Weatherability of ACQ-D Treated Wood Modified with Nano-SiO_2 and Emulsified Wax after One-year Outdoor Exposure

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Southern pine samples were vacuum-pressure impregnated with nano-SiO_2 with Nano-SiO_2 based on different specific surface areas (60, 150, 200, 380$m^2.g^{-1}$) and impregnated with alkaline copper quat type D (ACQ-D) preservatives or ACQ-D preservatives modified with emulsified wax. The treated wood was exposed outdoors for one year. The effects of emulsified wax and different conditions of nano-SiO_2 on the color change, wettability, and thermal stability of the treated wood were evaluated by colorimeter, contact angle tester, and thermogravimetry (TG). The results showed that nano-SiO_2 modification was helpful to keep the surface color stable for ACQ-D treated wood, while the effect of emulsified wax and the specific area of nano-SiO_2 on the color stability was slight. The static contact angles were increased after outdoor exposure with nano-SiO_2 and/or emulsified wax because of the reduction of the free hydroxyl groups, especially for nano-SiO_2 with smaller specific surface area. The thermal stability of ACQ-D treated wood could be increased to different degrees depending on the type of nano-SiO_2 and whether emulsified wax were present in the treated wood, although the emulsified wax only played an auxiliary effect on the thermal stability of the treated wood.

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INTRODUCTION

Wood treatment with organic or inorganic chemical preservatives has been shown to protect its structural integrity from the harmful effects of fungi, mold, termites, and other pests, improve the efficiency of wood utilization, and prolong the service life of wood-based products (Freeman and Mcintyre 2008; Terzi *et al*. 2016; Emmerich *et al*. 2021). In response to growing concern with environmental safety and human health, some traditional preservatives contained arsenic or chromium have been mainly withdrawn from the residential market (Lim *et al*. 2015; Nakiguli *et al*. 2020). ACQ has become one of the most widely used wood preservatives in the recent market because of its resistance to pests and fungi and because it is not harmful to mammals (Janin *et al*. 2021). However, due to the absence of the water repellent and weathering resistant ingredients in the ACQ formulas

(Wang *et al*. 2011), it is very common to find that ACQ-treated wood exposed outside for some time would exhibit discoloration, cracking, warping, and other issues of wood weathering (Evans *et al*. 2008; Liu 2019; Heshmat *et al*. 2020). When wood-based products are placed in outdoor conditions, they are affected by many complicated climate factors including rainfall, UV component of sunlight, seasonal changes in relative humidity (RH) and temperature, atmospheric pollution, oxygen, and human activities (Denes and Young 1999; Yalinkilic *et al*. 1999; Temiz *et al*. 2005). As a result, some wood components are degraded, which changes the chemical, physical, optical, and mechanical properties of wood (Lim *et al*. 2015; Nakiguli *et al*. 2020; Cen *et al*. 2023).

Many studies have demonstrated that discoloration of wood surfaces is due to the photo-degradation of wood components and colonization by melanin-rich fungi (Temiz *et al*. 2005; Nejad and Cooper 2011), and the checking and distortion of wood is due to surface stresses generated by moisture-induced anisotropic swelling and shrinkage of wood (Wang *et al*. 2011; Heshmat *et al*. 2020). Such effects are exacerbated by the surface photo-degradation of wood (Evans *et al*. 2008).

Nano SiO_2 can be considered to improve the resistances to UV radiation, scratch and abrasion, flammability, hygroscopicity, anti-microbial characteristics, and the mechanical properties of the treated wood when exposed outdoors (Filpo *et al*. 2013). Li *et al*. (2024) reported that nano SiO_2-IPBC (3-iodo-2-propynylbutylcarbamate) microcapsules had better bonding performance and UV-resistance than IPBC due to the synergistic effect of nano SiO_2 and IPBC in the microcapsules. Additionally, the water repellents such as wax or oil emulsion additives incorporated into aqueous wood preservatives have been shown to improve the weatherability of treated wood exposed outdoors.

