Combined Surface Treatment of Polyethylene Wood-plastic Composites to Achieve Rapid Bonding with Desired Adhesion Properties

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To achieve rapid bonding with desired adhesion properties, a combined treatment of sanding then coating with polyisocyanate, followed by plasma discharge was implemented on the surfaces of polyethylene wood-plastic composites (WPCs). The surface properties of polyethylene WPCs were studied by evaluating the contact angle and bonding strength, as well as analyzing it *via* Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and X-ray photoelectron spectroscopy (XPS). The results indicated that the shear strength and durability of the bonding joints of polyethylene WPCs increased considerably due to the synergetic effect from the surface treatment. Thus, a rapid bonding with desired adhesion properties of polyethylene WPCs was achieved. The roughness and the oxygen content on the composites' surface increased after the combined treatment. Polar functional groups, such as –OH, –C=O, and –O–C=O, formed on the surface. At the same time, the presence of –NCO and –NH functionalities, generated during the combined treatment, showed that chemical bondings between polyisocyanate and the wood fibers of the composites occurred. The changes on the surface properties, such as roughness, wettability, as well as formation of chemical groups, substantially affected the adhesion properties of the bonding joint for polyethylene WPCs.

Keywords: Polyethylene wood-plastic composites; Rapid bonding; Combined surface treatment; Polyisocyanate coating; Plasma discharge; Surface properties

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INTRODUCTION

Polyethylene wood-plastic composites (WPCs), a typical representative of WPCs, have been widely used in several fields due to their easy processing and recycling capabilities (Ashori 2008; Ozdemir and Mengeloglu 2008; Perisić *et al*. 2009). Unfortunately, the polyethylene ingredient present within the polyethylene WPCs results in poor polarity, lower surface energy, and difficult adhesion of the composites (Tahara *et al*. 2003; Gramlich 2006; Liu *et al*. 2010). Hence, a surface treatment is necessary for the polyethylene WPCs to obtain optimal bonding properties.

Many surface treatment methods, such as flame treatment, water treatment, chromic acid treatment, oxyfluorination treatment, oxygen plasma treatment, and coupling agent coating treatment, are found in the present literature (Gupta *et al*. 2007; Gupta and Laborie 2007; Oporto *et al*. 2007; Laborie and Gupta 2008; Wolkenhauer *et al*. 2008; Moghadamzadeh *et al*. 2011; Sogutlu *et al*. 2016; Sogutlu 2017). Besides the individual surface treatments mentioned, a combined treatment was also employed to treat the WPCs materials. For example, Gramlich *et al*. (2006) combined flame and water surface treatments to treat the surface of the WPCs. Moghadamzadeh *et al*. (2011) employed a combination treatment of sanding followed by a corona discharge treatment to improve the bonding strengths of the WPCs. The experimental results showed that the combined treatment for the composites would produce better bonding performance, and the combination of the two treatment methods may display an unexpected synergetic action. Thus, it is clear that using combined processing methods to modify the surfaces of polyethylene WPCs materials is more effective than using a single processing method.

Among the above-mentioned treatments, it is noteworthy that the plasma treatment and coupling agent coating treatment were the preferable treatment methods. The plasma surface treatment has the advantage of achieving a relatively fast and effective treatment result. Unfortunately, the stability and ageing of the plasma treatment (Øiseth *et al*. 2002; Arpagaus *et al*. 2005), and the durability in a wet environment of the bonding joint for plasma-treated composites, have been found to be poor (Di *et al*. 2012; Wang and Di 2012; Di and Wang 2013). In contrast, the polyethylene WPCs coated with a coupling agent showed a high bonding strength and good durability. However, the processing of coating with a coupling agent requires a high temperature heating procedure, causing a longer processing time.

In daily applications, both the bonding strength and bonding speed of the bonding joint are important factors. Regarding polyethylene WPCs, the bonding speed should embrace two layers of meaning. One is the processing speed of the surface treatment, and the other is the curing speed of the adhesive used. As mentioned in a previous study on the bonding of polyethylene WPCs, the bonding properties for the bonding joint, which was adhered using an epoxy resin adhesive, are satisfied both in the aspect of bonding strength and bonding durability. However, as is well known, the curing speed of epoxy resin adhesive is relatively slow, such that an increase in the curing speed would lead to the degradation of the bonding performance. Accordingly, the rapid bonding of polyethylene WPCs could not be achieved by using epoxy resin as the adhesive. Among several alternative adhesives available for adhering the polyethylene WPCs, the moisture-curable polyurethane hot melt adhesive is desirable when the aspects of adhesive fixing speed and bonding properties are considered.

