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FLAME RETARDANCE AND ANTIBACTERIAL PERFORMANCE OF WOODEN WALLPAPER TREATED WITH COMPOSITE MODIFYING AGENT

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ABSTRACT

To improve the flame retardance and antibacterial properties of wooden wallpaper, a composite modifying agent, containing primarily a phosphorus-nitrogen flame retardant (APP/PER/GP), synergistic and smoke suppression agent (OMMT) and silver loaded with nano TiO_2 (Ag-TiO_2), was prepared and applied to the wooden wallpaper using an ultrasonic immersion method. The combustion performance (including smoke suppression performance), antibacterial activity and the surface micrographs of the flame retardant/antibacterial wooden wallpaper (FRAW) were investigated and characterized using a cone calorimeter test (CCT), antibacterial rate (AT) and inhibition zone, atomic force microscope (AFM), field emission scanning electron microscope (FE-SEM) and energy dispersive spectrometer (EDS). Furthermore, CIE1976 (L^* a^* b^*) color system was used to estimate the surface color change of the FRAW. It was found that the composite modifier distributed on the surface of FRAW with a micrometer, improved the flame retardance of FRAW, but also improved its antibacterial property. In addition, the smoke release behavior of the FRAW indicated that OMMT had good smoke suppression properties. Furthermore, the result of color difference test showed that the flame retardant and antibacterial treatment had little effect on the decorative appeal of the wooden wallpaper.

KEYWORDS: Wooden wallpaper, flame retardance, antibacterial properties, modification.

INTRODUCTION

Wallpaper is one of the most popular interior wall decoration materials, and as such, it has a great influence on the quality and safety of residential indoor air (Moriske et al. 1998; Yu et al.

2011; Usman et al. 1997). At present, the most common wallpapers are composed of polyvinyl chloride (PVC), generally arranged so that PVC is the surface layer and non woven paper is the bottom layer. In the event of a house fire, the flammable nature of the wallpaper can cause the flames to easily spread and rapidly expand along the wallpaper, threatening life and property. In addition, wall paper can harbor bacteria and other microbes that can adversely affect human health. Consequently, it is necessary to conduct research to determine how to impart flame retardancy and antibacterial properties to wallpaper.

Wood exhibits excellent comprehensive performance and it is a renewable resource. Wood wallpaper is composed of a decorative veneer (surface layer) and a non-woven paper (bottom base paper), making it an environmentally friendly interior decorating material. However, both wood and paper are flammability and contaminated materials. Consequently, this material, like PVC wallpaper, could also use surface modification to impart flame retardancy and antimicrobial properties.

A series of related studies have been devoted to the flame retardancy and antibacterial modification of wood (Li et al. 2012; Chen et al. 2012; Zuckeri et al. 2013; Quin and Zhang 2012), paper (Li et al. 2014; Pelton et al. 2006; Zhang et al. 2013) and other animal and plant fibers (Aguedach et al. 2008; Nassar and Youssef 2012; Zhang et al. 2015; Wang et al. 2013). With regard to flame retardant wood, Luo et al. (2003) examined this subject in depth and the authors reported on the flame retardant mechanism of wood treated with a variety of inorganic flame retardants including ammonium polyphosphate APP and other retardant additives, which were also detailed by Wang et al. (2014). And Rudra et al. (2013) reported on methods to impart antibacterial properties to paper. These previous research efforts have had a positive impact on improving the comprehensive safety performance of wood and paper. However, this research primarily involved improvement of a single property, focusing solely on flame retardancy or the sterility of the material. Studies which included a comprehensive examination of how to simultaneously decrease both the flammability and sterility of paper or wood have been sparse in the literature.

In this reported research, the goal is to prepare novel wood wallpaper with both flame retardancy and antibacterial activity (FRAW). To accomplish this, a composite modifying agents, were added to the wallpaper that included in ammonium polyphosphate (APP) which served as an acid and gas source of the intumescent flame retardant (IFR), pentaerythritol (PER) served as a source of carbon and guanidine phosphate (GP) was a flame retardant synergistic agent. In addition, nano organomontmorillonite (OMMT) was included to serve as a synergistic and smoke suppressant, and silver loaded with nano TiO_2 (Ag-TiO_2) was used as a bactericide. These agents were prepared and applied to wood wallpaper using an ultrasonic immersion method. The combustibility, smoke suppressing, antibacterial property, the surface micrographs and the color change of FRAW were investigated.

