Development of Photochromic Wood Material by Microcapsules

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To develop a smart, colour-changing wood material, photochromic microcapsules were incorporated into coatings while painting veneered plywood. The properties of microcapsules and coatings were investigated. The colour-changing behaviour of the photochromic wood material in response to sunlight exposure was evaluated. The microcapsules exhibited sensitive colour-changing function and had good thermal stability. The prepared photochromic wood material spontaneously altered its appearance from the veneer colour to a blue colour following intensity changes of the sunlight exposure on the sample. The incorporation of microcapsules had no obvious effect on coating adhesion, but it obviously reduced coating wearability. With the microcapsule content increasing from 2.5% to 10% (of the coating weight), the colour difference (Δ*E*) of photochromic wood stimulated by sunlight linearly increased from 7.45 to 21.58. The performance of the prepared photochromic wood material can be adjusted by controlling the addition amount of microcapsules.

Keywords: Photochromic wood material; Microcapsule; Colour-changing behaviour; Smart materials; Coating

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INTRODUCTION

Wood is one of the most widely used home furnishing materials, and it is favoured for its unique texture, grain, colour, gloss, and good processability. To improve the durability and fire safety of this biodegradable material, various chemical or physical methods have been utilized to manufacture preservative-treated and fire-retardant wood products (Marney and Russell 2008; Lowden and Hull 2013). Furthermore, many new functional materials such as dyed wood, electromagnetic shielding plywood, sound absorption wood-based material, wooden electric heating composite, and wood-based flexible supercapacitors have been developed to expand the application range of wood materials and to improve the added value of wood products (Lu *et al.* 2014; Yuan and Fu 2014; Liu *et al.* 2015; Lv *et al.* 2015; Smardzewski *et al.* 2015). With continuous increase of consumer demands for high-quality living environments, it is necessary to develop smart wood materials that spontaneously change their properties with the variation of environmental factors (Li *et al.* 2010).

As a new type of smart materials, reversible colour-changing compounds display repeated colour-changing features under external stimuli such as temperature, light, electricity, pressure, and magnetism; they also have potential applications in building, textiles, printing, electronics, and advanced materials (Ferrara and Bengisu 2014). Thermochromic wood, which has reversible colour-changing properties in response to

changes in temperature, has been developed by impregnating veneers with a thermochromic agent consisting of a dye, a chromogenic agent, and a solvent (Liu *et al.* 2011).

The dynamic colour-changing features can enrich the decorative effect of wood materials and enable them to be used as temperature indicators (Basnec *et al.* 2014). Photochromic compounds are also important smart colour-changing materials with possible applications to wood products; reversible colour changes are induced by ultraviolet or visible light (Li *et al.* 2010). The unique colour-changing features enable photochromic materials to be applied both in daily life such as smart textiles, toys, security printing, and architecture (Chowdhury *et al.* 2014) and in high-tech fields such as camouflage and ultraviolet reactors (Solari *et al.* 2015). Some physical properties of photochromic materials, such as absorption spectra, fluorescence emission, electron conductivity, and magnetic properties, may be tuned by light during the reversible colour-changing process. Such possibilities have motivated people to apply such technology to data storage, molecular switches, and energy harvesting (Zhang *et al.* 2013; Wahid *et al.* 2015). Recently, photochromic wood material was also prepared by forming a film consisting of organic photochromic materials, polyvinyl alcohol, and dextrin on the surface of wood blocks, and it showed excellent coloration and fading functions under the ultraviolet irradiation and visual irradiation from the solar simulator, respectively (Hui *et al.* 2015).

For both thermochromic and photochromic materials, microencapsulation covers them with an integrated and firm shell, which protects them from leaching and environmental effects and allows more flexibility in their manufacturing and use (Ferrara and Bengisu 2014). The majority of commercial thermochromic and photochromic products are in the form of powders or microcapsule suspensions. Wood materials suffer from heat, pressure, and friction during processing and utilization, which requires that colour-changing materials have high stability. Thus, microencapsulated colour-changing agents are more suitable to develop smart wood products.

In previous studies, microcapsules have been successfully incorporated into wood materials by means of pressure impregnation and blending with adhesives (Hayward *et al.* 2014; Duan *et al.* 2015). Adding microcapsules into coatings is another potential way to apply them to wood substrates because wood materials often require finishing treatments before use. As the colour-changing features of materials only refer to the colour variation of their surface layers, it would be more cost-effective to treat merely the surface part of the substrates than to treat the whole of them while preparing thermochromic and photochromic composites. Adding thermochromic and photochromic microcapsules into coatings is a preferable way to combine them with wood materials. However, the addition of microcapsules and their effects on coating performance need to be studied.