In the authors' previous study, a combined surface treatment of sanding then coating with a silane-coupling agent, followed by plasma discharge was proposed. The advantage for the combined treatment lies in its rapid processing speed and desired bonding properties. This study proposes combining the advantages of both the combined surface treatment as mentioned above with the moisture curable polyurethane hot melt adhesives. To study the synergetic effect of the combined treatment on the surfaces of polyethylene WPCs, and to test and analyze the bonding properties, a contact angle test, Fourier transform spectroscopy (FTIR), scanning electron microscopy (SEM), X-ray photoelectron spectroscopy (XPS), and the bonding property tests were used.

EXPERIMENTAL

Materials

The polyethylene WPCs were extruded by the Material Science and Engineering College of Northeast Forestry University of Harbin, China.

According to the manufacturer instructions, the WPC constituent materials by weight were 60% poplar flour (the particle diameters of 380 μm to 830 μm), 30% high-density polyethylene (HDPE), and 10% additives (maleic anhydride grafted polyethylene and release agent). A polyisocyanate coupling agent, which contains 90% chlorobenzene and 10% polyisocyanate, was used for the surface coating treatment (Heilongjiang Institute of Petrochemistry, Harbin, China). A moisture-curable polyurethane hot melt adhesive was used to adhere the composites (Heilongjiang Institute of Petrochemistry, Harbin, China).

Surface Treatment

The surface treatment of the polyethylene WPCs was performed following three sequential steps. First, the surface sanding treatment of polyethylene WPCs with 180-mesh (80-μm) sandpaper was conducted. Second, the surface coating treatment of the WPCs using 10 wt.% polyisocyanate in chlorobenzene was conducted immediately, after which the composites were left to stand for 5 min at room temperature. Finally, the plasma discharge treatment of the polyethylene WPCs was performed in a GSL-1100X-PJF-A low temperature plasma apparatus (KeJing Automation Equipment Co., Ltd., Shenyang, China).

The specimens were then positioned vertically under the plasma beam in the device. The ambient air at room temperature and atmospheric pressure was blown through the 30 mm discharge gap between the specimens and the plasma beam current, and the specimens were treated for 20 s, which produced the best bonding adhesion strength.

Rapid Bonding Procedure

The polyethylene WPCs were treated by a combined treatment of sanding then coating with polyisocyanate, followed by plasma discharge and bonded with moisture-curable polyurethane hot melt adhesive. The moisture-curable polyurethane hot melt adhesive needed heat to melt at the temperature of 130 °C before bonding, and the melted hot melt adhesive was used to bond the polyethylene WPCs specimens at the pressure of 20 psi (Pounds per square inch). The bonded specimens were put in an environment of a room temperature and a relative humidity of 60% for 24 hours and 15 days respectively, and then the shear strength of the samples were tested.

The shape and size diagram of bonding specimen was shown in Fig. 1, and a diagram of whole experimental process is shown in Fig. 2.

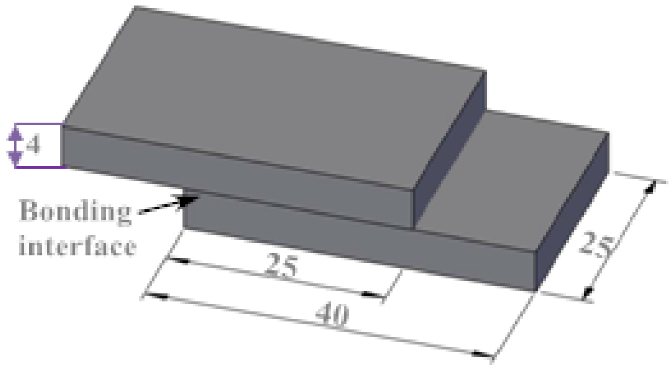


Fig. 1. The shape and size diagram of bonding specimen (Unit: mm)