Evans *et al*. (2009) had evaluated radiata pine samples treated with a chromated copper arsenate preservative containing oil (7% or 14% w/w) or wax emulsion additive (2.5%) after being exposed outdoors in Australia for one year, and found the wax and oil emulsion additives were equally effective at reducing both the checking of treated wood exposed to weathering and the water absorption and swelling of treated wood. Copper azole (CA) treated southern pine (*Pinus* spp.) samples modified with paraffin wax emulsion showed better water repellency and improved dimensional stability compared with untreated wood (Wang *et al*. 2018). Bansal *et al*. (2024) reported that rubber wood specimens impregnated with linseed oil and paraffin wax and modified with nanoemulsions containing zinc oxide (ZnO) and copper oxide (CuO) nanoparticles exhibited good resistance to fungal decay. Liu *et al*. (2020) found that the paraffin Pickering emulsion stabilized by the nano-SiO_2 (diameter of ~76 nm) could penetrate the wood structure and enable the treated wood with a moderate anti-swelling efficiency (ASE), high water resistance, and low wettability, and moreover, the addition of nano-SiO_2 could improve the thermal stability of the treated wood.

In this study, southern pine samples were impregnated with emulsified wax and different types of nano-SiO_2 (specific surface area: 60, 150, 200, 380 $m^2.g^{-1}$) and exposed to outdoor conditions, and the weatherability of ACQ treated wood including the changes of deformation, color, wettability, thermal stability, and chemical groups were evaluated and analyzed after one-year outdoor exposure. The aim of this research was to determine the ACQ-D treated modification conditions for outdoor application and provide a theoretical basis to prolong the service life and reduce the maintenance cost of ACQ-D treated wood.

EXPERIMENTAL

Samples Impregnated with Nano-SiO_2 Solution

The sapwood of kiln-dried southern pine (*Pinus* sp.) without any defect was cut into small cubes with dimensions of 100 × 60 × 10 mm (length × width × height) and stored in a conditioning room (20 ± 2 °C, 65 ± 5% R.H.) to reach an equilibrium moisture content of 9 to 10%. The test samples were selected with similar weight, and 6 replicates were used in each set.

Hydrophilic nano-SiO_2 with four different specific surface areas of 60, 150, 200, or 380 $m^2.g^{-1}$ (the corresponding particle sizes about 385, 154, 115, and 61 nm, respectively) produced by Zhejiang Hongsheng Material Technology Co., Ltd., assisted by the corresponding proportions of the trisodium phosphate (analytically pure) produced by Tianjin Kemi Chemical Reagent Co., Ltd. according to Table 1 were dispersed in the deionized water, respectively, and then the mixture solution was stirred using a magnetic stirrer to disperse completely. The mass fraction of nano-SiO_2 with different specific surface areas was defined as maximum dispersity in Table 1. Wood samples were vacuum-pressure impregnated with different nano-SiO_2 water solutions respectively and then oven dried at 60 °C to achieve constant weight.

For the vacuum-pressure treatment, the samples were placed into the vacuum-pressure chamber and the vacuum was set to -0.1 MPa for 1 h to remove the air from the chamber and the wood samples, then the solution was introduced and filled into the chamber completely, and under these vacuum conditions the samples could be penetrated completely because of the excellent permeability of the sapwood of kiln-dried southern pine. After that, the vacuum was released, and pressure was elevated to 0.8 MPa for another 1 h to ensure the solution became evenly distributed in the samples. Finally, the pressure was released and the samples were removed from the pressure chamber.

ACQ-D treated Wood with Different Modifications

The ACQ-D concentrate (66.7% CuO, 33.3% didecyl dimethyl ammonium chloride (DDAC)) used in this study had a concentration of 15%, which was diluted with deionized water to 0.6% and mixed with 2.5% paraffin wax emulsion (melting point: 58 to 60 °C, mass fraction: 40%). Ten sets of samples were vacuum-pressure impregnated with different ACQ solutions as listed in Table 1. The vacuum-pressure treatment process was the same as those in the samples impregnated with nano-SiO_2 solution because of the stable solutions at room temperature. Then T1, T2, T3 and T4 were vacuum-pressure impregnated with different types of nano-SiO_2 solutions and then vacuum-impregnated with 0.6% ACQ-D preservatives. T5, T6, T7 and T8 were vacuum-pressure impregnated with different types of nano-SiO_2 solutions and then vacuum-pressure impregnated with 0.6% ACQ-D preservatives modified with 2.5% emulsified wax. T9 was only vacuum-pressure impregnated with 0.6% ACQ-D preservative. T10 was vacuum-pressure impregnated with 0.6% ACQ-D preservative modified with 2.5% emulsified wax. After ACQ-D preservatives and ACQ-D preservatives modified with 2.5% emulsified wax impregnation, the samples were post-treated at 70 °C, 80% R.H. for 10 h with hot air circulation to achieve better copper fixation and more uniform distribution of emulsified wax, followed by oven drying at 60 °C to achieve constant weight.

