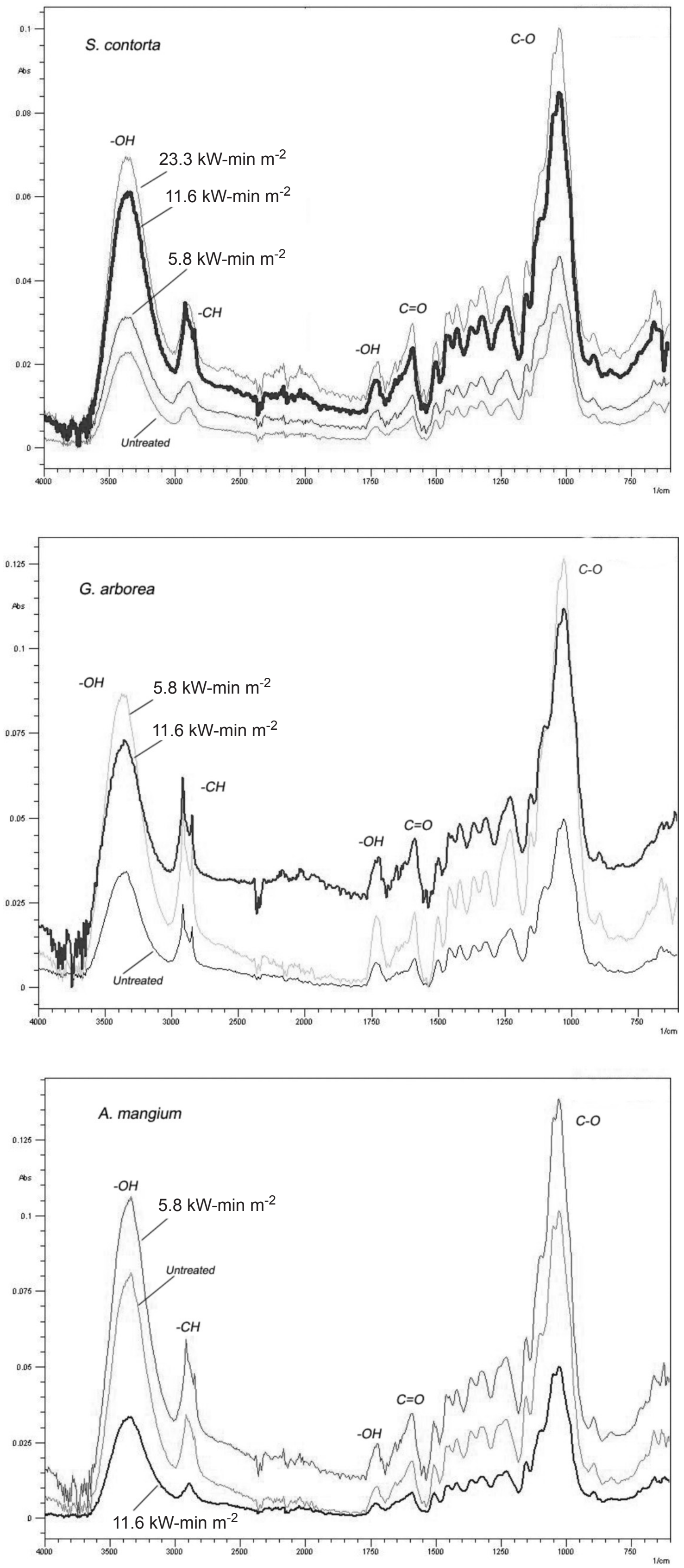

Figure 3 FTIR spectra of plasma-treated and untreated specimens of *S. contorta, G. arborea* and *A. mangium*

plasma treatment. Earlier studies with plasma modification of wood showed increased surface polarity due to the formation of polar functional groups from possible oxidation reaction of plasma particles with hemicelluloses and extractives on wood surface (e.g. Odrasková et al. 2008, Blanchard et al. 2009).

