

EFFECTS OF MODIFICATION WITH MELAMINE–UREA–FORMALDEHYDE RESIN ON THE PROPERTIES OF UREA PRETREATED EUCALYPTUS

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ABSTRACT

Eucalyptus urophylla was treated with melamine-urea-formaldehyde resin (MUF) after pretreatment with urea solution. The properties of the wood were then determined. The weight percent gain (WPG) and, antiswelling efficiencies (ASE) of the wood treated with urea+MUF were 14% and, 45% higher than those of untreated wood, respectively. The water absorption (WA) of the wood treated with urea+MUF was decreased approximately 50% lower than that of the untreated wood. However, the mechanical properties of the wood treated with urea+MUF were weakened due to the destruction of the structure of wood. An X-ray photoelectron spectroscopy (XPS) analysis revealed that the atomic concentration ratio of O/C was increased. The ratio of C1 was decreased as the lignin and extractives contents were decreased, while the ratios of C2 and C3 were increased. The urea solution pretreatment was conducive to the impregnation of wood functional modifiers.

KEY WORDS: Urea, pretreatment, melamine–urea–formaldehyde, wood properties.

INTRODUCTION

Plantation fast growing eucalyptus was introduced to locals worldwide because of the growing rapidity and short rotation (5-7 years). As of 2009, there were 95 countries with planted eucalyptus, with an eucalyptus area of 20.07 million hm² (Chen et al. 2018b). Eucalyptus is mainly used for particleboard, medium density fiberboard, wood pulp, and export wood chips in China (Chen et al. 2019, Tao and Chen 2016). The wood of eucalyptus is suitable for furniture and floor material because of its fineness, high hardness and beautiful tread patterns. However, the disadvantage of large growth stress, enrichment extractives, and poor permeability have led to dimensional instability and limited structural timber of eucalyptus (Lu et al. 2017, Zhou et al.

2019). Permeability is a physical property of porous materials (Chaiyo and Rattanadecho 2013), which reflects the porous structure and its interconnectivity. It may also provide useful information for practical wood applications, such as the optimization of impregnation with fluids to achieve protection against biological deteriorating agents and fire. Owing to the high porous structure variability between and within species, the permeability of species is crucial for wood modification and protection.

The permeability of wood is affected by wood species, fluids, infiltration processes, etc. Among them, the pore size, size and number of pore membranes, gum in the cell cavity, etc. were main factors affecting the permeability (Li et al. 2011, Lv et al. 2019). For eucalyptus, the main factors affecting the permeability are the extracts contained in wood and tylosis in the conduit (Silva et al. 2010). It is very challenging to effectively improve the permeability for the functional improvement of eucalyptus and utilization of high value-added solid wood. Detailed studies have been carried out on methods to improve the wood permeability (Comstock 1970, Esmailpour et al. 2019, Nicholasd 1977, Torgovnikov and Vinden 2009). Yang (2004) treated *Larix gmelinii* with different microwave powers and times, and reported that the microwave treatment destroyed the weak position on the cell wall and formed a fluid channel, which improved the permeability of *Larix gmelinii* without changing the mechanical properties of wood. Lv (2000) used alcohol to replace the water in the heartwood and sapwood of *Picea jezoensis* Carr. var. *microsperma*, and green wood of *Abies nephrolepis* to maintain with green wood for the pores state, while dissolving a part of the extract. As a result, the average permeability of the replacement wood was 101.5 times that of the ordinary air-dry material.

Since the 1990s, microwave, steam, and blasting technologies have been used to treat eucalyptus. Its microstructure and permeability have been studied (Li et al. 2007, Lv et al. 2001, Wang et al. 2013, Zhang and Cai 2008, Zhou et al. 2009). Owing to the evaporation of moisture or increase in air pressure inside the wood, which affected the weak parts such as the pore membrane, the microstructure of the wood was destroyed and eventually, the permeability was improved. Microwave, steam, and other technologies have improved the permeability of wood, but are complex and costly. To simplify the process and reduce costs, based on previous studies, the author (Qin et al. 2020) used urea solution to treat wood. The gas permeability of eucalyptus was increased by 40%, while the liquid permeability was increased by 180%. This provides a new method for a functional improvement of wood.

Based on the previous research, this study used melamine-urea-formaldehyde resin to modify eucalyptus, which was pretreated with urea solution to determine the properties of the modified wood, and investigated the effect of the urea solution pretreatment on wood functional improvement.