The aim of this study was to develop a photochromic wood material by adding microcapsules into coatings while painting wood-based panels. The morphologies, chemical structures, and thermal properties of microcapsules were characterized. The thickness, wearability, and adhesion of the coating as well as the distribution of microcapsules in the coating were investigated. Four different addition levels of microcapsules were tested. Colour changes in the photochromic wood after sunlight exposure were evaluated by a chroma meter, and a simple application of this methodology was performed.

EXPERIMENTAL

Materials

Commercial 7-ply eucalyptus plywood overlaid with one layer of 0.5-mm red cherry veneer was supplied by the Treezo Group (Zhejiang, China). The veneering plywood had a moisture content of 8.4% and a density of 520.0 kg/m^3, and its thickness was 11.9 mm. Sample panels with dimensions of 100 mm × 100 mm were cut from the faced plywood. Photochromic microcapsules, which consisted of a melamine-formaldehyde (MF) shell and a spirooxazine compound core, were provided by the Peking Boda Green Hi-Tech Co. (Beijing, China). IVY® pure water-based wood coating containing acrylic and polyurethane dispersions with a solid content of 32.4% was purchased from PPG Industries (Shanghai, China).

Preparation of Photochromic Wood Material

The photochromic wood material was fabricated by incorporating microcapsules into the formulation and process for painting (Fig. 1). Sixteen grams of wood coating was diluted in 4 g distilled water. Microcapsules were added to the diluted coating to final concentrations of 2.5%, 5%, 7.5%, and 10% of the total weight, and the mixture was stirred for 10 min to form a homogeneous suspension. The photochromic suspension (100 g/m^2) was evenly brushed over the sample panels. A thin film of each suspension was painted on a clear glass slide. The coated sample panels were placed indoors for two weeks before testing. Panels coated without microcapsules were prepared as controls.

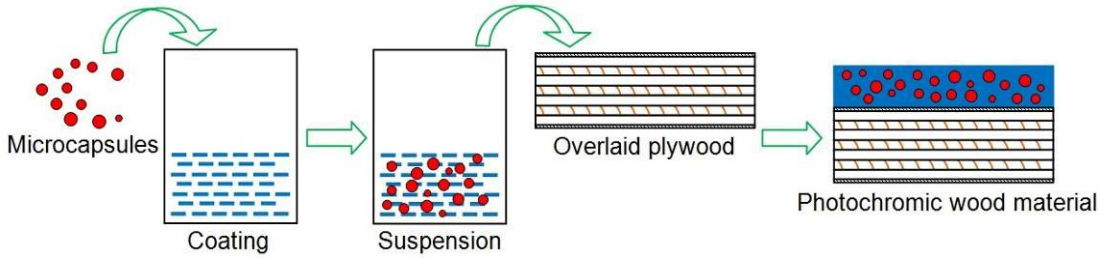

Fig. 1. Schematic diagram for preparing photochromic wood material

Characterization of Microcapsules

Optical micrographs of the microcapsules dispersed in water were obtained with an Olympus CX 31 optical microscope (Olympus (China) Co., Beijing, China). The diameters of the microcapsules were obtained with SPOT metrical software (Guangzhou Ming-Mei Technology Co., Guangdong, China), and over 200 samples were measured. The histogram and normal distribution curve of the particle sizes were then plotted by Origin 8.5 software (OriginLab Corp., USA). Microcapsule morphologies were observed on a field emission scanning electron microscope (model S-4800, Hitachi, Japan) at an operation accelerating voltage of 10 kV. The particles were sprinkled onto double-sided tape and sputter-coated with a gold layer.

Fourier transform infrared (FTIR) spectra were recorded using a VERTEX 70 V spectrophotometer (Bruker, Germany). Samples were mixed with KBr powders and pressed into a transparent tablet. The wavelength ranged from 4000 cm^{-1} to 400 cm^{-1}. Thermogravimetry (TG) tests were conducted under air flow on a DTG-60 apparatus (Shimadzu, Japan) from 40 °C to 550 °C at a heating rate of 10 °C/min. TG curves were

directly obtained from the tests, while the corresponding DTG curves were plotted by Origin 8.5 basing on differential methods.

Evaluation of Coating Performance

The distribution of microcapsules was observed on glass slides with photochromic coatings on an optical microscope. The thickness of the sample panels before and after finishing were measured using a 0.01-mm micrometer, and the difference was calculated as the coating thickness.