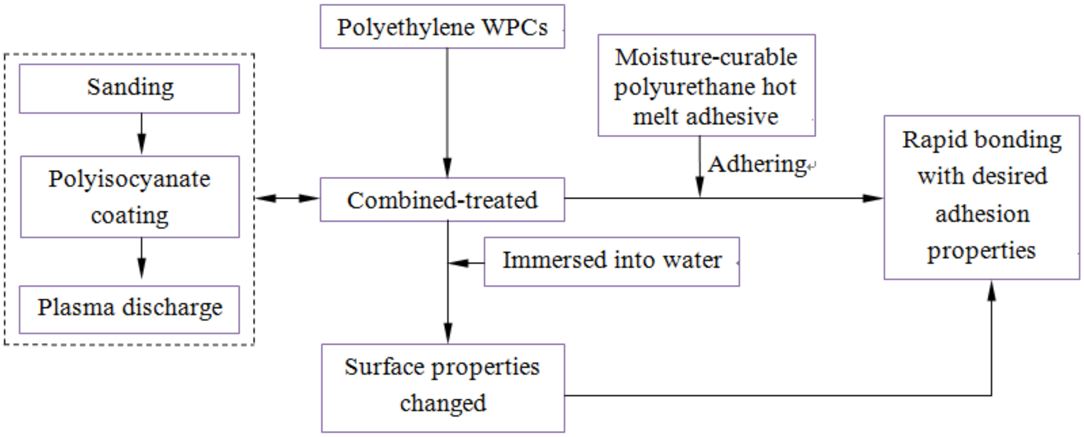


Fig. 2. The diagram of experimental process

Analysis Methods

A JC2000A goniometer, provided by ZhongChen Digital Technique Equipment Co. Ltd. (Shanghai, China), was used to implement the contact angle test of the WPCs specimens at room temperature by using distilled water as the test liquid. The contact angle was measured for at least five different locations by single drops (2 μL) of the test liquid per specimen. The FTIR spectral analysis was conducted on a Magna-IR 560 Fourier transform infrared spectrometer provided by Nicolet Co., Ltd. (Madison, USA), to measure the functional groups on the surfaces of the WPCs specimens. The SEM images of the WPCs surfaces were acquired using a Quanta 200 scanning electron microscope provided by FEI Co., Ltd. (Hillsboro, USA). The XPS spectra of the specimens were obtained using K-Alpha X-ray photoelectron spectra (XPS) equipment provided by Thermo Fisher Scientific Co., Ltd. (Waltham, USA) with a vacuum chamber pressure of 5×10^{-7} Pa. The energy scale using the energy of the C1s peak of adventitious aliphatic carbon (285.00 eV) was calibrated as the reference. During the curve fitting, the shape of the fitting curves was determined by the Gaussian and Lorentzian distributions, in which the Gaussian distribution ratio was not less than 80%. The shear bonding strength measurements of the specimens that were bonded with the moisture-curable polyurethane hot melt adhesive were conducted on a CMT 5504 universal mechanical testing machine provided by XinSanSi Co., Ltd. (Shenzhen, China) according to ASTM D905-08 (2013), and the rate of testing used was 5 mm/min.

RESULTS AND DISCUSSION

Shear Strength

Figure 3 shows the shear strengths of untreated, sanding-treated, sanding then plasma-treated, and combined surface-treated polyethylene WPCs bonded with the moisture-curable polyurethane hot melt adhesive. The bonding durability of the above-mentioned composites in a wet environment at 25 ^{o}C for various lengths of time was evident from Fig. 3. As shown, the shear strength of the untreated sample was only 0.47 MPa, and the value increased to 3.71 MPa after the sanding treatment, which indicated that the sanding treatment was beneficial to improve the bonding properties of the WPCs. This result was attributable to the removal of the polyethylene layer on WPCs surface, which increased the polarity and roughness of the composites surface and caused the mechanical inter-locking effect simultaneously. The shear strength of sanding-treated sample increased to 6.55 MPa after plasma treatment due to plasma discharge increase the polarity and the surface energy of polyethylene ingredient on WPCs surface. After the combined surface treatment of sanding, coating with polyisocyanate, followed by plasma treatment, the shear strength of the sample reached the remarkable level of 8.26 MPa, which showed that the combined treatment improved the shear strength of the composites considerably. Figure 3 also revealed that whether the surface was treated or not, the bonding strength of the composites bonding joint decreased after the immersion in water, especially for the untreated specimens, whose water resistance was rather poor. The shear strength was zero after immersion for 100 h for the untreated specimens, and the shear strength of the sanding-treated sample was only 1.04 MPa after 200 h of immersion, while the bonding strength of the combined treated sample still achieved 3.86 MPa. These results indicated that the combined treatment on the surface of the polyethylene WPCs improved the adhesion properties of the composites effectively.