MATERIAL AND METHODS

Materials

After air drying, eucalyptus (*Eucalyptus urophylla*) was cut according to the required specifications, and the defects were removed. Wood was extracted using a benzene-ethanol mixed solution (2 : 1). 300 × 20 × 20 mm, seven pieces in one group, a total of three groups; 20 ×

20 × 20 mm, five pieces in one group, a total of four groups; 30 × 20 × 20 mm, seven pieces in one group, a total of three groups), and then placed in oven and dried to a constant weight at 103 ± 2°C. The size and weight of the sample were then recorded. Ethanol, benzene, and NaOH were purchased from Beijing Chemical Plant. Urea, formaldehyde and melamine were purchased from Xilong Science Co., Ltd.

Preparation of the MUF resin

Melamine, 37% formaldehyde, and urea solution (with the same amount of urea), were placed in four-round bottom flask. The mixture was adjusted to pH of 7.0 - 8.0 by adding 40% NaOH solution (the pH value was maintained throughout the reaction). The mixture was then heated to 75°C, maintained for 10 min. The temperature was then decreased to 50°C and maintained for 30 min to obtain the MUF resin. The final molar ratio of MUF (formaldehyde to urea and melamine) was 0.8. The viscosity and solid content of the MUF resin were 6.58 mPa.s and 42.6%, respectively (measured according to GB/T 14074, 2006).

Pretreatment and modification of test pieces

Samples (apart from the untreated sample) were placed in 20% urea solution and soaked for 36 h. Some of them were removed, and placed in a MUF solution (concentration of 30%). The impregnate MUF samples were then exposed to atmospheric pressure for 3 h after application of vacuum for 30 min (-0.095 MPa). Then, the solution on the surface of all samples was removed. All samples were placed in an oven to dry to a constant weight their weights were the measured.

Scanning electron microscopy (SEM) observation

Treated and untreated samples were adhered to double-sided tapes and then sputter-coated with a gold layer. SEM images were recorded using a ZeissEVO18SEM analyzer (Germany) with an acceleration voltage of 10-15 kV.

Physical property tests

The weight percent gain (WPG) was calculated for the treated samples by:

$$\text{WPG} = (W_1 - W_0) / W_0 \times 100 \quad (1)$$

where: W_0 and W_1 are represent the weights of the untreated and impregnated MUF wood specimens, respectively, after oven drying to achieve constant weight.

The treated and untreated specimens were placed into beakers (filled with water), subjected to vacuum for 30 min (-0.095 MPa), and then exposed to atmospheric pressure for 12 h. The weights and sizes of the samples were measured. The water absorption rate (WA) and anti-swelling efficiencies (ASE) of the samples during water absorption were calculated by the volumetric change:

$$\text{WA} = (M_2 - M_0) / M_0 \times 100 \quad (2)$$

where: M_0 is the oven dried weight of the sample and M_2 is the weight of the sample after water absorption:

$$ASE = (S_u - S_t) / S_u \times 100 \quad (3)$$

where: S_u represents the volumetric swelling of the untreated wood samples and S_t represents the volumetric swelling of the treated samples.

The volume change rate (S) is the percentage of sample volume change upon immersion in water,

$$S = (V_2 - V_0) / V_0 \times 100 \quad (4)$$

where: V_0 represents the oven dried volume before impregnation (cm^3) and V_2 represents the volume after immersion in water (cm^3).

The following equation was used to determine the volume bulking coefficient (B) (oven dried) owing to sample modification:

$$B = (V_1 - V_0) / V_0 \times 100 \quad (5)$$

where: V_1 represents the oven dried volume after impregnation (cm^3).

X-ray photoelectron spectroscopy (XPS) tests

Treated and untreated wood pieces were cut with a blade along the tangential direction of the sample, (size $10 \times 10 \times 2$ mm, longitudinal \times tangential \times radial). The energy of the wood surface was measured using photoelectron spectroscopy with a monochromatic Al target, full spectral step of 1 eV, energy of 160, narrow spectral step of 0.1 eV, energy of 40, and beam size of 300×700 μm .

Mechanical property tests

The bending properties (MOR) and compression strength (CS) were determined according to the Chinese standards GB/T 1936.1 (2009) and GB/T 1935 (2009). The lengths, widths, thicknesses (± 0.02 mm), and weights (± 0.01 g) of the samples were measured.