MATERIALS AND METHODS

Materials

A commercial decorative veneer (Sapele, *Entandrophragma cylindricum* used for wallpaper surface) was purchased from Beijing timber market. A non woven paper (used as the wallpaper base paper) and a PVC wallpaper (control sample) were provided by Shenyang Leadership Development Technology Co., Ltd. Chemical reagents ammonium polyphosphate (APP) (water soluble, degree of polymerization 10-20), pentaerythritol (PER), phosphoguanidine (GP), organic montmorillonite (OMMT), silver loaded nano titanium dioxide (Ag-TiO_2) and bamboo charcoal glutinous rice glue were purchased commercially. Bacteria cultures, ATCC 25922 (*Escherichia coli*) and ATCC 6538 (*Staphylococcus aureus*) were purchased from the Institute of Microbiology, Chinese Academy of Sciences.

Preparation of flame retardant and antibacterial wood wallpaper

Based on previous experiments, the content of APP and PER was m_{APP}: m_{PER}: m_{GP}=10: 4: 5. APP was first thoroughly mixed with PER for ten minutes in a 500 ml beaker to obtain a good soluble mixture at room temperature. Then 5 % OMMT (based on the quantity of APP) was added to the beaker and dissolved in the mixed solution. In succession, 2 % mass ratio of Ag-TiO_2 (based on the quantity of APP) was added to the beaker and stirredng for 5 min, and the beaker was immersed in a ultrasonic bath for 5 min at temperature 30°C. The mixture was stirred with a glass rod during the process so that the solid compounds dissolved in the aqueous solution. Finally, a multi component modifier was obtained with a degree of turbidity, and the concentration of the component modifier was set to 20 % based on the weight of APP.

The prepared modifier was applied to prepare the flame retardant/antibacterial wood wallpaper (FRAW). Using an ultrasonic impregnation equipment (Made in China, instrument model JP-020S) set up the temperature 35°C and time 5 min, and then, put the modifier into it, next, the decorative veneer and non woven paper were each soaked in the ultrasonic equipment so that to avoid pollution of non-woven paper because of the veneer fade. Following this, the samples were wiped with a dry rag and dried in a blast dryer at 60°C for 3 h. The appropriate amount of wallpaper glue was uniformly coated on the surface of wallpaper base paper, and the decorative veneer was covered with the paper using a roller to flatten along the width and length direction manually. This produced the final FRAW. All of the FRAW samples were conditioned at (23±1)°C with 50 % humidity for 48 h before testing the samples based on the Chinese national standard GB/T10739-2002.

Characterization techniques

The cone calorimeter test (CCT) was performed on the treated samples using a cone calorimeter instrument (Made in British, instrument model FTT 0006) according to standard of ISO5660-1:2002 with a sample size 100 × 100 × 0.5 mm. The composition of the produced smoke was determined according to ISO/DIS21489-2006 using an FTIR gas analysis spectrophotometer configured with FTT 0006, and a heat radiation flux of 35 $kW \cdot m^{-2}$. Three replicates samples were tested for each sample group. Antibacterial properties of the samples was determined based on the method of Japan standard (JIS Z 2801: 2012), in accordance with this standard, the control was standard polyethylene (PE). Based on this analysis procedure, the control and FRAW samples were cut into 50 ×50 mm, and all the samples were saturated with water vapor, then immediately used for the antibacterial test. The surface micrographs of FRAW were examined using an atomic force microscope (AFM) (BRUKER, Multimode 8) and field emission scanning electron microscope (FE-SEM) (Japan, instrument model JSM-7001F). And the energy spectrometer was installed on the FE-SEM instrument (instrument model is the same as above) as a subsidiary device, which can be used for quantitative determination of the specific point or area of the sample. The surface elemental composition of the FRAW was determined by energy dispersive spectroscopy (EDS).

