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Volatile organic compound and formaldehyde emissions from *Populus davidiana* wood treated with low molecular weight urea-formaldehyde resin

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Abstract

Populus davidiana wood was usually impregnated with low molecular weight thermosetting resins to improve its physical and mechanical properties. However, volatile organic compounds (VOCs) and formaldehyde emitted from treated wood have lead to poor indoor air quality (IAQ). The trends of VOC and formaldehyde emissions as a function of the weight percent gain (WPG) factor were mainly investigated in this work. Aldehydes and alkanes were the predominant compositions indentified in the VOC emissions, although low amount of ketones, terpenes and alcohols were also found. With the increase in WPG, VOC and formaldehyde concentrations improved. However, their concentration began to decrease when WPG was over 44.06% (VOC) and 36.35% (formaldehyde), respectively. The modulus of elasticity (MOE) of untreated and treated wood at different WPG levels was detected. It showed that treatment of wood with UF resin significantly improved the mechanical properties. Therefore, it is probably helpful to comprehensively analyze correlations among environmental performance, mechanical performance and processing costs.

Key words

Formaldehyde, IAQ, Impregnation, *Populus davidiana* wood, VOC

Introduction

With decrease in natural forest resources and implementation of protecting policies, fast growing wood has replaced natural forest wood as the main material to meet the increasing demand. Due to its characteristics such as low density, poor quality, and readily susceptible to biodeterioration, fast growing wood is limited to indoor applications. Therefore, an approach of penetration of low molecular weight resins into wood has been explored to improve its physical, mechanical and biological properties (Yu *et al*., 2011; Zhang *et al*., 2006; Ahmed and Moren, 2012). Impregnation of wood with melamine-formaldehyde resin results in increase in the MOE and dimensional stability (Deka *et al*., 2002; Cai *et al*., 2007). Low molecular phenol-formaldehyde (PF) resin could penetrate cell walls, leading to a clear increase in the dimensional stability and anticorrosion performance (Furuno *et al*., 2004). After treatment of wood with UF resin, the physical and mechanical properties significantly improved. The modulus of rupture (MOR) and MOE of treated samples increased by 21% and 5.62% compared with untreated wood at 33.8 WPG level, respectively (Deka and Saikia, 2000).

However, low-density wood treated with resin penetration also released VOCs and formaldehyde, resulting in poor IAQ (Hakan *et al*., 2010; Qian *et al*., 2007). VOCs and formaldehyde emitted from wood-based materials have become an issue of increasing concern. At present, no studies have been reported on VOC emissions from *Populus davidiana* wood treated with UF resin. The aim of this work was to examine the speciation of VOCs and the influence of different WPG levels on formaldehyde and VOC emissions from treated *Populus davidiana* wood. Furthermore, it will be discussed whether a sustainable reduction or control emission can be achieved by adjusting WPG.

Materials and Methods

Populus davidiana tree (forest farm of Shuangfeng, Tieli, China) was collected on October 11, 2013. The age of the tree was 20 yr and its diameter at breast height (DBH) was 38.50 cm. Non-deficient, knotless, normally grown (no pitch line, no reaction wood, no decay, and no insect or fungi damage) wood was cut into blocks of 300 mm × 100 mm × 20 mm (longitudinal × tangential × radial), which were dried to 5% moisture content. Low molecular weight urea-formaldehyde resin was produced by an adhesive factory (Chenlong biobased material company limited, Jilin, China) on November 13, 2013. The molar ratio of urea and formaldehyde was 1.05 and free formaldehyde content was 0.17%. The resin viscosity was 14.5 s at 20 ºC and the pH value was 8.0.

UF resin was impregnated into four blocks in each experimental factor using vacuum/pressure method according to the designed process parameters in Table 1. The maximum working pressure and vacuum were 1.50 MPa and -0.1 MPa, respectively. The vacuum pump reached -0.08 MPa within 30 s. Impregnated samples were maintained for 3 days in an air-dry state, dried at a temperature of 60 ºC until the moisture content was approximately 5%. The resin was cured at a temperature of 120 ºC for 2 hr. As soon as the samples cooled down, their edges were sealed with aluminum-coated adhesive tape; they were wrapped tightly in aluminum foil to prevent the release of edges and exposed areas.