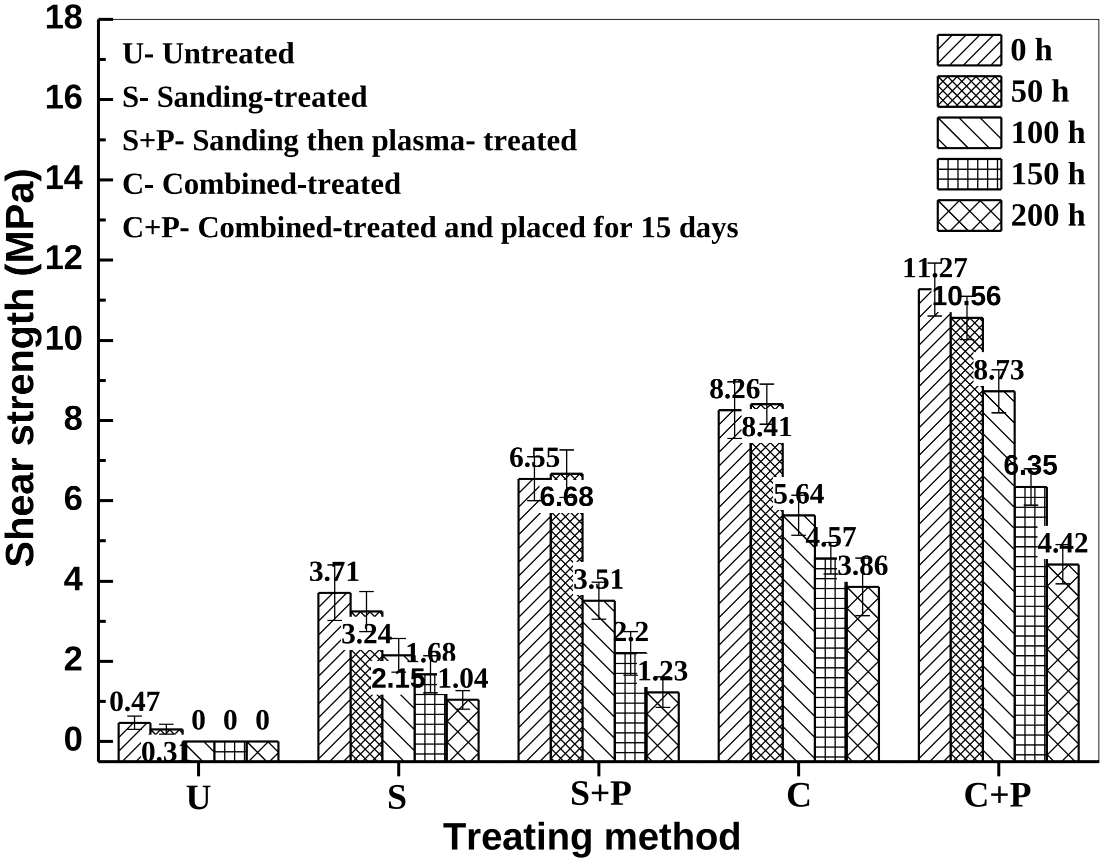


Fig. 3. The shear strengths and bonding durabilities of the bonding joints of polyethylene WPCs under water