FTIR spectra of treated and control *A. mangium* wood specimens showed higher absorbance at 3000–3400 cm^{-1} wavelength and 5.8 kW-min m^{-2} plasma intensity derived from strong O–H stretching (Figure 3). Higher absorbances at about 1020 cm^{-1} derived from C–O stretching in cellulose and hemicelluloses were also observed. However, lower absorbances were observed at higher plasma intensities (Figure 3). Results seemed to show two opposite effects occurring at the lower and higher range of plasma intensity used in this study. Slight increase in surface polarity occurred at 5.8 kW-min m^{-2} while a decrease was noted at 11.6 kW-min m^{-2}. The increased polarity has already been discussed above. However, the opposite effect seemed problematic. Surface deposition of non-polar contaminants and reorganisation of broken down cell wall components may be responsible for this observation.

Topographical 3D-images of the surface of all three wood species investigated were examined using AFM. The heterogeneous structure of wood due to the presence of excised fibres and vessel elements, cross-sections of ray cells and remnants of cell wall material sticking out of the surface made it difficult to scan without damaging the cantilever tip of the AFM. Three dimensional images obtained from untreated specimens of *S. contorta*, *G. arborea* and *A. mangium* showed the presence of nodule-like deposits dispersed throughout the surface of the wood (Figures 4a, 5a and 6a respectively). This is due to the presence of amorphous materials such as extractives and hemicelluloses on the surface of wood (Vander Wielen et al. 2005).

Three dimensional AFM phase images of *S. contorta* specimens exposed to oxygen plasma at 5.8 kW-min m^{-2} showed a relatively clean surface with no surface contaminants (Figure 4b). Reactive plasma gases have been known to remove surface contaminants through oxidation reaction between oxygen ion and hydrocarbons to produce carbon monoxide and carbon dioxide (Deires et al. 2005). Surface etching is also believed to occur simultaneously with oxidation of organic materials (Goossens et al. 2001). Etching is caused by physical collision of electrons, free radicals and other excited particles against a solid surface (Flamm & Herb 1989). The collision breaks down amorphous organic materials and contaminants on the surface into small fragments that volatilise off with the plasma stream. In this study, at 11.6 kW-min m^{-2}, primary cell wall and S_1 layer of the secondary wall were degraded causing the exposure of the S_2 layer (Figure 4c). The S_2 layer was evident due to its distinct fibrillar pattern and acute angle of orientation. Similar observations were reported by Snell et al. (2001) for AFM study using kraft fibres. The exposure of S_2 layer could explain the generally high polar component of the surface free energy and higher FTIR absorbance of plasma-treated *S. contorta*. The polar characteristics could be traced to the availability of more hydroxyl groups from the exposed cellulosic fibrils compared with untreated controls.

AFM images of *G. arborea* and *A. mangium* treated at 5.8 kW-min m^{-2} showed the presence of apparently larger nodules on the surface of both wood species compared with untreated controls (Figures 5b and 6b). At higher plasma intensity, the nodules seemed to change in structure and morphology suggesting surface reorganisation. These changes could be the result of cell wall (hemicellulose) degradation and extractives polymerisation reaction through free radical activation. Proximate chemical analyses of *G. arborea* and *A. mangium* showed that both species have relatively high extractive content (Fidel & Tamayo 1999). Hardwood extractives consist mostly of fats, waxes and sterols (Sjostrom 1993). The cause and mechanism of surface reorganisation or polymerisation observed in this study are still unclear and more tests are needed to confirm this hypothesis. However, surface deposition of polymers on wood surface using plasma processes to enhance hydrophilicity or hygroscopicity is common and widely reported in the literature (e.g. Wang et al. 2002, Zanini et al. 2008). Surface reorganisation of non-polar cell wall materials on the surface of *G. arborea* and *A. mangium* could explain the high disperse components of their surface free energy, the reduced FTIR absorbance and decreased polarity of these wood species at higher plasma intensity.