Table 1. Treatment Conditions for ACQ-D Treated Wood with Different Modifications

Test Group	Nano-SiO_2		ACQ-D (%)	Emulsified Wax (%)
	Specific surface area ($m^2.g^{-1}$)	Maximum dispersity (g/100g)		
T1	60	0.6	0.6	0
T2	150	0.8		
T3	200	1.3		
T4	380	1.9		
T5	60	0.6		2.5
T6	150	0.8		
T7	200	1.3		
T8	380	1.9		
T9	0	0		0
T10	0	0		2.5

Natural Weathering

The tangential section of ACQ-D treated samples without and with different modifications as listed in Table 1 were horizontally exposed outside on the roof of the Wood Value Promotion and Sustainable Development Center, Beijing, China for 12 months, and the ends of the samples were changed once a month. The average annual temperature of Beijing is about 12.6 ℃, and the average precipitation is about 640 mm. The annual average relative humidity in Beijing is approximately 50% to 60%, and the corresponding equilibrium moisture content of the test samples is roughly around 10% to 12%. After one-year outdoor exposure test, the treated samples were dried at 60 °C to achieve constant weight and the weatherability of the treated wood including the dimensional and thermal stabilities, the surface color and the wettability during outdoor exposure was evaluated.

Color Measurement

The color indexes of samples before and after weathering were measured by the Color Difference Meter NH310 (Shenzhen Guangdong, China). Three specific places of each sample before and after weathering were selected for testing of the average color change of the samples during outdoor exposure test. Colour change evaluations were done according to the CIE-L*a*b* colour system on the basis of *L**, *a**, and *b** colour coordinates (ISO 2022), where: *L** is lightness from 0 (black) to 100 (white), *a** is chromaticity coordinate + (red) or − (green), and *b** is chromaticity coordinate + (yellow) or − (blue). Relative colour changes of samples ΔL^*, Δa^* and Δb^* between their weathered and initial state were determined, and finally, according to the Euclidean distances the total colour differences ΔE^* were calculated by Eq. 1,

$$\Delta E^* = \sqrt{\left(\Delta L^*\right)^2 + \left(\Delta a^*\right)^2 + \left(\Delta b^*\right)^2} \qquad (1)$$

where ΔL^*, Δa^*, and Δb^* are the differences in individual axes (difference between the values measured before and after weathering exposure of the samples). Untreated samples at 20 °C were chosen as references for each treatment. The measured color values were evaluated in Statistica 10 software by a two-factor analysis, and the analysis factors were the wood species and the temperature.

Contact Angle Test

The samples with different treatments after one-year exposure were measured on a JC2000D3R Contact Angle Analyzer (Shanghai Zhongchen Powereach Company, Shanghai, China) at ambient temperature using 5 μL of DI water. The contact angle results were recorded based on the average of measurements taken from five different positions after five seconds following droplet application.

Thermogravimetric Analysis (TG)

The thermogravimetric analysis of the samples with different treatments after one-year exposure was carried out by using the Q500 thermogravimetric analyzer produced by TA Instruments (Shanghai, China). In order to eliminate the effect of the thickness on the ACQ-D components in the samples, each sample was ground to pass through a 100-mesh sieve and then oven dried. Approximately 10 mg of wood powder was placed into the thermogravimetric analyzer and heated at a rate of 20 ℃/min in nitrogen atmosphere. The tested temperature range was 25 to 700 ℃ to explore the classification temperature and the corresponding mass loss.