Determination of VOC emission : Sampling and the equipment were in accordance with ISO 16000-9 (2006) and ISO 16000-6 (2011). Samples were tested in environmental glass test chambers with a volume of 15 l. A good comparability of such chambers with 1 m^3 test chamber for VOC emissions (Simplewell Scientific Co., Dongguan, China) has been demonstrated earlier (Shen *et al*., 2012). Clean and humidified air was led through the chamber at 250 ml min^{-1}, providing 1.0 air exchanges per hour. One panel without drying defects was selected and the loading factor was 2.5 m^2 m^{-3}.

The temperature was 23 ± 0.5 ºC in the chambers and the relative humidity was 50 ± 3%. VOC emissions were measured in the outlet port. Air samples were collected on a Tenax TA (200 mg, 60-80 mesh) at a flow rate of 150 ± 1 ml min^{-1} for a period of 20 min, which was a total air volume of 3 l.

Table 1 : Vacuum/pressure process parameters of treated wood with UF resin

Sample	Pressing time (hr)	Pressure (MPa)	Resin concentration (%)
A	2.5	0.85	32
B	4	0.85	32
C	4	1.05	24
D	4	0.95	32
E	4	1.05	32

After sampling, the tubes were thermally desorbed with a thermal desorption sampler (Beifen Instrument Technical Co., Beijing, China). They were then characterized and quantified by DSQ II gas chromatography and mass spectrometry (Thermo Fisher Scientific Inc., Waltham, USA). Thermal desorption conditions were as follows: desorption temperature, 280 ºC; desorption time, 5 min; injection time, 1 min; and split ratio, 30:1. A 30 m analytical column (HP-5MS, film 0.25 µm, i.d. 0.25 mm) was used in the GC. The temperature program was that an oven temperature of 40 ºC was maintained for 2 min, raised to 50 ºC @ 2 ºC min^{-1} and maintained for 4 min, increased to 150 ºC @ 5 ºC min^{-1} and maintained for 4 min, and finally raised to 250 ºC @ 10 ºC min^{-1} to be maintained for 8 min. The ion source of MS was EI and its temperature was 230 ºC. The scan mode was full scan (40--450 amu). The individual VOC was identified by using retention time and a standard mass spectra library with the match quality not less than 90% and quantified based on their response factors derived from the standard curves.

Determination of formaldehyde emission : The experimental chamber system was constructed in accordance with ASTM D 6007-02 (2002). The chambers (15 l) were located within a conditioned room maintained at 23 ºC. High purity nitrogen (N_2) was passed into the chamber to reduce the formaldehyde background concentration as the air source and the background concentration was less than 0.02 mg m^{-3}. Formaldehyde concentrations emitted from samples were measured at conditions of 25 ± 1 ºC and 50 ± 4% relative humidity. The loading factor was 0.95 m^2 m^{-3} and the ratio of air flow through the chamber (N/L) was 0.526 m h^{-1}. Air sampling was collected for 30 min at a flow rate of 1 l min^{-1}. The collection solution was analyzed by UV-2450 ultraviolet spectrophotometer (Shimadzu Co., Kyoto, Japan). A standard curve was prepared using six concentration levels for the quantitative determination of formaldehyde concentrations.

Measurement of mechanical properties : According to GB/T 1936.2 (2009), the MOE of treated and untreated woods was measured. The final results were obtained by average MOE of four samples.

Results and Discussion

The WPG levels of samples A--E were respectively 28.94, 36.35, 44.06, 49.05 and 55.11%. VOC emissions from impregnated wood were measured in a small chamber for 28 days. Aldehydes and alkanes were the predominant compositions indentified in the VOC emissions from *Populus davidiana* wood treated with low molecular weight UF resin, although low amount of ketones, terpenes and alcohols were also found (Table 2). The species detected in the VOC emissions did not change with the increase of WPG. The species of VOCs after 3 days were in accordance with the ones of VOCs after 28 days, but VOC concentration was constantly decreased.