RESULTS AND DISCUSSION

Physical properties

The properties of wood are showed in Tab. 1. Compared to the MUF treated wood, the WPG of the MUF modified wood with the urea pretreatment was increased by 14%. After the pretreated with urea solution (pH = 7.2), the lignin, pectin, wax lipids and other substances

inside the wood reacted with urea or were dissolved, and the amide bonds of some protein molecules in the nitrogen containing compound were hydrolyzed (Chen et al. 2018a, Chen 2019), resulting in an increased internal porosity and significantly improved wood permeability (Qin et al. 2020). More MUF entered into the interior of wood, and thus the WPG of urea+MUF treated wood was higher than that of MUF treated samples. Urea removes a part of the lignin and extracts of wood to improve its permeability. Water and small urea molecules can enter into the cellulose noncrystalline area and swell the cellulose, resulting in higher WPG. The treatment agent entering the interior of the wood cell wall changed the wood volume, which led to gain *B* of the wood.

Tab. 1: Properties of the wood samples treated with different methods.

Treatment method	WPG (%)	WA (%)	S (%)	ASE (%)	B (%)
untreated	-	78.35 (27.00)	12.89 (3.56)	-	-
urea+MUF	8.31 (1.34)	40.20 (19.00)	7.26 (1.34)	43.70 (21.99)	6.65 (1.23)
urea	9.45 (2.50)	41.02 (15.78)	7.16 (2.37)	44.46 (18.38)	6.72 (0.77)
MUF	7.29 (6.56)	45.54 (14.42)	12.06 (4.56)	30.94 (17.55)	3.99 (1.50)

Note: The numbers in parentheses are standard deviations.

The WA and *S* values of treated samples were significantly reduced. Compared to the untreated wood, the WA of urea+MUF treated wood was decreased by approximately 50%. The hydroxyl groups on lignin easily form hydrogen bonds with water molecules. When urea-pretreated wood, lignin, hemicellulose, and extract were dissolved. The hydroxyl groups that would be combined with water were reduced, which reduced the WA of wood. The reduction in WA promoted an improvement in the dimensional stability of the wood and decrease in the volume expansion of water absorption. Owing to the dissolution of lignin, the wood shrinks (Dong 2018). These interaction of the two factors reduce the volume expansion of the urea-treatment wood.

Microstructure

The microstructures of wood samples treated by different methods are shown in Figs. 1 and 2. After the urea pretreatment, the porous structure of wood was not changed, but the cell wall became smoother and the pore membrane was damaged. There was a separation between the cell walls (Dong 2018). The lignin content in the intercellular layers was high. After the urea pretreatment, the horizontal penetration of the wood was increased, because the lignin was broke down, resulting in cell gaps and cracks in the pores of the wood cell wall, which was conducive to resin-impregnation. In addition, after the pretreatment with urea solution, a fraction of hemicellulose was dissolved, leading to the formation of many micropores on the wood cells. These micropores are conducive to the increase in the permeability of wood and penetration and diffusion of modifiers. Previous studies confirmed the improvement in wood permeability by urea treatment (Qin et al. 2020). The MUF resin deposited in the cell cavity basically disappeared after the pretreatment with urea, which indicates that, after the pretreatment with urea, the resin entered the cell wall, causing expansion of cell wall (swelling rate of 6.65%), and significantly improved the dimensional stability of wood. This is consistent with the above results.

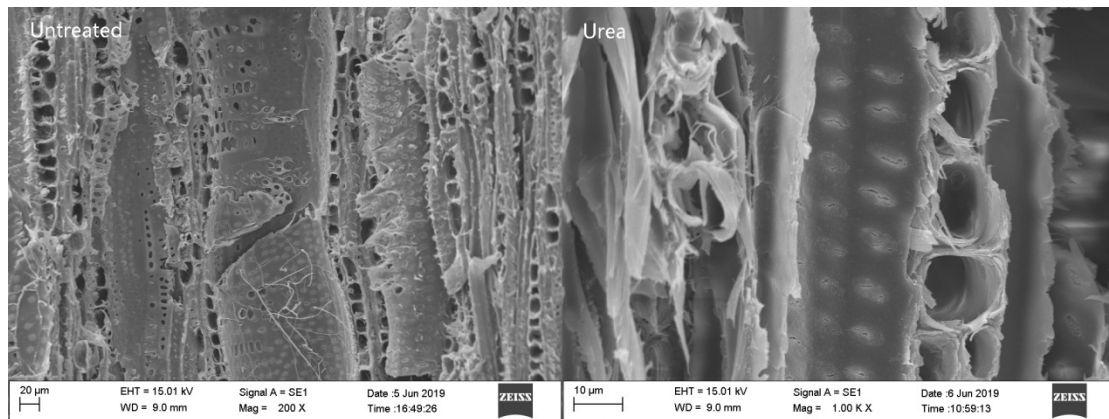


Fig. 1: Microstructures of untreated and 20% urea solution treated wood samples.