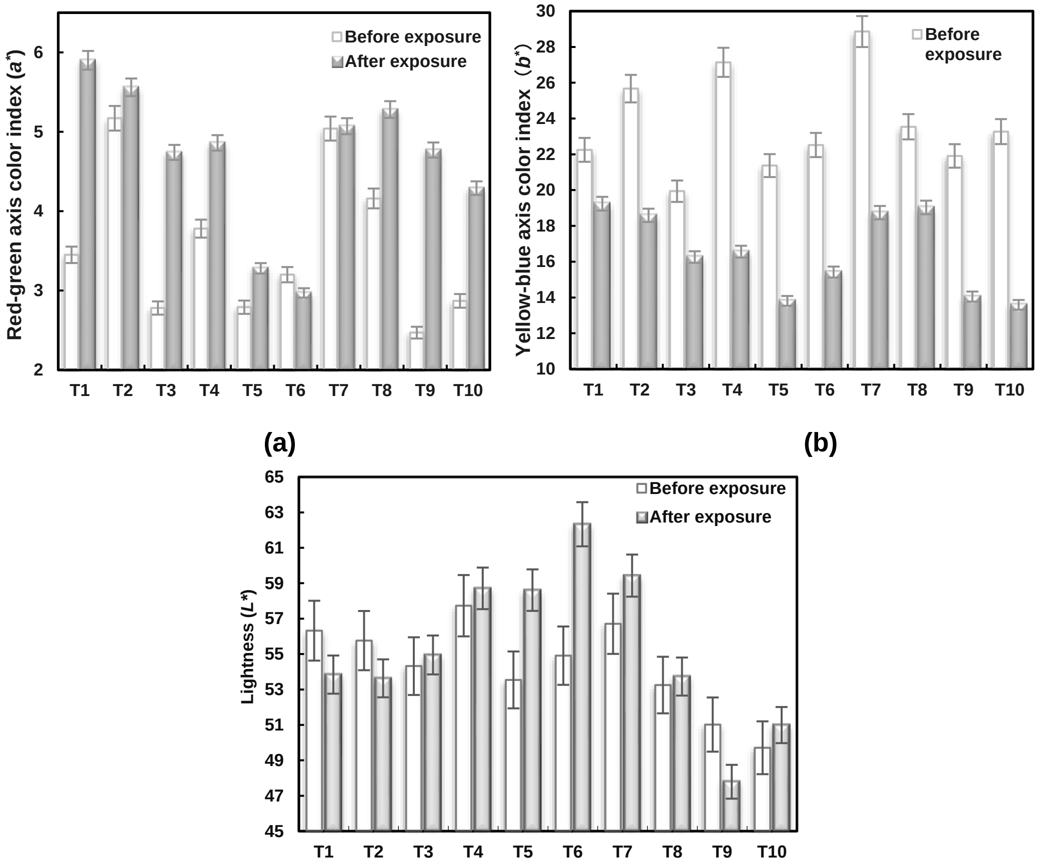
RESULTS AND DISCUSSION

Color Change of ACQ-D Treated Wood Surface

The appearance quality of ACQ-D treated wood with different modifications for one year outdoor exposure and without outdoor exposure are shown in Fig. 1, and the corresponding color indexes changes were evaluated in Fig. 2 and Table 1.

Fig. 1. Appearance quality of ACQ-D with different treatments without outdoor exposure (C) and after one year outdoor exposure(T1-T10)

Compared to the treated wood before outdoor exposure, except for T6, the surfaces of ACQ-D treated wood with different modifications had significant darker reddish and lighter yellow color (Fig. 2a, 2b). These changes were attributed to natural weathering factors, such as UV component of sunlight, seasonal changes in relative humidity (RH) and temperature, atmospheric pollution, oxygen (Kim *et al*. 2008). As a result, the lignin and carbohydrates had been degraded and formed the colored unsaturated carbonyl compounds (quinones) since photochemical reactions of lignin occur (Temiz *et al*. 2005). ACQ-D treated wood with nano-SiO_2 and emulsified wax presented less change of a^* value, especially for T7, for which the a^* value only increased by 0.60% after outdoor exposure, while the b^* values in all the treated wood decreased significantly. As observed from Fig. 2c, the surface lightness of ACQ-D treated wood became a little darker after outdoor exposure because of the surface components degradation (Temiz *et al*. 2005), while except for T1 and T2, L^* values of the other modified treated wood had a little growth. The change regularity of L^* values was the result of the introduction of emulsified wax into ACQ-D treated wood, which was favoured for keeping the surface lightness unimpaired, especially for the treated wood with smaller specific surface areas during outdoor exposure.