Terpenes originated from wood extractives are important compounds in defense against insects and fungi (Makowski and Ohlmeyer, 2006b). Terpenes themselves do not cause problems related to indoor environments and human health. However, terpenes react with ozone and form stable products such as aldehydes, peroxides and condensed phase compounds, some of which are sensory eye and airway irritants (ECA, 2007). D-limonene and α-pinene occurred within the testing period (Table 2). Terpene emissions ranged from 5.70 to 3.18 μg m^{-3} after 3 days and varied between 3.08 and 2.32 μg m^{-3} after 28 days. The terpene composition measured in the study was consistent with the results of Long and Wang (2007), who detected D-limonene and α-pinene released from *Populus davidiana* at normal temperature. The level of terpene emissions is not comparable, because terpene concentrations were measured from raw *Populus davidiana* wood. Terpenes are volatile due to their high vapor pressures at ambient temperature. Consequently, the influence of drying temperature on terpene emissions is obvious (Makowski and Ohlmeyer, 2006a).

Aldehydes are the most universal compounds in the indoor air of new homes. With respect to low odour threshold, elevated concentrations of aldehydes can lead to problems with perceived indoor air quality (Hodgson *et al.*, 2002). Straight-chained aldehydes originated from free unsaturated fatty acids degraded through autoxidation (Makowski and Ohlmeyer, 2006b).

Hexanal, nonanal and decanal were the main components of aldehydes, which accounted for 59.31--79.82% of aldehydes after 3 days and 58.94--72.54% after 28 days (Table 2). Heptanal, bezaldehyde and octanal were also prevalent compouds measured in the VOC emissions from wood products. Hexanal, heptanal, bezaldehyde, octanal, nonanal and so on were detected from 53 particleboard and 16 medium-density fiberboard (MDF) samples, while nonanal and decanal were also determined from wood chip and MDF in the previous study (Baumann *et al.*, 2000; He *et al.*, 2012), which were similar to the results of the present study.

Aldehyde concentrations constantly reduced during the testing time (Fig. 1). Initially (after 1 day), aldehyde emissions varied from 50.98 to 71.62 μg m^{-3} (samples 55.11% and 44.06%) decreasing to concentrations of 20.27 and 30.55 μg m^{-3} (samples

Table 2 : VOC emissions from wood treated with UF resin at different WPG levels

Compound	VOC chamber concentration (μg m^{-3}) after									
	Samples									
	3 days					28 days				
	A	B	C	D	E	A	B	C	D	E
Aldehydes										
Hexanal	10.36	6.73	9.27	9.96	8.97	2.02	3.24	4.75	5.13	3.59
Heptanal	3.52	ND	ND	1.67	2.06	1.03	1.21	0.89	1.02	1.02
Benzaldehyde	7.05	9.33	12.84	6.61	5.24	3.36	4.26	5.78	2.44	3.82
Octanal	3.41	3.34	6.37	4.34	3.77	2.14	2.03	1.88	2.06	2.00
Nonanal	10.02	13.33	12.09	8.44	10.64	4.60	6.92	8.10	5.41	5.29
Dodecanal	4.57	6.30	4.60	5.30	3.94	1.63	2.23	1.93	0.95	1.92
Decanal	13.36	11.85	13.35	9.90	6.41	5.49	5.12	7.23	6.53	3.70
Terpenes										
α-Pinene	3.70	3.29	3.53	3.73	2.62	2.32	1.35	2.31	1.96	2.39
D-Limonene	1.51	2.12	1.57	1.97	1.19	ND	1.13	0.77	0.98	ND
Alkanes										
Nonane	4.40	5.59	4.16	2.97	2.34	1.60	1.33	1.01	1.10	1.23
Decane	3.01	3.15	2.78	2.81	1.09	1.32	1.41	0.94	1.25	1.04
Undecane	4.76	6.09	4.75	3.69	6.82	2.69	3.64	3.04	4.82	2.61
Dodecane	3.47	5.80	6.23	4.95	5.53	4.34	3.54	3.44	5.13	3.42
Other long-chain alkanes	39.41	38.44	39.12	38.07	30.73	18.61	22.31	22.86	18.59	22.37
Tetradecane	24.24	27.38	28.33	25.61	21.85	19.85	20.18	22.31	19.81	23.08
Pentadecane	12.98	10.59	16.02	15.40	12.80	12.03	8.26	10.15	12.33	9.71
Hexadecane	3.19	2.51	2.48	4.06	2.61	2.81	1.77	2.86	2.30	1.33
Ketones										
Acetophenone	7.28	12.92	11.51	7.57	6.96	5.10	5.58	8.69	4.78	5.78
5-Hepten-2-one, 6-methyl	6.73	3.12	4.26	3.44	2.07	1.25	2.86	1.89	1.44	1.72
others	40.92	34.28	37.81	41.16	45.04	26.35	30.72	26.06	28.08	22.07