The color changes of wood wallpaper before and after flame retardant/ antibacterial treatment were evaluated using a color measuring instrument (China, instrument model Data-color110) according to China national standard GB/T7921-2008. The CIE1976 (L^*a^* b^*) color system is used to characterize the color space. The sample moisture content was 10 %. The color test parameters were: D65 standard light source, color temperature 6 504 K, 10° wide eyeshot, measured hole diameter 20 mm. All samples were replicated in 3 groups, each group of samples was determined by 6 points and the average value of the data was used for comparison. The color differences between the two samples were calculated according to Eq.:

$$\Delta Eab^{*}=[(\Delta L^{*})^{2} + (\Delta a^{*})^{2} + (\Delta b^{*})^{2}]^{1/2}$$

where: ΔEab^{*} the color difference of two samples
ΔL^{*} lightness difference,
$\Delta L^{*} = L_{sample}^{*} - L_{control}^{*}$.
a^{*} and b^{*} chromaticity index difference respectively, a^{*} for the red and green opposite dimension, $\Delta a^{*}= a_{sample}^{*} - a_{control}^{*}$, b^{*} for the blue and yellow opposite dimension,
$\Delta b^{*}= b_{sample}^{*}- b_{control}^{*}$.

RESULTS AND DISCUSSION

Combustion performance and smoke suppression

The CCT data of the control sample (untreated wood wallpaper), treated wood wallpapers and PVC wallpaper are listed in Tab. 1. These data show combustion performance based on the following parameters: TTI - time to ignition, HRR- heat release rate, PHRR- peak heat release rate, THR-total heat release, EHC- effective heat of comb., TSR- total smoke release, SEA-specific extinction area, and COY- carbon monoxide yield.

Tab. 1: CCT data for control, treated wood wallpaper and PVC wallpaper.

Sample	TTI (s)	HRR/ ($kW.m^{-2}$)	PHRR/ ($kW.m^{-2}$)	THR/ ($MJ.m^{-2}$)	EHC/ ($MJ.kg^{-1}$)	TSR/ ($m^{-2}.m^{-2}$)	SEA/ ($m^{2}.kg^{-1}$)	COY/ ($kg.kg^{-1}$)
Control	9	207.0	708.9	9.3	31.8	63.1	212.97	0.0584
APP/PER/GP	10	64.5	115.1	3.3	14.9	147.4	681.03	0.0525
APP/PER/ GP+OMMT	11	45.8	76.3	2.1	10.0	117.2	568.6	0.0373
APP/PER/ GP+OMMT +Ag-TiO_2 (FRAW)	14	40.6	143.7	4.1	13.2	148.4	467.1	0.0456
PVC wallpaper	5	77.2	407.9	7.4	30.9	267.4	1124.5	0.1141

The CCT data comprehensively describe the heat release parameters and smoke parameters when samples achieved combustion. It can be seen from Tab. 2 that compared to the control sample, the TTI of the treated wood wallpapers was remarkably prolonged and the values of HRR, PHRR, THR and EHC also decreased. The HRR and the peak PHRR are usually considered as the most effective and important parameters for characterizing the fire behavior of a material (Babrauskas and Peacock 1992; Hansen 2002). The heat release behavior of the wood wallpaper with various processing conditions was shown in Fig. 1 where the HRR curves for the samples with APP/PER/GP were wide and the peak values further decreased following addition OMMT. This demonstrated, the synergistic effect of OMMT on the flame retardant system. Compared with the control, the PHRR of the FRAW clearly decreased and went backwards, meaning that its combustion process was slower. The intumescent flame retardant system containing nitrogen and phosphorus caused the effect a simultaneous flame retardant effect in condensed phase and gas phase, and the addition of OMMT and Ag-TiO_2 also resulted in a positive synergistic effect. Furthermore, the TTI value was delayed 2.25 times, and the HRR, TSH and COY decreased by 47.4, 44.5 and 60 % respectively in comparison to the PVC wallpaper, demonstrating the flame retardant advantages offered by the FRAW.