(a) (b)

(c)

Fig. 2. Red-green color indexes (*a**) (a), yellow-blue color index (*b**) (b), lightness (*L**) (c) of ACQ-D treated wood with different modifications before and after one year outdoor exposure.
Note: the test group numbers of the experimental conditions can be found in Table 1.

From Table 2, the surface color of ACQ-D treated wood without any modification (T9) as well as T1 and T2 were darker than those before outdoor exposure, as indicated by the negative ΔL^* values, while other treated wood modified with emulsified wax and/or nano-SiO_2 presented the positive ΔL^* values. The results demonstrated that although the modification with emulsified wax or nano-SiO_2 with higher specific surface areas were beneficial for improving the lightness of ACQ-D treated wood, the modifications with the combination modification of emulsified wax and nano-SiO_2 with lower specific surface areas seemed to bring brighter surfaces for the treated wood. The other reason was that the samples in the groups of T1, T2, and T9 were prone to forming black spots on the surfaces when exposed to the high humidity and heavy rainfall. After one-year outdoor exposure, except for T6, the surface color of all the treated wood became much more reddish and blueish (Δa^*, Δb^*). This was partially due to the lignin degradation (Kim *et al*. 2008). Another reason for a blue shift after long-time outdoor weathering may be the growth of blue-stain fungus. The largest change in Δa^* and Δb^* values were observed in T9, and nano-SiO_2 with different specific surfaces played different roles in the changes of Δa^* and

Δ*b** values, but no obvious regularity had been found between the modification conditions and Δ*a** and Δ*b** values. Based on the Δ*E* values obtained from Table 2, the Δ*E* of ACQ-D treated wood after outdoor exposure was the largest, which was similar with those only modified with emulsified wax, and the Δ*E* values of the treated wood modified with nano-SiO_2 (4.22 to 10.67) were a little lower than those modified with the combination of emulsified wax and nano-SiO_2 (4.67 to 10.48). The Δ*E* values of ACQ-D treated wood before and after outdoor exposure proved that nano-SiO_2 had excellent photodegradation resistant ability during long-term outdoor exposure. The larger specific surface area of nano-SiO_2 seemed a positive factor for maintaining the surface color stability of ACQ-D treated wood during outdoor exposure; for example, the similar lower Δ*E* values for ACQ-D treated wood were observed in the samples in T3 and T8. The result may be due to the fact that with a larger specific surface area of nano-SiO_2, more surface atoms were exposed to the outside(Yang 2019). It was speculated that the higher surface area of the SiO_2 particles may allow them to adsorb free-radical species generated by the UV light exposure. The large surface area may help to allow the free radicals to gradually return to their ground state without as much harm to the wood polymers, especially the lignin.

Table 2. Color Difference of ACQ-D Treated Wood with Different Modifications after One Year Outdoor Exposure

Test group	Δ*L**	Δ*a**	Δ*b**	Δ*E**
T1	-2.48	2.45	-3.01	4.61
T2	-2.13	0.39	-7.08	7.40
T3	0.63	1.96	-3.68	4.22
T4	0.98	0.96	-10.58	10.67
T5	5.07	2.49	-7.56	9.44
T6	7.42	-0.23	-7.10	10.27
T7	2.72	0.03	-10.12	10.48
T8	0.48	1.12	-4.51	4.67
T9	-3.23	2.30	-12.21	12.84
T10	1.28	1.42	-12.67	12.81