Figure 3 also illustrates that for the combined-treated specimens, at the beginning of the immersion time, the shear strength of bonding specimens did not decline because of the soak, but rather indicated a slight increase. As the immersion time was further extended, the shear strength of the specimens started to decrease. This was ascribed to the moisture curing of the polyurethane hot melt adhesive, resulting in an increased bonding strength. The damage that occurred to the bonding-interface from water did not counteract the increase of bonding strength caused by adhesives post curing during early immersion. With the extension of the immersion time, the bonding-interface damage caused by water became stronger, while the adhesive reached moisture absorption saturation gradually, and resulted in a decrease in the bonding strength. As shown in Fig. 3. The shear strength improved extensively when the samples with combined treatment were bonded with moisture-curable polyurethane hot melt adhesive and then were put in an environment of a room temperature and a relative humidity of 60% for 15 days. This result was attributed to the improvement of adhesive post curing effect. When the bonding joint cured by moisture was immersed in water, and as immersion time increased, the shear strength of the bonding specimen slowly declined. This indicated that the post curing was beneficial to the bonding strength and the water resistance of bonding joints for the combined surface-treated polyethylene WPCs bonding with moisture-curable polyurethane hot melt adhesive.

The change of the shear strength demonstrated that the combined surface treatment on the polyethylene WPCs' surface improved the bonding strength of the composites, and it was beneficial to the improvement of the water resistance of the bonding joint of the composites.

Contact Angle

Table 1 shows the contact angle values and the corresponding standard deviations, of the surfaces for untreated, sanding-treated, and combined-treated WPCs. The distilled water on the surface of the polyethylene WPCs specimen was infiltrated for 20 s. As shown in Table 1, the contact angle values increased after the sanding treatment compared to that of the untreated specimen. This may have been due to the fact that the sanding treatment caused extreme roughness of the WPCs surface because the polyethylene layer on the surface was removed, which resulted in the delayed wetting. After the combined treatment, the contact angle of the surface of the polyethylene WPCs clearly decreased when compared with the surface of the untreated and sanding-treated samples. It indicated that the combined treatment resulted in a considerable change on the surface properties of the polyethylene WPCs.

Table 1. Contact Angles and Standard Deviations (SD) of the Surfaces of the Polyethylene-WPCs

	Untreated	Sanding-treated	Combined-treated
Contact angle (°)	86.5	108.8	38.3
SD	0.5	1.9	2.3

Surface Morphology

The surface morphology of the polyethylene WPCs specimens for untreated, sanding-treated, and combined-treated were analyzed by SEM images as shown in Fig. 4. As shown, the pristine sample surface was relatively smooth, and the molding process caused slight undulations on the surface. After the sanding treatment, the surface morphology of polyethylene WPCs changed substantially due to the increase of surface roughness. The surface roughness of WPC specimens after combined treatment decreased compared with the sanding treatment because of the discharging effect from the plasma discharge. The change on the roughness for the WPCs' surface influenced the wettability and adhesion of the material directly.

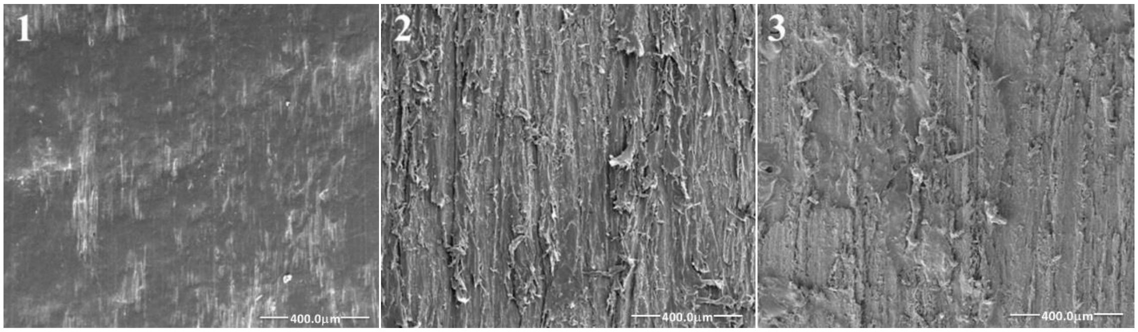


Fig. 4. SEM micrographs of the surface of polyethylene WPCs: 1-Untreated; 2-Sanding-treated; and 3-Combined-treated