The wearability of the coating was evaluated with a MGL-5A abrasion tester (Jinan Times Assay Instrument Co., Shandong, China) in accordance with ISO 7784-2 (1997). The sample size was 100 mm by 100 mm. The sample was set on a revolving disc and pressed by a pair of abrasive rubber wheels. After 50 r of the disc at 60 rpm, the loss of coating (g) was estimated to evaluate the wearability of the sample.

Coating adhesion was measured by means of a YC1052 coating cross-cut test device (TST Instruments, Fujian, China), according to ISO 2409 (2013). The surface of the sample was processed by a multiple-blade cutting tool to form a 5 × 5 scratch-matrix with 2 mm by 2 mm squares as basic units. The scratches were deep enough to penetrate to the substrate. A soft brush was then used to clean the loose paint. The cut area was carefully observed with a magnifying glass, and the adhesion of the sample was classified into five grades according to the degree of coating detachment.

Measurement of Photochromic Properties

The surface colorimetric parameters of the samples, including L^* (lightness index), a^* (red-green index), and b^* (yellow-blue index), were obtained from a CR-400 chroma meter (Konica Minolta Sensing, Inc., Japan) after their exposure to indoor light or sunlight. Furthermore, the variation of these parameters at different times on a sunny day (September 7, 2015) was evaluated.

The colour difference (ΔE) of sample surfaces was quantified based on the CIELAB system (Dong *et al.* 2011). In this study, the L^*, a^*, and b^* values from samples exposed to indoor light (denoted as L^*_0, a^*_0, and b^*_0) were regarded as the reference values. The colour difference of a sample under sunlight exposure (denoted as $\Delta E'$) was calculated with the following formula,

$$\Delta E' = [(L^{*\prime} - L^*_0)^2 + (a^{*\prime} - a^*_0)^2 + (b^{*\prime} - b^*_0)^2]^{1/2} \quad (1)$$

where $L^{*\prime}$, $a^{*\prime}$, and $b^{*\prime}$ were values produced under sunlight exposure.

A simple application of the photochromic wood material was performed. A paper was cut with a star pattern and the phrase "Smart Wood". The paper was placed on a photochromic sample to block the sunlight. After a 1-min exposure, the panel was removed from sunlight, and it was imaged with a Cannon digital camera.

RESULTS AND DISCUSSION

Microcapsule Characteristics

The photochromic microcapsules were white powders when placed in indoor light, but when they were exposed to sunlight, their colour changed to blue (Fig. 2). The colour returned to white when they were transferred indoors. The colour-changing

phenomenon was evident and sensitive and could be ascribed to the reversible change of chemical structure in the spirooxazine compound (Partington and Towns 2014).

SEM images showed that most photochromic microcapsules were smooth and spherical (Fig. 3). Agglomeration was not observed. The particle size was normally distributed in a narrow fluctuation range of 1 μm to 6 μm (Fig. 3). The average microcapsule diameter was 3.28 μm, which was comparable to microcapsules of similar composition (Qiao *et al.* 2011; Zhou *et al.* 2013). The size of the photochromic microcapsules was noticeably smaller than the coating thickness (dozens of microns to over 100 μm) for wood materials. Therefore, it is feasible to add microcapsules to coatings to develop photochromic wood materials.