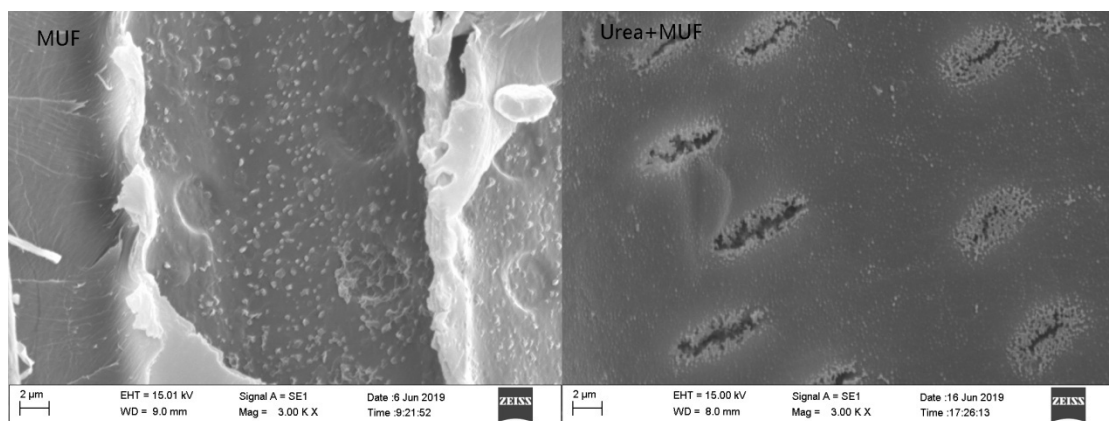


Fig. 2: Microstructures of MUF and urea+MUF treated wood samples.

Mechanical properties

The mechanical properties of wood are shown in Tab. 2. Compared to the untreated wood, the MOR and CS of urea+MUF treated wood were decreased by 7% and 8%, respectively, while the MOR and CS of MUF-treated wood was increased by 12% and 22%, respectively. The urea pretreatment had an adverse effect on the mechanical properties of wood.

Wood is mainly composed of cellulose, lignin, and hemicellulose. Cellulose is the main determinant of the strength and stiffness of the wood. Lignin has a supporting role in the hardness and toughness of the wood. After the impregnation with urea solution, the acids, lignin and other substances in the wood were partially dissolved. The urea solution infiltrated and swelled the cellulose, broke the bond between cellulose and hemicellulose, and destroyed the structure of the wood cell wall (Figs. 1 and 2), making wood prone to slack (Chen et al. 2018a). Many nanoscale pores were formed in the wood cell wall (Li et al. 2016), which were conducive to the impregnation of resin, while weakening the mechanical properties of wood. The resin was impregnated into the wood to form a polymer, which would crosslink with the hydroxyl groups of the wood at high temperatures, and enhanced the resistance of the wood to external forces. Therefore, the reductions in the MOR and CS of urea+MUF treated wood were due to the combined effect of the above factors.

Tab. 2: Properties of wood treated by different methods (moisture content 5.35 - 8.70%).

Approach	WPG (%)	MOR (MPa)	CS (MPa)
untreated		102.53 (25.03)	49.79 (3.97)
urea+MUF	4.00 (2.50)	94.99 (35.80)	46.43 (12.93)
MUF	1.63 (1.14)	114.91 (42.38)	60.73 (6.55)
urea	4.53 (2.13)	89.46 (20.08)	40.27 (4.19)

Note: The numbers in parentheses are standard deviations.

XPS analysis

Various properties of polymer materials are closely related to their surface compositions. Wood is a complex material composed mainly of lignin, cellulose, and hemicellulose. In addition to these polymer components, wood may contain large amounts of extractives.