FTIR Analysis

Figure 5 shows the infrared spectra of the surfaces of untreated, sanding-treated, and combined-treated polyethylene WPCs. As shown in spectrum 1, the surface of the untreated WPCs contained peaks from excessive $–CH_2$ symmetric and anti-symmetric stretchings (2918 cm^{-1} and 2848 cm^{-1}), and C–H in-plane bending vibration (1471 cm^{-1}), which confirmed that the polyethylene group was concentrated on the surface of the polyethylene-WPCs. After the sanding treatment, the surface indicated many characteristic absorption peaks of cellulose, hemicelluloses, and lignin (spectrum 2). In this spectrum the peak at 3348 cm^{-1} was assigned to the stretching vibration for –OH; the peak at 1721 cm^{-1} to the stretching vibration for C=O, and the peaks at 1593 cm^{-1} to the vibration for the benzene ring carbon skeleton of lignin, and the peaks at 1236 cm^{-1} and 1021 cm^{-1} to the stretching vibrations for C–O groups. As the sanding treatment removed most of the polyethylene layer on the surface of WPCs, the internal wood fiber that contained cellulose, hemicelluloses, and lignin was exposed (Kandey 1999; Stark and Matuana 2004).

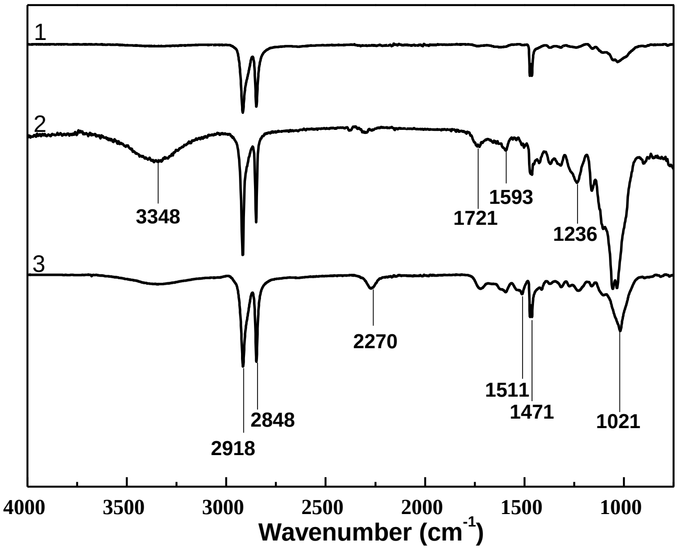


Fig. 5. Infrared spectra of the surface of polyethylene-WPCs: 1-Untreated; 2-Sanding-treated; and 3-Combined-treated

Spectrum 3 in Fig. 5 shows that the –N–C– stretching vibration peak (2270 cm^{-1}) and –NH bending vibration peak (1511 cm^{-1}) appeared on the specimen surface after the combined treatment. Compared with spectrum2, the –OH stretching (3348 cm^{-1}) and the –C–O stretching vibration peaks (1236 cm^{-1} and 1021 cm^{-1}) were weakened. Clearly, the –N=C=O groups in polyisocyanate reacted with the –OH groups in the wood fiber of the specimen surface, thus the –N–C=O and –NH groups formed on the material surface and the intensities of–OH and –C–O stretching decreased. At the same time, the peaks for polar groups, such as –OH, C=O, and C–O stretchings, also increased, which were attributed to the plasma discharge effect. The generation of new polar groups and the increase of oxygen-containing groups was beneficial for the wettability and adhesion of polyethylene-WPCs.

XPS Analysis

Table 2 illustrates the elemental content change on the surface of untreated, sanding-treated, and combined-treated polyethylene WPCs. As shown in Table 2, the elemental content of C for the untreated WPCs' surface was more than that of O, which showed that the WPCs' surface was mainly composed of the polyethylene component. The content of the carbon element decreased and the oxygen elemental content increased after the sanding treatment. Thus the ratio of O/C increased, and this trend continued after the combined treatment. It was also seen that the nitrogen element was generated after the combined treatment. The generation of the nitrogen element confirmed that the chemical bonds had formed between the polyisocyanate coupling agent and the surface of the WPCs formed. The change in the chemical elemental content was consistent with the FTIR analysis results, and the introduction of these polar groups had a positive role to improve the wettability and the adhesion of the surface for WPCs.