Fig. 2. Microcapsules after exposure to indoor light or sunlight

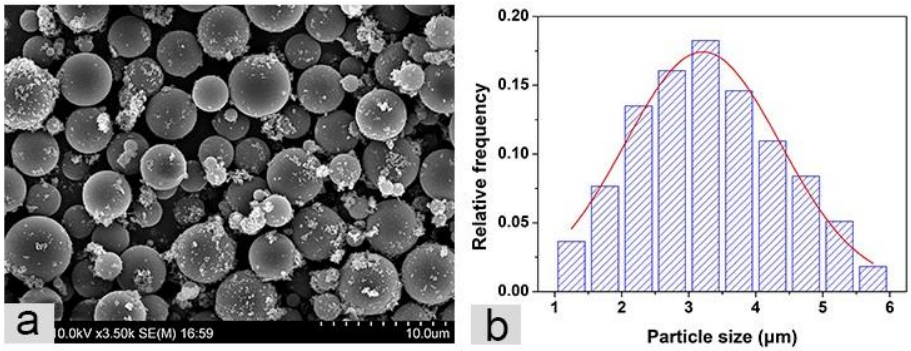

Fig. 3. SEM micrograph (a) and particle size distribution (b) of microcapsules

The FTIR analysis of microcapsules is presented in Fig. 4. The absorption peak at 3401 cm^{-1} was due to the N-H stretching vibration of the secondary amine, while the peaks at 1566 cm^{-1} and 814 cm^{-1} were assigned to the in-plane and out-of-plane vibrations of the triazine ring, respectively (Luo *et al.* 2007; Yu *et al.* 2009). This data indicated that MF resin was present. The characteristic peaks of spirooxazine appeared at 2924 cm^{-1}, 2855 cm^{-1}, 1493 cm^{-1}, 1461 cm^{-1}, 1166 cm^{-1}, and 744 cm^{-1} (Zhou *et al.* 2013). Absorption bands in 2924 cm^{-1} and 2855 cm^{-1} were the stretching vibration of C-H bonds in a saturated carbon chain, while peaks at 1493 cm^{-1} and 1461 cm^{-1} designated benzene skeleton vibration. Absorption at 1166 cm^{-1} corresponded to the stretching vibration of Ar-O, whereas the peak at 744 cm^{-1} was ascribed to the in-plane flexural vibration of C-H bonds in the benzene ring. In addition, the peak at 1343 cm^{-1} was due to the stretching vibration of C-N. In sum, FTIR analysis confirmed the chemical composition of the photochromic microcapsules.

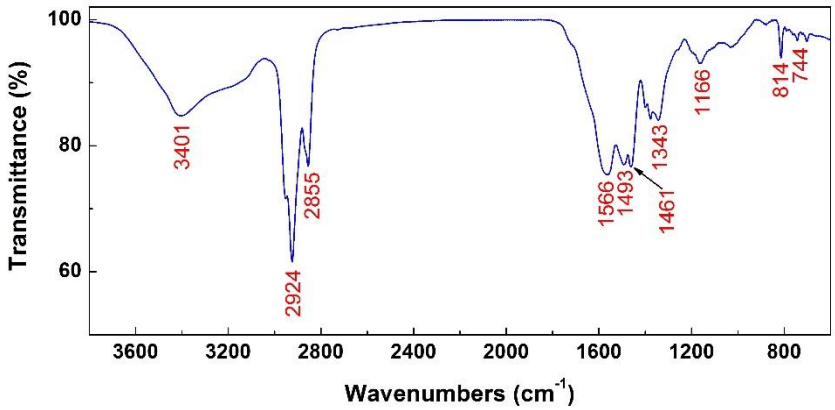

Fig. 4. FTIR spectrum of microcapsules

Photochromic microcapsules exhibited excellent thermal stability, with merely 9.98% mass loss up to 300 °C (Fig. 5). This loss may have represented the evaporation of residual water and some low molecular weight chemicals (such as formaldehyde) from the shell as well as some leakage of core materials (Wu *et al.* 2014). Most mass loss occurred from 320 °C to 360 °C, with a peak at 347 °C. This peak indicated thermal degradation of the MF resin shell and the evaporation of core materials. Over 360 °C, nearly 40% of the mass was lost, which corresponded to further pyrolysis of the residual shell and core materials.

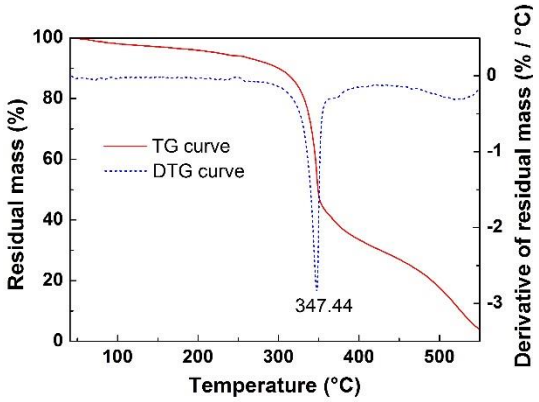

Fig. 5. Microcapsule TG and DTG curves

Coating Performance

The optical images of photochromic films with different amounts of microcapsules are presented in Fig. 6. In general, microcapsules were evenly dispersed, suggesting that they would form even coatings and promote sensitive colour-changing properties on wood materials. When the addition amount of microcapsules rose from 2.5% to 10%, the intensity of the microcapsules increased correspondingly. At 10%, the microcapsules aggregated.

The average coating thickness of the sample panels varied from 56.4 μm to 67.4 μm (Fig. 7). The thickness of the 7.5% and 10% groups was 9 μm to 11 μm greater than in the other groups, possibly due to the rise in solid content resulting from the incorporation of more microcapsules. Nevertheless, the small differences in thickness were not expected to affect the coating performance or photochromic properties.