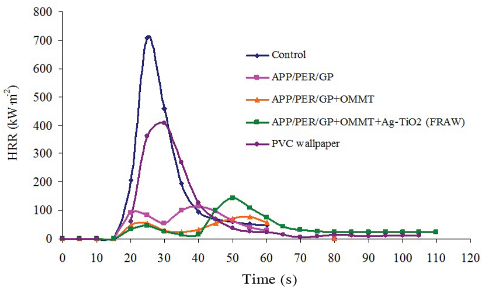

Fig. 1: Heat release behavior of wood wallpaper following various processing conditions.

The smoke release parameters for the wood wallpaper treated with the different flame retardants is shown in Fig. 2. It can be seen that the untreated wood wallpaper had the lowest smoke release. In fact, the complete combustion of treated material was inhibited, which resulted in an increase in the degree of smoke emission. Compared with the wood wallpaper treated using APP/PER/GP, the smoke release of the FRAW was significantly reduced, meaning both OMMT and Ag-TiO_2 had suppressed smoke emission. The reason for this was that the flame retardant containing OMMT and Ag-TiO_2 can melt into a sticky substance at high temperature, which covered the surface of the FRAW, forming barrier layer to oxygen and heat. This played an important role in preventing the release of the smoke. Compared with the PVC wallpaper, the amount of smoke released and the time to peak combustion of the FRAW was significantly reduced and delayed, exhibiting better safety performance.

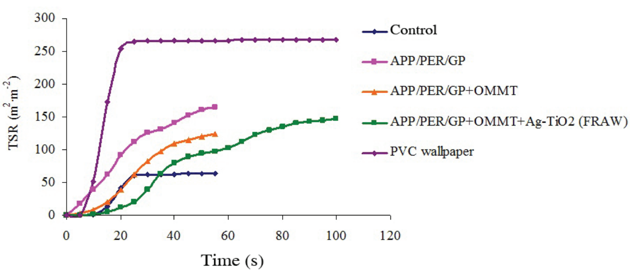

Fig. 2: Total smoke release behavior of wood wallpaper with various processing conditions.

Fig. 3 showed the type and concentration of smoke produced by the FRAW and PVC wallpapers during the combustion. As shown, the concentration of NO_2 and NH_3 released from the burning FRAW is much higher than that of the PVC wallpaper. In fact, both the released NO_2 and NH_3 can dilute the oxygen content in the air, slowing the burn rate of the treated wallpaper. This phenomenon also validated that the compound modifier composed of a flame retardant (APP/PER/GP), synergistic agent (OMMT) and antibacterial agent (Ag-TiO_2)

provided the added effect of a gas phase flame retardant. Fig. 3 also showed that the burning PVC wallpaper released far more noxious gases than FRAW, indicating that the PVC material could the cause more serious hazards for humans when a fire occurs.

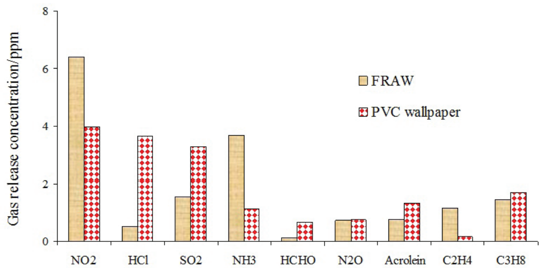

Fig. 3: Type and concentration of gases released from the FRAW and PVC wallpapers.

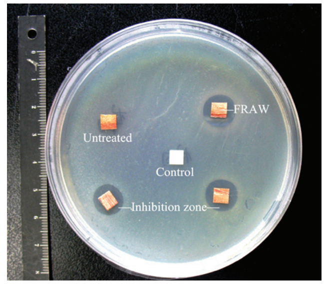

Fig. 4: Inhibition zones for Escherichia coli of FRAW.