ND, not detected; Others, including 3-methylheptyl acetate, 2-octanol, naphthalene and 2-methyl-naphthalene

29.49% and 44.06%) after 28 days. Furthermore, aldehyde emissions were clearly affected by WPG levels ($F > F_{0.01}$). With an increase in the WPG, aldehyde concentrations improved significantly, and then began to decrease. Aldehyde concentrations increased to 71.62 μg m^{-3} at 44.06 WPG level from 53.02 μg m^{-3} at 28.49 WPG level after 1 day, which increased by 35.08%. However, aldehyde concentrations reduced directly to 50.98 μg m^{-3} when WPG increased to 55.11%. F value was 34.05 after 1 day which was 22.99 more than the one after 28 days (Table 3). This indicated that the impact of WPG on aldehyde emissions at initial testing time was more obvious.

Alkanes emissions, such as nonane, decane, dodecane, tetradecane, and pentadecane, exceeded other composition emissions and accounted for more than 43% in the sum of VOC emissions (Table 2). Tetradecane and pentadecane were main compounds detected in VOC emissions. Nonane, dodecane and hexadecane also measured from MDF in a small environmental chamber in the previous study (Shen *et al.*, 2012). It was similar to the results in this work. Furthermore, tetradecane, pentadecane and hexadecane were tested from fiber drying without an adhesive (Huang, 2012), which indicated that the degradation of wood was probably responsible for the presence of alkanes. However, no clear source of alkanes were identified.

Emission pattern of alkanes was same as the one of aldehydes (Fig. 2). Alkane concentrations reduced to 62.43-66.61 μg m^{-3} (samples 28.94% and 44.06%) after 28 days from 126.68--96.46 μg m^{-3} (samples 55.11% and 44.06%) after 1 day. Furthermore, alkane emissions decreased more obviously on first 7 days and reduced slowly after 21 days. The effect of WPG on alkane emission was in accordance with the one on aldehydes. Table 4 showed that alkane emissions were obviously influenced by WPG at the initial testing time ($F > F_{0.01}$) and there was no effect at the anaphase testing time ($F < F_{0.05}$).

Therefore, VOC emissions are directly influenced by the degree of impregnation of low molecular weight thermosetting resin into low-density wood. The degree of impregnation was closely related to the chemical structure of impregnation compound and wood structure. Low molecular weight resin penetrated into hardwood by wood vessels and wood rays. Due to the structure of hardwood, the impregnating process acts as a multi-stage filtration of the resin. Relatively high molecular weight resins were kept in vessels, and low molecular weight resins coated the inner surface of the lumina of fiber and parenchyma cells. Only lower molecular weight resins (290 and 470 for PF resin) penetrated easily into the cell walls of the fiber and parenchyma cells (Furuno *et al.*, 2004). After curing of UF resins, the free radicals in wood activate high activity hydroxymethyl groups of the resins filling the voids of cell walls and inner surface of cell lumina and produce carbonyl (—C=O) instead of hydroxyl (—OH) and other cell wall materials (Gao and Li, 2007). Covalent bonds strengthen the cell walls.