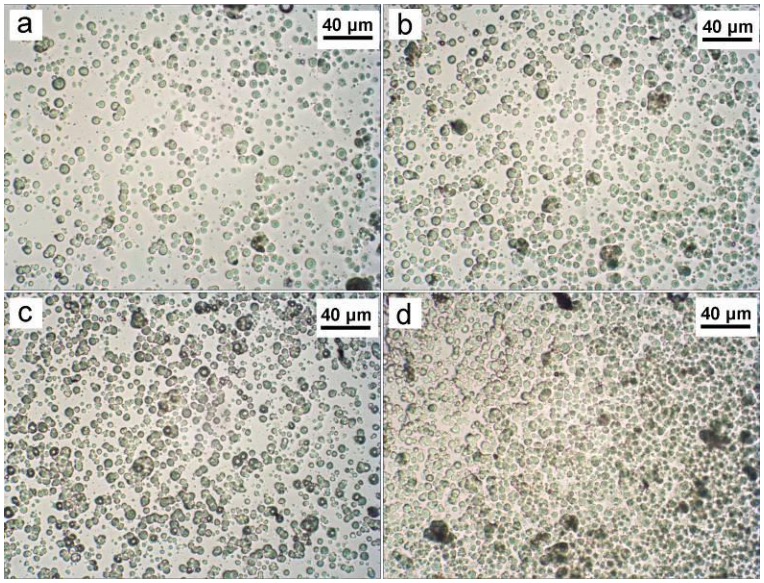

Fig. 6. Coatings with the addition of (a) 2.5%, (b) 5%, (c) 7.5%, and (d) 10% microcapsules

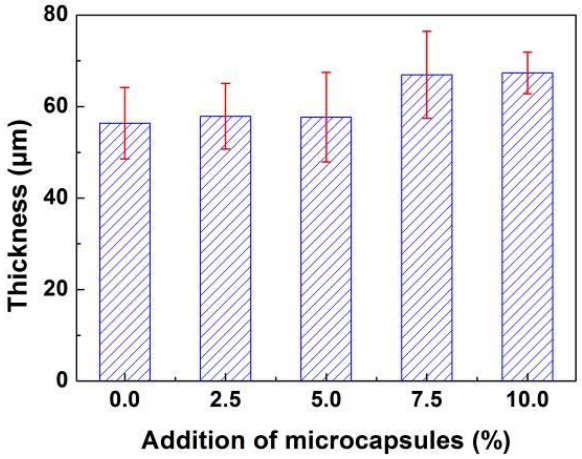

Fig. 7. Coating thickness with different amounts of microcapsules. Error bars indicate standard deviation.

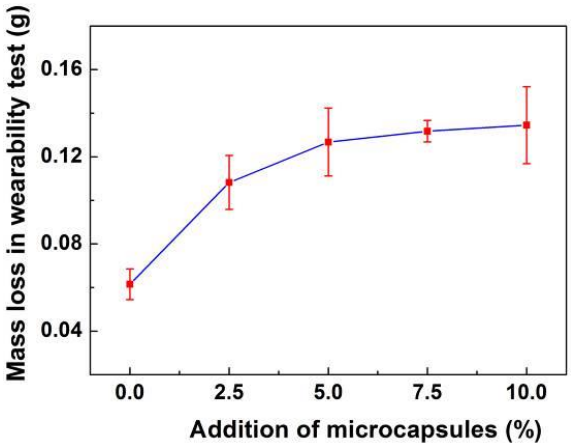

Fig. 8. Wearability test for coatings with different amounts of microcapsules. Error bars indicate standard deviation.

The wearability test examined mass loss from coatings with different amounts of microcapsules (Fig. 8). Mass loss increased with increasing microcapsule content. Mass loss for the 10% group (0.13 g) was more than 2-fold that of the control group; the microcapsules had poor binding forces within the coating material and were removed easily by abrasion. Therefore, it would be better to incorporate microcapsules into a primer or intermediate coat, rather than into a top coat.

Cross-cut tests of the various coatings showed smooth edges for all groups, but small flakes of the coating detached from the intersections of cuts (see red circles, Fig. 9). The affected areas were less than 5% of the total cross-cut area of each group. Hence, the control and the photochromic samples were classified as grade 1, which is the second highest adhesion grade regulated in ISO 2409. The addition of microcapsules had no obvious influence on coating adhesion, possibly due to the even distribution of microcapsules in coatings as well as the small size of microcapsules compared to the coating thickness.