Antibacterial property

The antibacterial property of the control sample and FRAW were shown in Tab. 2, and Fig. 3 was the inhibition zone produced for *Escherichia coli*. Although the antibacterial activity of the FRAW on *Escherichia coli* was higher than it was against *Staphylococcus aureus*, the antibacterial rate of the FRAW to two kinds of bacteria attained 99 %, meaning the composite modifying agent containing flame retardant and antibacterial agent possessed a synergistic antibacterial effect. The antibacterial mechanism of this coating appeared to be due to the Ag+ from the Ag-TiO_2 that penetrated the cell wall of the bacteria and cause protein coagulation. This effect would damage the activity of cell enzymes, resulting in the loss of cell reproductive capacity and eventual organism death (Youssef et al. 2013). In addition, the NH_3+ from APP and GP can interfere with the respiratory function of the bacteria and prevent nutrients from feeding the microbial cells, thereby playing a role in antimicrobial sterilization (Shi and Neoh 2006; Gao et al. 2012).

Tab. 2: Antibacterial results of control sample and FRAW.

Sample	Escherichia coli				Staphylococcus aureus			
	Average survival bacterium number (CFU•cm^{-2})		Antibacterial activity value	Antibacterial rate (%)	Average survival bacterium number/ (CFU•cm^{-2})		Antibacterial activity value	Antibacterial rate (%)
	0	24 h			0	24 h		
Control	1.8×104	4.0×106	-	-	2.2×104	1.0×105	-	-
FRAW	-	<1.3	>6.5	>99	-	<1.3	>4.9	>99

FE-SEM, AFM and EDS analysis

Fig. 5 illustrates the FE-SEM micrographs of the control and FRAW. It can be seen that comparer to the untreated sample having clean cell wall surface, there were many fine grains distribution on the fiber surface of FRAW. AFM provides a method for accurately evaluating the surface morphology change of materials, and the test result of AMF (showed in Fig. 6) proved that these particles on the surface layer of FRAW formed a covering layer with a certain thickness. In fact, these particles of composite modifying agent isolated a part of the heat and oxygen circulation channels, therefore prevented the spread of the flame when the wood wallpapers burning. Meanwhile, these modifiers can decomposed to produce non combustible gas (such as ammonia, nitrogen dioxide, and so on) when the temperature was increased, which had the function of reducing the system temperature, reducing the oxygen concentration and shielding heat radiation. Meanwhile, the compound modifier of antibacterial ingredients (as set out silver titanium dioxide) was covered on the cell surface of FRAW, inhibited the bacterium damage to wood wallpaper.

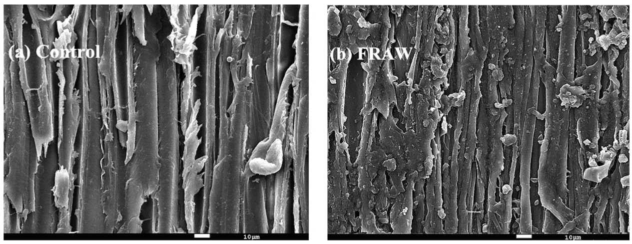

Fig. 5: FE-SEM micrographs of the control sample and FRAW (×600).

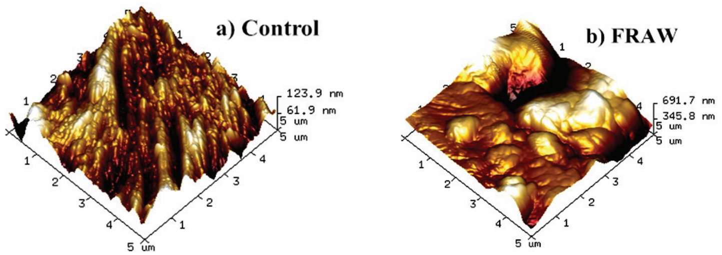

Fig. 6: AFM 3-dimensional images of the control and FRAW.