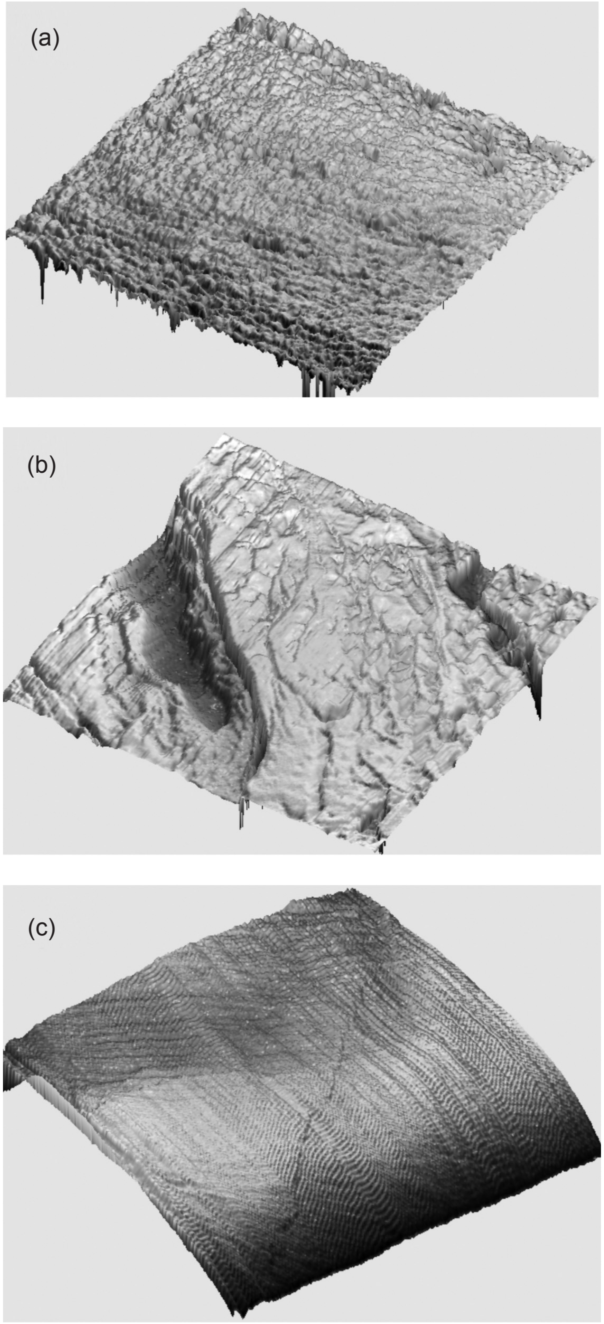

Figure 4 AFM 3D-images (2.5 × 2.5 µm) of *S. contorta* (a) untreated specimen with nodule-like deposits dispersed on the surface of wood, (b) plasma-treated at 5.8 kW-min m^{-2} with clean surface and no deposits, natural crevices on surface evident and (c) plasma-treated at 11.6 kW-min m^{-2} showing fibrillar orientation

CONCLUSIONS

In general, the study investigated the use of dielectric barrier discharge to modify surface properties of *S. contorta, G. arborea* and *A. mangium.* Wood specimens exposed to oxygen plasma resulted in several fold increase in surface free energy. No apparent surface defects or changes in bulk properties were detected. The increased surface free energy could be positively correlated with the development of good joint strength during gluing with polar adhesives and coating systems. Surface characterisation using ATR–FTIR showed typical absorbance attributed to functional groups from both carbohydrates (cellulose and hemicellulose) and lignin. However, higher absorbance was observed at plasma intensities of 5.8 to