Wettability of ACQ-D Treated Wood

The surface wettability of ACQ-D treated wood with different modifications before and after one year outdoor exposure were compared and analyzed based on the static contact angles. Results are shown in Fig. 3. Compared with T9, the static contact angles of the treated wood modified only with nano-SiO_2 were decreased to below 90° because of the hydrophilic properties of nano-SiO_2, while the static contact angles of ACQ-D wood was increased with the introduction of emulsified wax into the treated wood. The specific surface area of nano-SiO_2 was also an important factor for the surface wettability of ACQ-D treated wood with different modifications, and the larger static contact angles were obtained in the treated wood modified with smaller specific surface area of nano-SiO_2. After one-year outdoor exposure, except for the similar values in T10 before and after outdoor exposure, the static contact angles in other ACQ-D treated wood increased. The effect was attributed to the reduction of the free hydroxyl groups decomposed by the reactions between climate factors and ACQ-D components with different modifications. Almost all of the static contact angles obtained from the treated wood were more than 90°, which meant that the surface wettability of ACQ-D treated wood was decreased due to ACQ-D solid particles deposited on the cell wall. These particles gradually infiltrate into the secondary wall microfibrils (Chouc *et al*. 1973); the micrometer sized solid deposits and covers the cell cavity (Maldas and Kamdem 2012), which increased the porosity and roughness of the treated wood surface. When copper ions in copper-based preservatives interact with wood, the surface roughness of the treated wood increases (Temiz *et al*. 2005). Zhang *et al*. (2023) pointed out that the wettability would decrease as the surface roughness of the preservative treated wood increases. Overall, all the surface static contact angles of ACQ-D treated wood modified with the combination of emulsified wax and nano-SiO_2 had been increased more significantly after one-year outdoor exposure and presented the lower surface wettability.

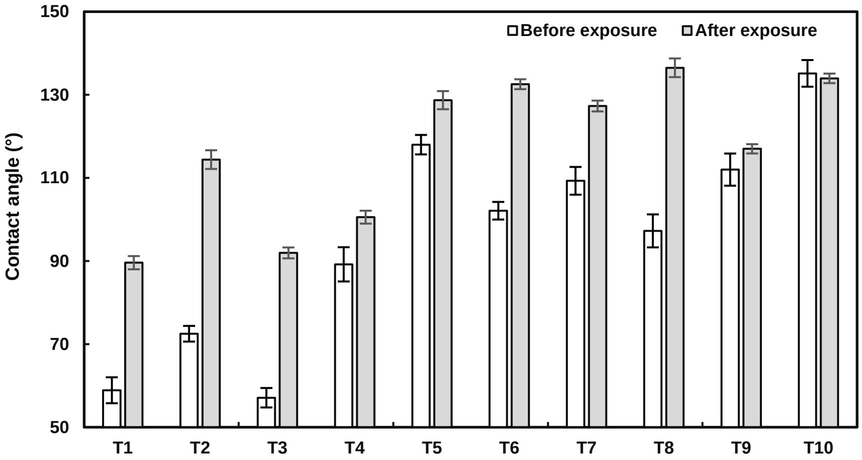
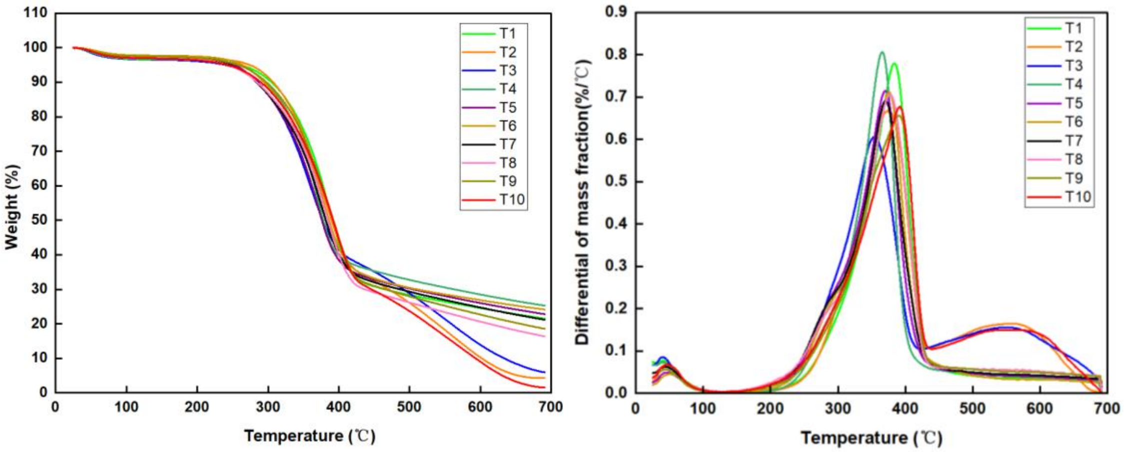


Fig. 3. Static contact angles of ACQ-D treated wood with different modifications before and after outdoor exposure