An increase in resin content impregnated into the wood may improve VOC emission level. This is consistent with the results showing that with rising resin content, VOC emissions from wood-based panels also increased (Sun and Shen, 2010; He *et al.*, 2012). However, wood structure is more complicated than wood-based panel resin interface. After the resin content impregnated into wood (WPG) reached 44.06%, VOC concentrations began to decrease. This different emission pattern is likely due to decrease the wood porosity during the treatment of wood with excessive resin. When the resin content reached a certain value, superabundant resins that deposited in the wood vessels and surface of the cell walls would lead to blocked vessels and cell wall voids after curing of the resin (Wu *et al.*, 2012). Meanwhile, the resin molecules that bulked the cell walls form chemical bonds with hydroxyl groups of the cell wall materials causing the reduction of cell wall voids. In the porous

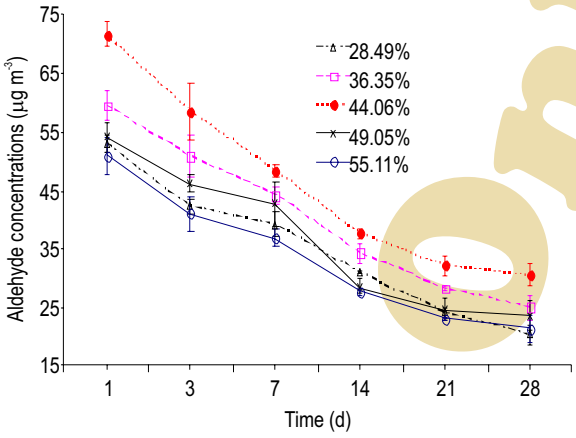

Fig. 1: Chamber concentrations of aldehydes from wood treated with UF resin at different WPG levels

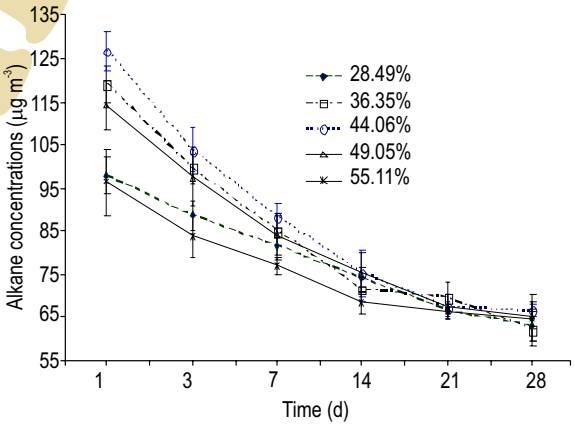

Fig. 2: Chamber concentrations of alkanes from wood treated with UF resin at different WPG levels

Table 3 : Variance analysis of aldehyde emissions after 1 d and 28 d

	SS	df	MS	F	$F_{0.01}$
1 day					
Between groups	829.83	4	207.46	34.05	5.99
Within groups	60.92	10	6.09		
Total	890.75	14			
28 days					
Between groups	194.85	4	48.71	11.06	6.00
Within groups	44.04	10	4.40		
Total	238.89	14			

material, the relation between the effective diffusion coefficient (D_e) and the total porosity (ε) could be demonstrated from the equation (Xiong *et al.*, 2008):

$$D_e = D_m \frac{\varepsilon}{\tau} \quad (1)$$

Where τ is the tortuosity factor; D_m is the mean (reference) diffusion coefficient in the pores of the material, which indicated that low wood porosity resulted in a decreasing VOC diffusion coefficient. When VOC concentration in the material is higher than VOC concentration in the small chamber, VOC diffuses from material interior to material surface. The mass transfer process is line with Fick's second law (Huang and Haghighat, 2002; Zhang and Xu, 2003; Yang *et al.*, 2001):

$$\frac{\partial C(x,t)}{\partial t} = D \frac{\partial^2 C(x,t)}{\partial x^2} \quad (2)$$

Where $C(x, t)$ is the VOC concentration in the material (μg m^{-3}), D is the VOC diffusion coefficient of the material (m^2 s^{-1}), x is the coordinate in which the VOC diffusion in the material takes place (m), t is the time (s). According to Eq. (2), it indicated that the diffusion coefficient directly contributed to VOC emissions. Therefore, excess resin impregnation would lead to a reduction of the wood porosity to prevent VOC release.