XPS was used to analyze the surface chemical elements of eucalyptus. The contents of the elements on the surface of the wood sample are shown in Tab. 3. The XPS spectra of wood are shown in Fig. 3. All wood samples had strong peaks near 285 and 535 eV, which are the absorption peaks of C and O atoms, respectively. The main elements of eucalyptus were C, O, and small amount of N. The contents of these elements changed largely upon the treatments with different reagents. After MUF treatment, the content of C was decreased, while that of O and concentration ratio of O/C were increased. The O/C ratio from 0.26 increased to 0.46 for untreated wood. Tserki et al. (2005) reported that the O/C ratio closed to 0.35 can be explained by the removal of waxy substances on the surface of wood, and large exposure of hemicellulose and cellulose on the wood surface. The proportion of oxygen atoms on the surface increased, i.e., the lignin and extracts on the surface were reduced. However, when the urea solution was used to treat wood, the content of O decreased sharply. This indicates that the amounts of OH, C=O, and -COOH on the wood surface decreased (Wang et al. 2009). After urea+MUF treatment, the O/C ratio was close to 0.35. The urea+MUF treated wood was largely affected by urea, but the OH contents of the MUF and, urea+MUF treated wood samples were higher than that of urea treated samples. Thus, the WA after urea treatment was smaller than those after the MUF and urea+MUF treatments.

Tab. 3: Elemental composition on the surface of wood.

Approach	WPG (%)	Element (%)			
		C	O	N	O/C
untreated wood	-	79.11	20.63	0.26	0.26
MUF	7.29	60.29	27.76	11.94	0.46
urea + MUF	8.31	73.49	24.29	2.22	0.33
urea	9.45	79.91	1.16	18.90	0.24

The intensity of the N1s peak in the spectrum of the treated wood at 399.88 eV was increased. This indicated that N elements was introduced on the surface of the treated wood. The change in the peak intensities of C, O, and N showed that the wood surface had obvious chemical changes. However, the urea+MUF treated wood was affected by urea and the O atom content was lower than that of the MUF treated wood.

An XPS peak fitting diagram after C 1s peaking on the sample surface is shown in Fig. 4.

After the Gaussian fitting of the untreated wood C 1s spectrum, three C1, C2 and C3 peaks were obtained. This indicates three states of carbon on the wood surface. ① The carbon atom C1 (-C-C, C-H), which is only combined with C or H atoms, mainly originates from the carbon connected to the lignin, hemicellulose, and C-C groups in the extract (such as fatty acids and hydrogen groups in lignin and extracts). ② C2 was only bound to a single non-carbonyl oxygen, which is correspond to the O-CH group of lignin and C-O-C bond of wood extracts and polysaccharides. There is more C2 in cellulose and hemicellulose than in lignin, which is considered to be derived mainly from cellulose. ③ C3 is bound to the carbonyl group or two non-carbonyl oxygen atoms, which are considered to originate from the O-C-O structure and carbonyl group in the cellulose and hemicellulose molecules (C=O) (Inari et al. 2006, Sinn et al. 2001, Stark and Matuan 2007). The C 1s spectrum of the modified wood was also fitted with three peaks. This indicates that the type of carbon after treatment was not change. Tab. 4 shows that, after the MUF treatment, the proportion of C1 decreased, which was reflected in the spectrum as the height of the C1 peak became smaller and the peak area decreased, corresponding to the reduction of lignin and carbon in the extract connected to the carbon (C-C) group (Qin et al. 2019). However, the C1 ratio of urea treated wood was decreased slightly. The extracts and lignin reductions of the urea treated wood were confirmed by the previous analysis.