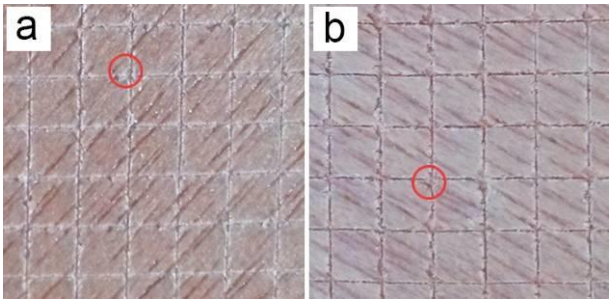

Fig. 9. Coating cross-cut test for (a) a control sample and (b) a photochromic sample with 10% microcapsules. Red circles mark representative detachment areas.

Photochromic Properties

The appearance of photochromic wood materials in different sunlight conditions is displayed in Fig. 10. When the samples were placed indoors without direct sunlight, clear veneer grain was observed for all groups. Under sunlight, the surface of samples with microcapsules turned blue, which was dramatically different from the control group. The colour change was reversible and sensitive, in accordance with the photochromic properties of microcapsules. The reversible colour changes in photochromic wood material were similar to those in photochromic textiles, glasses, and windows (Qiao *et al.* 2011; Eppig *et al.* 2012; Meng *et al.* 2013). As the addition of microcapsules increased from 2.5% to 10%, the intensity of the blue colour increased.

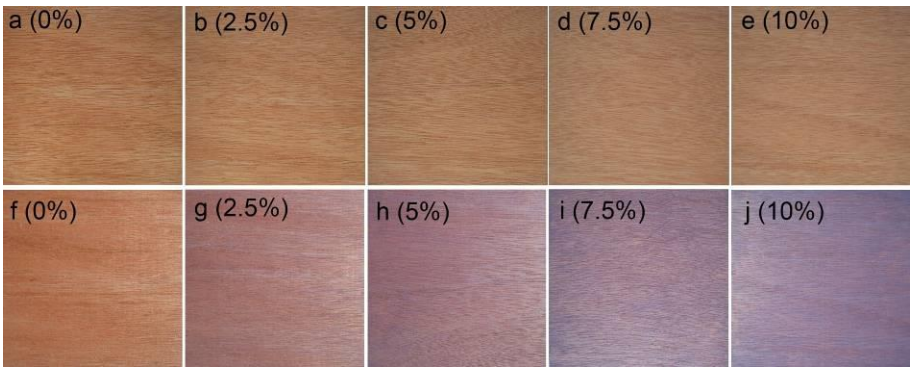

Fig. 10. Photochromic wood materials after exposure to indoor light (a to e) and sunlight (f to j). Microcapsule additions to coatings are listed in parentheses.

In addition to visual observations, the colour difference (ΔE) and colorimetric parameters for samples under sunlight exposure were calculated (Fig. 11). L^*, a^*, and b^* of the sample showed a similar decline trend following the increase of microcapsule content in coatings. Correspondingly, the colour difference increased from 7.45 to 21.58 when the addition of microcapsules rose from 2.5% to 10%, respectively. Moreover, both colorimetric parameters and ΔE showed a linear positive correlation with the microcapsule content, demonstrating that the shade of photochromic materials could be adjusted by changing the amount of microcapsules.