Thermal Stability of ACQ-D Treated Wood

The TG and DTG profiles of ACQ-D treated wood with different modifications after one year outdoor exposure are shown in Fig. 4. From the TG profiles, the highest weight loss was observed in T10, in which the value was up to 98.5%. The losses of other groups were less than 80%, demonstrating that the introduction of nano-SiO_2 or/and emulsified wax solution could improve the thermal stability of ACQ-D treated wood when exposed outdoors because of the better thermal stability of nano-SiO_2 and wax (Evans *et al*. 2009; Li *et al*. 2024). The weight losses were also more than 90% in T2 and T3. When the emulsified wax was introduced into the treated wood modified with the same specific surface area of nano-SiO_2 (T6 and T7), the weight losses could be reduced to 75.8% and 78.8%. These values were also lower than ACQ-D only modified with emulsified wax solution (T10), demonstrating the synergistic effect of nano-SiO_2 and wax on the thermal stability of the treated wood. However, the lowest weight loss was observed in T4, which was lower than those in T8, demonstrating that the synergistic effect of nano-SiO_2 and wax on the thermal stability of the treated wood was also affected by the type of nano-SiO_2. According to the DTG profiles, the modification conditions changed the maximum pyrolysis temperature and the shape of the pyrolysis peaks of ACQ-D treated wood. Three weight loss rate peaks were observed during pyrolysis. The first peak at 30 to 40 °C was attributed to the pyrolysis of monoethanolamine in ACQ-D formulas (Shi *et al*. 2018; Zhang *et al*. 2023). The second peak appearing in the range 200 to 400 °C was attributed to the pyrolysis of DDAC and most of the wood components (Sun 2008). The third peak at 400 to 700 ℃ was attributed to the pyrolysis of lignin and remaining hemicellulose gradually consumed under continuous high temperatures, and releasing more heat (Yang *et al*. 2015); the third peak was not obvious in the groups except for T2, T3, and T10. Thus, nano-SiO_2 and emulsified wax modifications resulted in the increase of weight loss at low temperatures and the decrease of weight loss at high temperatures. The reason may be that nano-SiO_2 could absorb heat at high temperatures and thereby form a dense protective layer isolating oxygen and heat from the treated wood and thereby inhibiting the progress of burning during the pyrolysis process (Shi *et al*. 2018). Although the emulsified wax itself does not possess flame retardant, the emulsified wax could play a certain auxiliary role to enhance the thermal stability of ACQ-D treated wood.



(a) (b)

Fig. 4. (a) TG profiles (b) DTG profiles of ACQ-D treated wood with different modifications after outdoor exposure

CONCLUSIONS

This study dealt with the changes of the color and wettability of ACQ-D treated wood modified with and without different nano-SiO_2 and/or emulsified wax modifications after one-year outdoor exposure.

1. Compared to the samples without any modification (T9), almost all of the surfaces of ACQ-D treated wood with different modifications had reddish, yellow and brighter color after one-year outdoor exposure. This was attributed to an improvement of photodegradation of lignin and carbohydrate. The color stability of ACQ-D treated wood was mainly dependent on the addition of nano-SiO_2, which could provide excellent anti-photodegradation ability for ACQ-D treated wood during outdoor exposure, while the effect of emulsified wax and the specific area of nano-SiO_2 on the color stability was slight.
2. The static contact angles in ACQ-D treated wood modified with nano-SiO_2 and/or emulsified wax would be increased more significantly after one-year exposure because of the reduction of the free hydroxyl groups. It is possible that -OH groups were lost due to reactions between climate factors and ACQ-D components with different modifications. The larger static contact angles were obtained in the treated wood

modified with nano-SiO_2 of smaller specific surface area, while the treated wood modified with the combination of wax and high-surface area SiO_2 was more able to achieve a long-lasting super-hydrophobic effect of some sort that helps to keep the treated wood dry during outdoor exposure.

3. Nano-SiO_2 with suitable specific area (60, 380 $m^2.g^{-1}$) or the combination of nano-SiO_2 with smaller specific area and emulsified wax would enhance the thermal stability of ACQ-D treated wood significantly, in which the emulsified wax mainly played the auxiliary effect on the thermal stability of the treated wood.

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