EDS analysis provided the elemental information for the material surfaces. The surface elemental compositions of the control and FRAW are shown in Tab. 3 and Fig. 7. The EDS readings reveal that the major elements on the surface of the FRAW were C, O, P, N. The nitrogen and phosphorus from the APP and GP played an important role in improving the flame retardant property of the FRAW. The Fig. 6 also shows the presence of Ti element and Ag element in FRAW, these two elements are of great significance for improving the antibacterial performance of the FRAW.

Tab. 3: EDS analysis of the control and FRAW.

Sample		**Major element compositions**							
		C	**O**	**N**	**P**	**Si**	**Al**	**Ti**	**Ag**
Control	Wt/%	56.71	43.29	-	-	-	-	-	-
	At/%	63.57	36.43	-	-	-	-	-	-
FRAW	Wt/%	12.78	40.92	37.42	4.74	2.28	0.67	0.48	0.10
	At/%	16.27	39.09	40.83	2.34	0.70	0.39	0.15	0.01

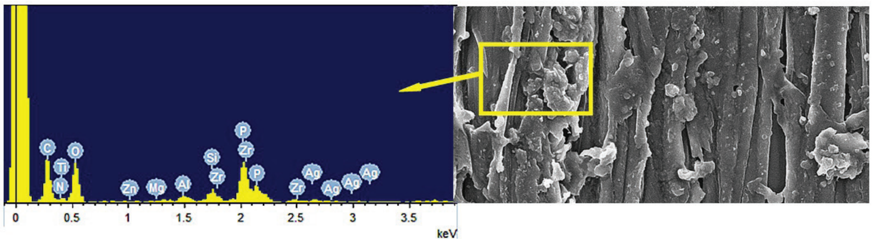

Fig. 7: EDS analysis of FRAW treated by composite modifier.

CIE1976 ($L^*a^*b^*$) analysis

Many evaluation methods are used to characterize the decorative effect of wood surface (Wang and Zhang 2015), in which the color change is usually evaluated by the uniform color space and the color difference formula. CIE1976 ($L^*a^* b^*$) color space can be used to characterization the color difference between the samples before and after treatment by color difference value ΔE_{ab}^*. Tab. 4 showed the CIE 1976 ($L^*a^*b^*$) results for the wood wallpaper before and after modification. It can be seen from the results in Tab. 4 that compared to the control sample (untreaed), the ΔL^* of the FRAW was negative meaning the lightness of the treated sample decreased slightly. At the same time, Δa^* was positive, indicating that the FRAW was more red (or less green); Δb* was negative, showing that the FRAW was blue (or less yellow). Overall, the ΔE_{ab}^* value of FRAW was only 1.734, indicating that the modification treatment of the wood wallpaper caused little change in the decorative effect of the product.

Tab. 4: CIE1976 (L^* a^* b^*) test results of the control and FRAW.

Sample	L^*	ΔL^*	a^*	Δa^*	b^*	Δb^*	ΔEab^*
Control	65.632	0	12.346	0	23.065	0	1.734
FRAW	64.025	-1.507	12.715	0.369	22.548	-0.517	

CONCLUSIONS

In this reported study, a novel composite modifying agent was prepared and applied to wood wallpaper to impart flame retardancy and antibacterial activity. The results showed that the FRAW treated with a composite modifying agent using ultrasonic impregnation produced a material with good flame resistance and antibacterial properties. Compared with PVC wallpaper, the TTI of the FRAW was delayed 2.25 times, and the HRR, TSH and COY also decreased by 47.4, 44.5 and 60 % respectively. Also and harmful gases produced during the combustion of the FRAW were also significantly reduced. In addition, the antibacterial activity of the FRAW for *Escherichia coli* and *Staphylococcus aureus* attained more than 99 %. Furthermore, both FE-SEM and AFM analysis revealed that the composite modifying agent covered the surface of the FRAW with micro-sized particles. Finally, CIE1976 ($L^*a^*b^*$) tests showed that the modification treatment did not adversely affect the decorative features of the wood wallpaper. All of these data prove that the FRAW has a significant advantage compared over PVC wallpaper. This work shows the potential of the proposed application of functional wooden wallpaper with flame retardant and antibacterial properties for use as interior wall decorative materials.

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