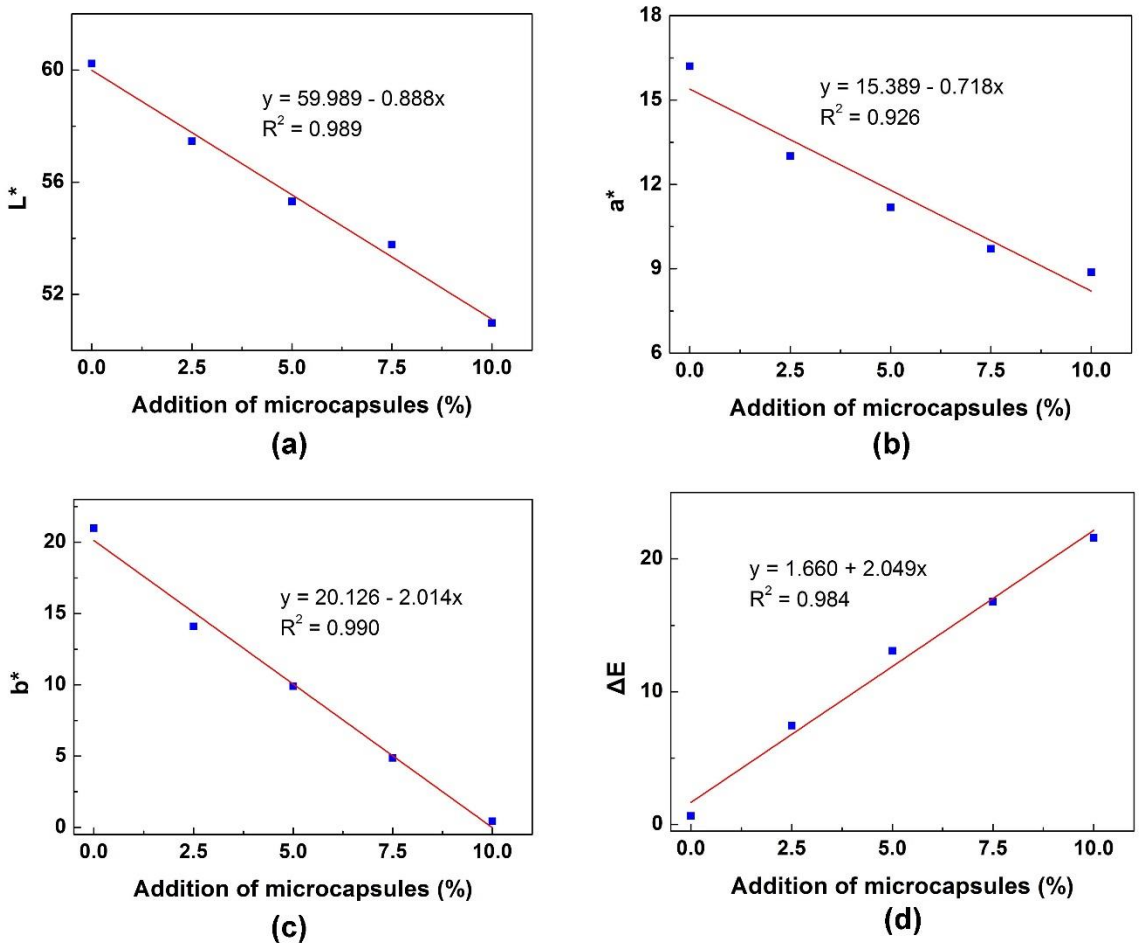

Fig. 11. Relationship between colour difference after sunlight exposure and amount of microcapsules

Variations in colorimetric parameters and colour difference were measured on a sunny day (September 7, 2015; Fig. 12). The colour of the control group remained stable because no colour change occurred. Photochromic samples showed maximum ΔE values varying from 14.56 to 31.42, and panels with more microcapsules presented notable colour differences. The trends in ΔE at different times of the day were similar for the four photochromic groups. From 08:00 to 15:00, colour differences were maintained at high values, due to the higher sunlight intensity. The sudden drop in ΔE at 12:00 resulted from the sunlight being partly blocked by white clouds. After 15:00, sunlight weakened, leading to dramatic declines in colour difference. At 18:00, the colour difference caused by the setting sun was less than 5. As for the variations of colorimetric parameters, a^* only presented a sharp increase in the time range of 17:00 to 18:00, while an obvious

increment of L^* and b^* began at 14:00 and was in accordance with the variations of ΔE. It indicates that L^* and b^* were more sensitive to sunlight intensity than a^*. Since the microcapsules were blue, a^* referring to red-green colour was less affected than b^* referring to yellow-blue colour. In sum, the photochromic wood materials altered their surface appearance according to changes in sunlight intensity.