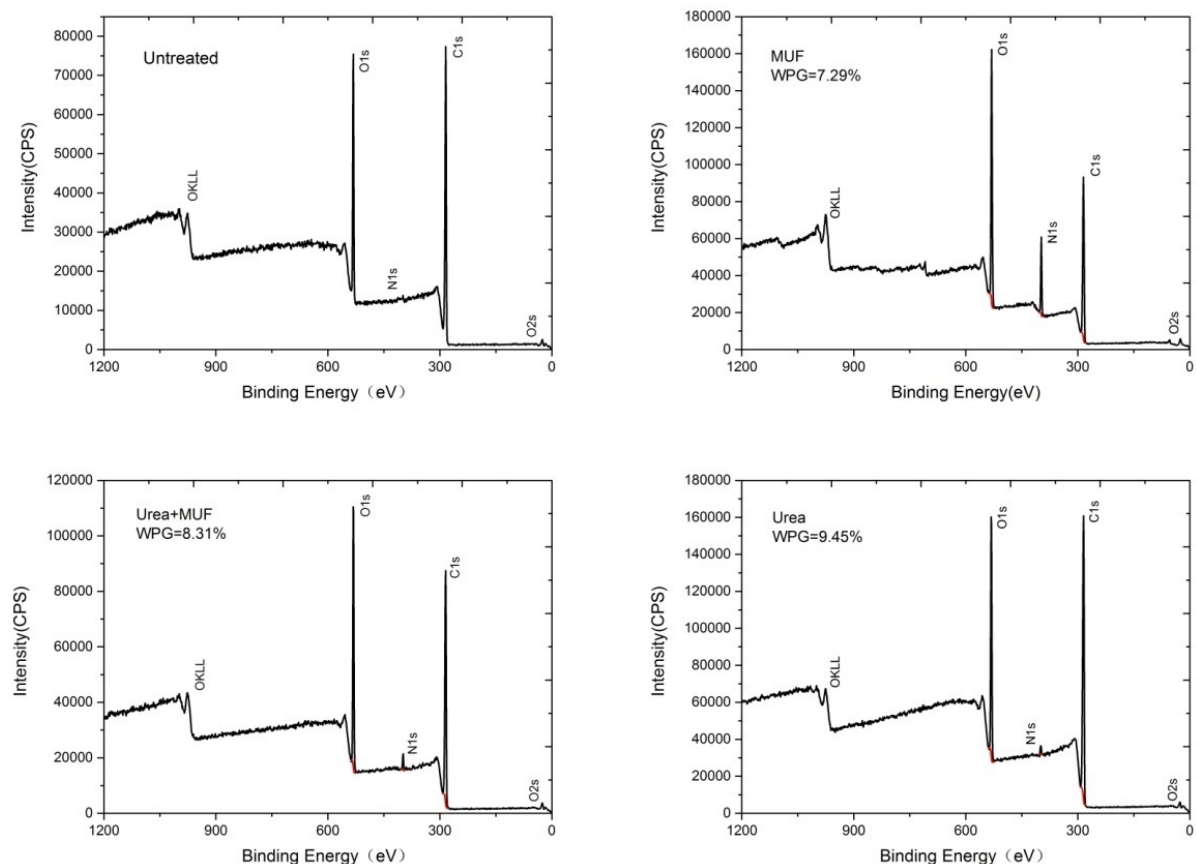


Fig. 3: Survey XPS of the treated and untreated eucalyptus wood.

After the treatment, the proportion of C2 varies largely due to the difference in treatment reagent and WPG. Because cellulose contributes more to C2, the change in C2 content indicated changed in the cellulose content on the wood surface. In the MUF impregnated wood, cellulose, and hemicellulose participated in the resin curing process, forming an ether bond, which resulted in a decrease C2 content. After the urea treatment, the wood surface C2 content increased by 20% compared to that of the untreated wood. The urea can decompose a part of the lignin, resulting in an increase in the cellulose and hemicellulose contents of the wood. The urea +MUF treated material was affected by urea, which increased the C2 content. The C3 contents of MUF and urea treated wood samples exhibited the opposite behavior, which may be caused by the different effects of MUF and urea on the chemical composition of the wood.

According to the above analysis, the changes in C1, C2, and C3 indicated that the MUF resin and urea caused chemical changes in the wood. A large number of -O-C=O bonds appeared in the wood after MUF treatment. The -O-C=O ratio was reduced after the urea treatment. This indicated that the cellulose and hemicellulose contents in the wood surface were reduced. The urea partially degraded wood cellulose and hemicellulose, which coincided with the wood damage (Fig. 1). In addition, the MUF would generate an -O-C=O bond after the reaction with the wood hydroxyl group. As a result, the contents of other C1 and C2 structures in the wood were relatively reduced.