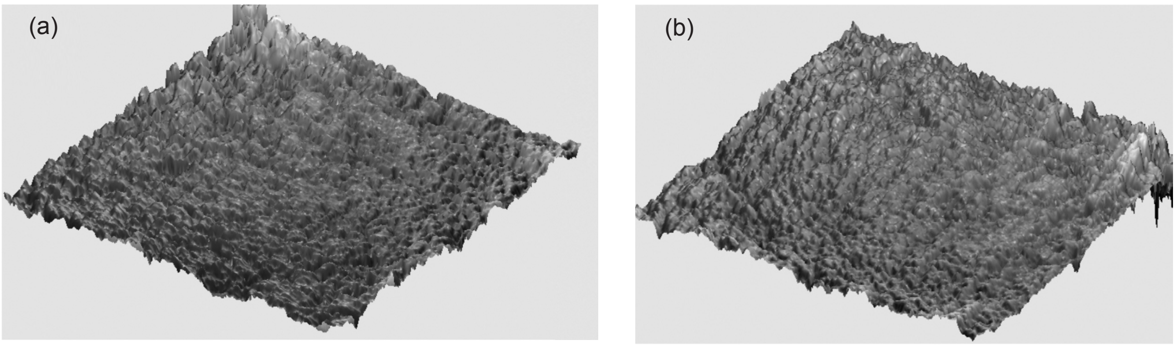

Figure 5 AFM 3D images (2.5 × 2.5 μm) of *G. arborea* (a) untreated specimen with surface nodules and (b) plasma-treated at 5.8 kW-min m^{-2} with apparently larger surface nodules

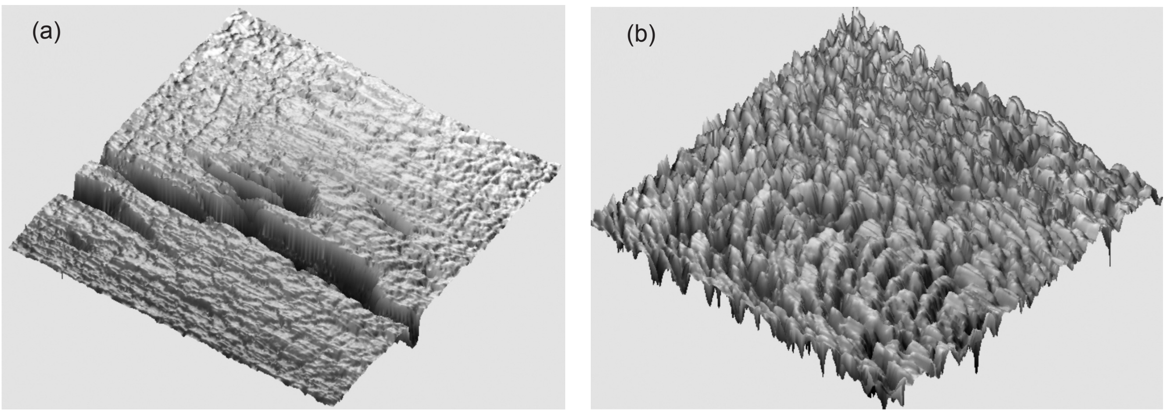

Figure 6 AFM 3D images (2.5 × 2.5 μm) of *A. mangium* showing (a) untreated specimen with surface nodules and (b) plasma-treated at 5.8 kW-min m^{-2} with apparently larger surface nodules

23.3 kW-min m^{-2} at wavelengths of 3200–3400 cm^{-1} and 1020 cm^{-1}. These peaks were attributed to hydroxyl (OH) and carbonyl (CO) functional groups respectively. Examination of AFM 3D topographic images revealed degradation of primary and secondary cell walls. Apparently, surface cleaning and etching occurred during plasma treatment that exposed fibrils from the S_2 layer of the secondary wall. It is possible that the availability of hydroxyl groups in the cellulose chains of the exposed fibrils could be responsible for the higher surface free energy and higher FTIR absorbance peak. Evidently, exposure to plasma conditions used in this study resulted in changes in surface characteristics that could affect reactivity of wood species investigated to polar adhesives and coatings.

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