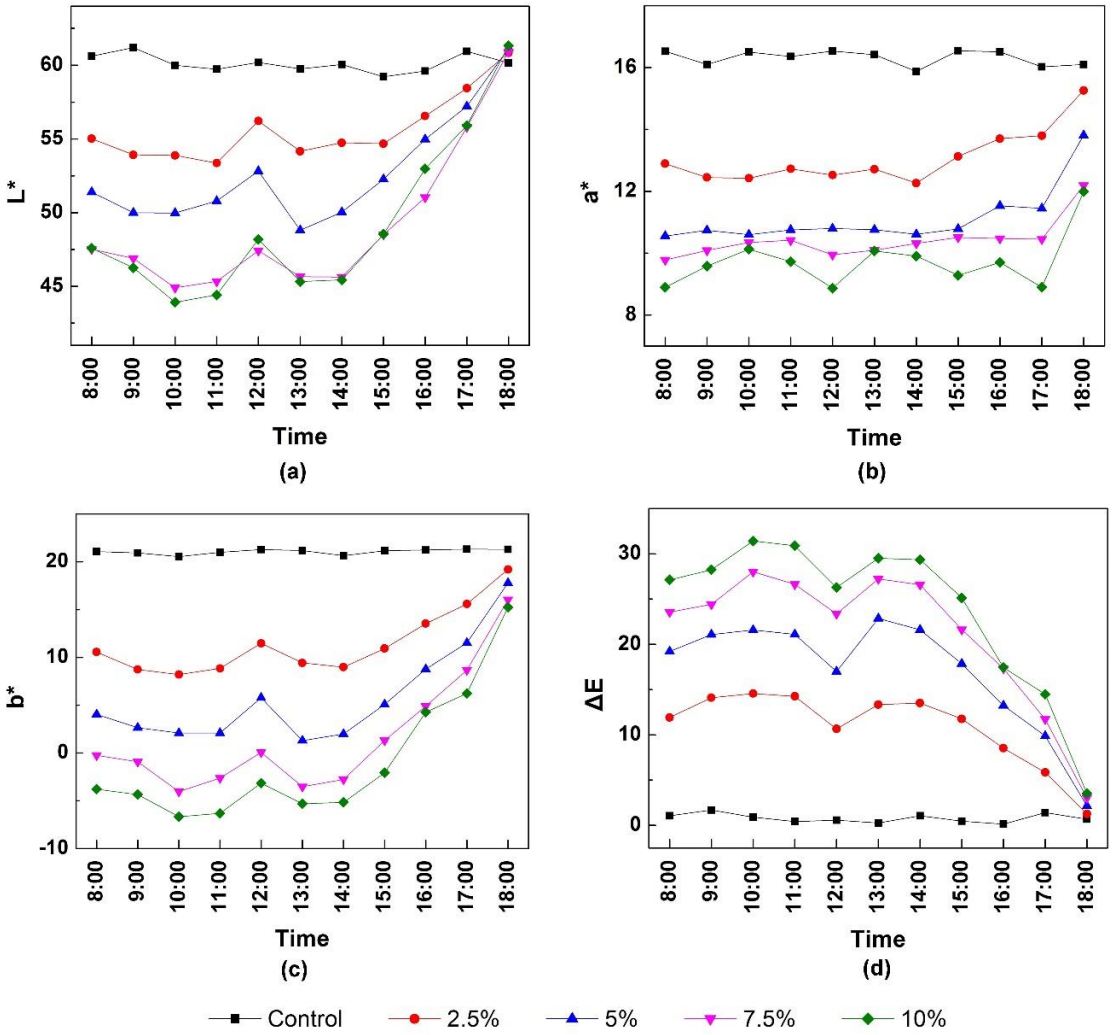

Fig. 12. Colour difference in photochromic wood materials with different additions of microcapsules on a sunny day

A simple application of this smart wood material is displayed in Fig. 13. Blue graphics were displayed on the surface of a sample panel for an "imaging effect". The unique reversible colour-changing properties of photochromic wood products could provide dynamic decorative effects similar to thermochromic wood materials (Liu *et al.* 2011), showing great potential in applications for furniture, decoration materials, and wood artworks. In addition, photochromic wood materials could be used as indicators of ultraviolet or sunlight, by using correlations between colour change and ultraviolet intensity (Brizio *et al.* 2015). Meanwhile, this smart wood also shows a promising application in ultraviolet protection of wood products, due to the ultraviolet absorption ability of photochromic materials. Above all, photochromic wood is a promising material for functional and intelligent wood products.

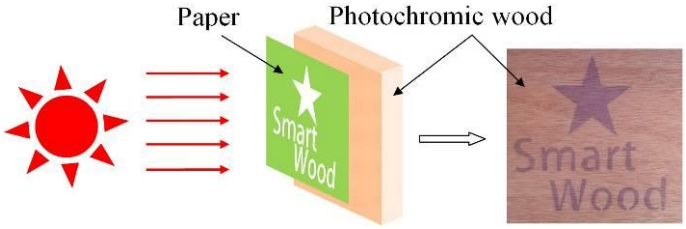

Fig. 13. Application of an image to photochromic wood material

CONCLUSIONS

1. Photochromic microcapsules were incorporated into the coating of veneered plywood to make a smart, light-responsive wood material.
2. Photochromic wood material altered its appearance from a veneer colour to a blue colour following exposure to sunlight. The potential applications of this smart wood material include furniture manufacturing, building decoration, and wooden crafts.
3. Microcapsules had little effect on adhesion of coating, but they reduced coating wearability.
4. When the addition of microcapsules was increased from 2.5% to 10% (of the coating weight), the colour difference in photochromic wood after sunlight exposure rose from 7.45 to 21.58. Linear correlations were found between colour difference and the amount of microcapsules, suggesting that the amount of microcapsules could be varied to adjust the performance of photochromic wood material.

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