Formaldehyde emissions after 28 days and MOE of treated wood curves as a function of WPG factor are illustrated in Fig. 3. The trend in formaldehyde emission was similar to the one

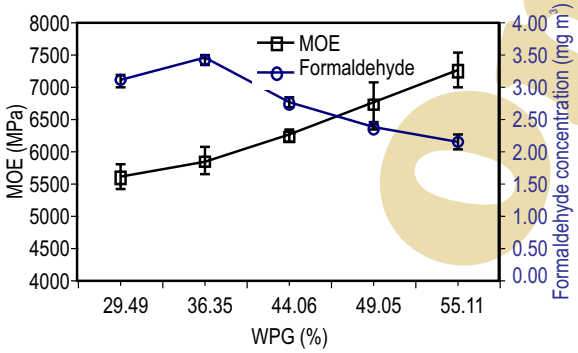

Fig. 3 : Chamber concentrations of formaldehyde emissions after 28 days and MOE of treated wood at different WPG levels

Table 4 : Variance analysis of alkane emission after 1 d and 28 d

	SS	**df**	**MS**	**F**	$F_{0.05}$	$F_{0.01}$
1 day						
Between groups	2114.71	4	528.68	18.22	3.48	6.00
Within groups	290.16	10	29.02			
Total	2404.87	14				
28 days						
Between groups	93.22	4	23.31	1.63	3.48	5.99
Within groups	143.23	10	14.32			
Total	236.45	14				

in VOC emissions, namely that with increasing WPG, formaldehyde increased to 3.45 mg m^{-3} from 3.12 mg m^{-3} and then clearly decreased to 2.17 mg m^{-3} when WPG was 55.11%. It is illustrated that formaldehyde emissions from treated wood are directly affected by resin content.

One of the reasons was identified in the previous analysis about the impact of WPG on VOC emissions. It was likely the other reason that in addition to reaction with the free radicals in wood, remaining resins reacted with each other to form more stable bonds when WPG was over 36.35%. With increase of WPG, this stable structure made it difficult to release free formaldehyde. Therefore, the inflection point of WPG in formaldehyde emissions was less compared with the one in VOC emissions.

The average MOE of untreated wood was 4664.81 MPa. Through impregnation of wood with UF resin, values of MOE increased by 19.45--55.19% (samples 28.49--55.11%). The relative standard deviation (RSD) varied from 1.66% (sample 44.06%) to 6.03% (sample 49.05%). It showed that treatment of wood with UF resin significantly improved the mechanical properties of wood. The improvement of MOE were owed to reinforcement of UF resins with the free radicals in wood (Wu *et al.*, 2012). It is consistent with previous research (Deka and Saikia, 2000).

Though an excessive amount of formaldehyde was measured in this work, general law of formaldehyde emissions from treated wood with random UF resins was mainly investigated. It should be considered how to reduce formaldehyde release from treated wood with UF resin in the next step of the work. Moreover, it was necessary to comprehensively analyze environmental performance, mechanical performance and processing costs to determine WPG according to different requirements, rather than blindly chase good impregnation performance.

Aldehydes and alkanes were the main compositions of VOC emissions from wood impregnated with UF resin. A small amount of terpenes, ketones and alcohols were also detected. Aldehyde and alkane emissions from treated wood constantly decayed over the testing period. With increasing WPG, there was

a clear increase in VOC concentrations. However, VOC emissions started to decrease when WPG continuously was over a certain value (44.06% in this work). Formaldehyde concentrations were directly related to the impregnation of UF resin and were influenced by WPG level. The trend in formaldehyde emissions along with WPG was similar to the one in VOC emissions.

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