Table 2. Elemental Content of the Surface of Polyethylene-WPCs

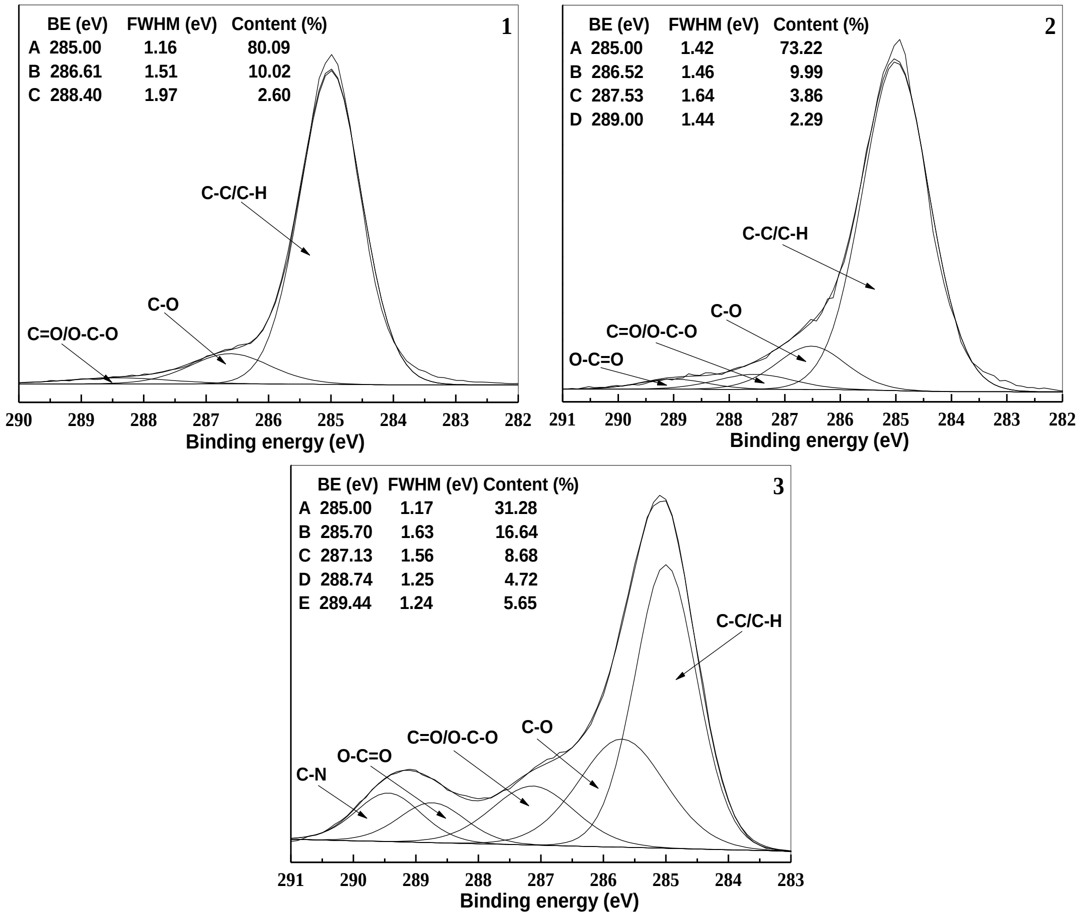
Treatment	C (%)	O (%)	N (%)	O/C
Untreated	92.70	7.30	-	0.079
Sanding-treated	89.36	10.63	-	0.119
Combined-treated	66.97	28.55	4.49	0.426

Figure 6 shows the curves for the C1s peak fitting on the surface of untreated, sanding-treated, and combined-treated polyethylene WPCs.

Figure 6-1 shows the curves for the C1s peak fitting of the untreated WPCs surface. It was fitted into three peaks (A, B, C), which corresponded to –C–C– and –C–H bonds (285.00 eV), –C–O– bond (286.61 eV), and –C=O and –O–C–O– bonds (288.40 eV). The relative content of –C–C– and –C–H bonds was 80.09%, which was attributed to the polyethylene layer of the WPCs' surface. The appearance of the carbon-oxygen bond was due to the presence of a small amount of wood powder contained and due to the slight oxidation by the air on the surface of the polyethylene WPCs.