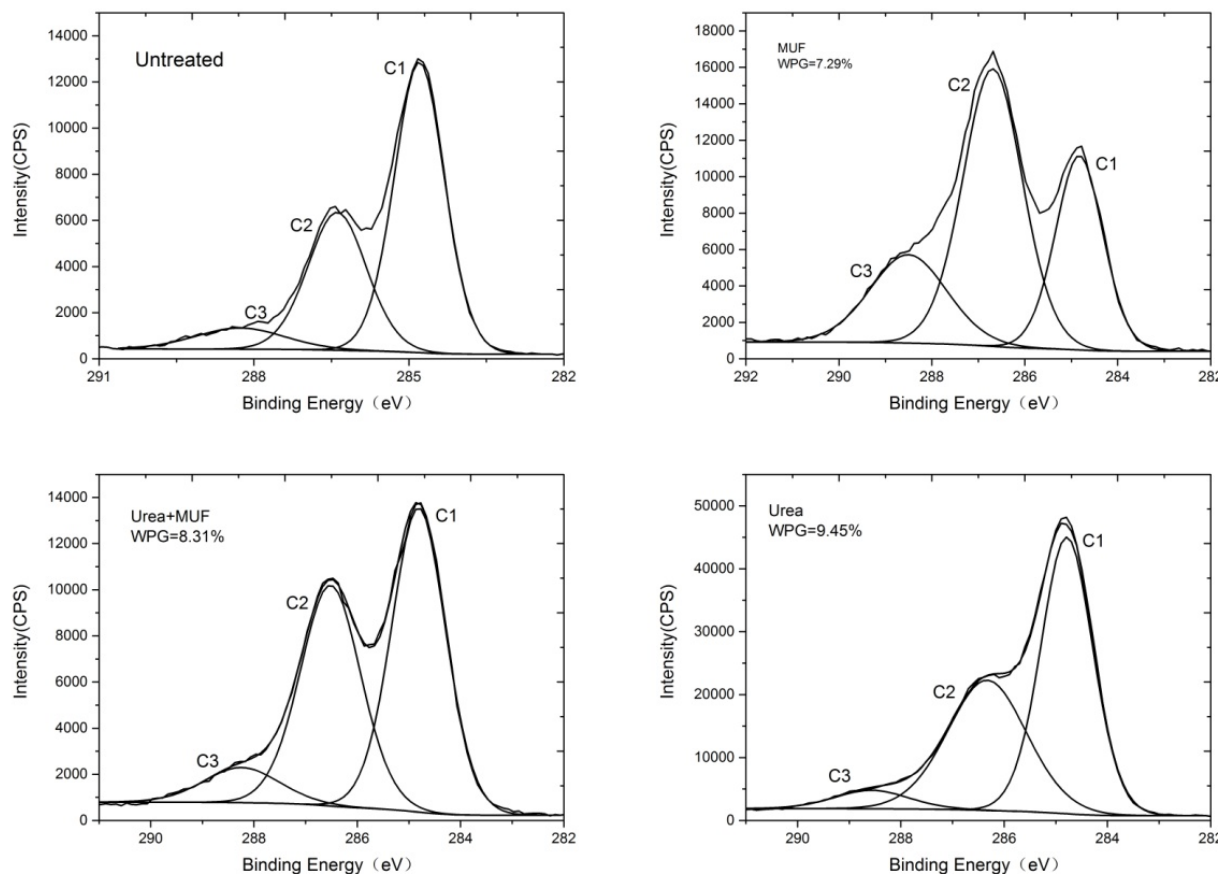


Fig. 4: XPS C 1s of eucalyptus treated and untreated wood surfaces.

Tab. 4: Elemental composition on the surface of eucalyptus.

Approach	WPG (%)	Fit peak area ratio		
		C1 (%)	C2 (%)	C3 (%)
untreated	-	59.40	32.95	7.65
MUF	7.29	50.80	28.41	20.79
urea + MUF	8.31	50.68	41.16	8.15
urea	9.45	55.17	39.54	5.28

CONCLUSION

The weakly alkaline urea improved the wood permeability by dissolving extracts and wood constituents in the wood structure and swollen cell walls. Compared to the MUF treatment of wood, the WPG of the urea+MUF treated wood was increased by 14%. The ASE was increased from 30% to 43.7%. The WA of the wood was reduced by approximately 50% compared to that of untreated wood. However, the mechanical properties of the urea+MUF treated wood were weakened owing to the destruction of the wood structure.

The XPS analysis revealed that the MUF and urea+MUF treatments caused obvious chemical changes on the wood surface. The C content decreased, while the O contents and ratio of O to C (O/C) increased. The proportion of C1 on the wood surface was reduced, mainly due to the reduced contents of lignin and extracts. The urea+MUF treated wood was affected by the urea-dissolved lignin, which led to an increase in the C2 content. The ratios of C3 of the MUF and urea+MUF treated wood samples were increased. This corresponded to the acetal structure in the wood cellulose and hemicellulose molecules. The contents of retinyl in lignin molecules were increased and decreased, respectively.

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