Figure 6-2 shows the curves for the C1s peak fitting on the surface of the sanding-treated polyethylene WPCs. The C1s peak was fit into four peaks (A, B, C, D), which corresponded to –C–C– and –C–H bonds (285.00 eV), –C–O– bond (286.52 eV), –C=O and –O–C–O– bonds (287.53 eV), and an –O=C–O– bond (289.00 eV). As shown, the relative content of –C–C– and –C–H bonds decreased noticeably compared to Fig. 6-1, while the content of the carbon-oxygen bond increased, due to the formation of an –O=C–O– bond. All of these attributed to the disappearance of the polyethylene layer and thus the internal wood fibers were exposed after the sanding treatment. The wood fibers contained large amount of polar oxygen-containing groups and thus resulted in an increase of the content of carbon-oxygen bonds on the surface of the polyethylene WPCs.

Figure 6-3 shows the curves for the C1s peak fitting on the surface of the combined-treated polyethylene WPCs. The C1s peak was fit into five peaks (A, B, C, D, E), which corresponded to –C–C– and –C–H bonds (285.00 eV), –C–O– bond (285.70 eV), –C=O and –O–C–O– bonds (287.13 eV), –O=C–O– bond (288.74 eV), and –C–N bond (289.44 eV). As shown, the relative contents of –C–C– and –C–H bonds continued to reduce remarkably compared with that in Fig. 6-2. The generation of the –C–N bond was attributed to the –N=C=O group in polyisocyanate, which reacted with –OH on the surface of the polyethylene WPCs and thus introduced more oxygen-containing groups on the surface of the composites. The generation of oxygen-containing groups, such as –C–O–, –C=O, and –O–C=O groups, occurred during the process of the plasma discharge, and it led to a decrease in the contents of –C–C– and –C–H– bonds at the same time. These results illustrated that the surface properties of the polyethylene WPCs changed after the combined treatment, and thus resulted in changes in the adhesion properties.



1

	BE (eV)	FWHM (eV)	Content (%)
A	285.00	1.16	80.09
B	286.61	1.51	10.02
C	288.40	1.97	2.60

2

	BE (eV)	FWHM (eV)	Content (%)
A	285.00	1.42	73.22
B	286.52	1.46	9.99
C	287.53	1.64	3.86
D	289.00	1.44	2.29

3

	BE (eV)	FWHM (eV)	Content (%)
A	285.00	1.17	31.28
B	285.70	1.63	16.64
C	287.13	1.56	8.68
D	288.74	1.25	4.72
E	289.44	1.24	5.65

Fig. 6. C spectrum analysis of the surface of polyethylene WPCs: 1- untreated, 2- sanding-treated, and 3-combined-treated

Analysis and Discussion

The polyethylene component was enriched on the surface of the polyethylene WPCs throughout the molding process, and the adhesion was difficult due to the poor polarity and low surface energy for the polyethylene layer. However, when the surface of the composites was modified, the adhesion was improved.

As mentioned above, the sanding treatment of the composites only removed the polyethylene on the surface of the composites, and revealed the woody ingredient of the WPCs. It did not change the properties of polyethylene and wood fiber in the composites. For the combined surface treatment, the plasma treatment could improve the adhesion properties of the polyethylene component in the WPCs through means of increasing the polarity and the surface energy. In addition, the chemical bonding between the polyisocyanate with the woody ingredient in the polyethylene WPCs would be promoted by the plasma discharge. Specifically, both the polyethylene and wood fiber components present in the polyethylene WPCs were modified simultaneously by the combined treatment. The formation of the polar groups on the surface of the polyethylene and the chemical bonding of polyisocyanate on the surface of the wood fibers were beneficial for increasing the bonding properties of the composites.

CONCLUSIONS

1. A rapid bonding with desired adhesion properties of polyethylene WPCs was achieved by using a combined surface treatment of sanding, then coating with polyisocyanate, followed by plasma discharge. A moisture curable polyurethane hot melt adhesive was used to adhere the composites.
2. Following the combined treatment, the oxygen content of elements on the surface of the polyethylene WPCs increased, the carbon content of elements decreased, and the O/C increased. Meanwhile, a chemical bonding between the polyisocyanate and the surface of the composites formed after the combined treatment.
3. After the combined treatment, the adhesion property of the WPCs was improved considerably due to the synergetic effect of the oxidation of the polyethylene ingredients and the chemical bonding between the polyisocyanate and the hydroxyl groups on the surface of the composites.

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