Study on UV-aging performance of fluorinated polymer coating and application on painted muds

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Abstract

The UV-aging tests were carried out on synthetic fluorinated polymer coating (poly (methyl methacrylate)-poly (dodecafluoroheptyl methacrylate), FP) to study its UV-aging mechanism, UV-aging resistance and application on painted muds with calcium carbonate, azurite, haematitum, mineral green and cinnabar pigments. The UV-aging mechanism was studied by Fourier infrared spectroscopy attenuated total reflection technique (ATR-FTIR) and x-ray photoelectron spectroscopy (XPS). The UV-aging resistance of FP was characterized by field emission scanning electron microscope (SEM), CR-400 color difference meter and static contact angle (SCA). The FP was applied on the painted muds samples and the protective efficacy was evaluated by measuring the chromatic variations (ΔE) and water contact angles of samples after treatment. The results of ATR–FTIR and XPS showed that FP only had microphase separation without chemical change after 360h of aging. After 360h of aging, the SEM shown that there were just some bumps on the FP film surface and further proved the phenomenon of microphase separation; the color change of the FP film was no difference (ΔE = 0.46); the FP film could retain adequate hydrophobicity (101.0°). After the painted muds samples were treated by FP, the color change of the calcium carbonate and haematitum was just noticeable difference (ΔE = 0.80 ~ 1.49), while the color change of the azurite, mineral green and cinnabar was fairly large difference (ΔE = 4.4 ~ 6.3); all pigments could maintain hydrophobicity at the end of aging. All the results demonstrated that the FP has excellent UV-aging resistance and could provide adequate protective efficacy for calcium carbonate and haematitum, but the protection efficacy for the azurite, mineral green and cinnabar is not ideal. Thus, it is believed that the FP can be used as a suitable UV resistant protective coating for calcium carbonate and haematitum.

1. Introduction

With the rapid development of industry, environmental pollution has become a serious problem facing the world. The influence of harmful gases, dust, microbial [1] and light on painted cultural relics [2, 3] on painted cultural relics cannot be ignored. Therefore, the protection of painted cultural relics is particularly important. An effective way to reduce the adverse effects of external environmental factors on cultural relics is to use polymer protective coatings [4–8] to protect the cultural relics, thereby improving the surface and internal performance of the cultural relics. The polymer protective coatings could protect the painted cultural relics by surface protection treatment [3, 9–11].

The fluorinated polymer coatings have attracted much interest of scientists operating in the field of hydrophobic coating [12, 13], anti-UV [14], antifouling [15], anticorrosion [16] and other aspects with low surface energy [17, 18], hydrophobicity [17, 19], thermal stability [20] and chemical stability [12]. Over the past years, fluorinated polymer coatings have played an important role in protecting ancient cultural relics [21–24]. For instance, perfluoropolymethers [25] and their derivatives [26] were used for the protection of natural and
artificial stones since the early 1980s. Since then, more fluorinated polymer materials such as fluorinated acrylic copolymers \cite{27, 28} and polyurethanes \cite{29} have been studied as coating materials in the protection of the ancient cultural relics.

At the same time, the used protective coatings should not change the color of the cultural relics surface and have good durability. However, under the influence of factors such as light, temperature and humidity, these protective coating materials will more or less undergo aging deterioration. After the failure of protection materials, they will affect the painted cultural relics, such as yellowing \cite{30}, chalking, micropores, and degradation of adhesion strength, etc. Therefore, it is necessary to study the stability, durability and protective efficacy of painted cultural relics protection coating materials.

At present, there are many reports on fluorinated polymer coating materials used for protection of stone cultural relics \cite{31–33}, but there are few studies on fluorinated polymer coating materials used for the protection of painted cultural relics. In this paper, we conducted the UV-aging tests on synthetic fluorinated polymer coating (poly(methyl methacrylate)-poly(dodecafluoroheptyl methacrylate), FP) and studied its UV-aging mechanism, UV-aging resistance and protective efficacy on painted muds with calcium carbonate, azurite, haematitum, mineral green and cinnabar pigments. The UV-aging mechanism of FP was studied by Fourier infrared spectroscopy attenuated total reflection technique (ATR-FTIR) and x-ray photoelectron spectroscopy (XPS). The UV-aging resistance was characterized by field emission scanning electron microscope (SEM), CR-400 color difference meter and static contact angle (SCA). The FP coating was applied on the painted muds samples and the protective efficacy was evaluated by measuring the chromatic variations ($\Delta E$) and water contact angles of samples after treatment.

2. Materials and methods

2.1. Materials

The materials were shown as follows. The fluorinated polymer coating (poly(methyl methacrylate)-poly(dodecafluoroheptyl methacrylate), FP) was synthesized by the polymerization of methylmethacrylate (MMA) and dodecafluoroheptyl methacrylate (12FMA) with the conventional initiator of ethyl 2-bromoisobutyrate (EBiB) by atom transfer radical polymerization (ATRP). The detailed preparation method was shown in our previous work \cite{34} and its chemical structure and molecular weight were shown in figure 1. The FP coating has excellent hydrophobicity, low surface energy and thermal stability.

2.2. Methods and characterization

2.2.1. UV-aging tests and characterization

The FP films were prepared by dropping the 5 wt% CHCl$_3$ solution on aluminum foil (30 mm × 30 mm × 0.2 mm) and dried at ambient (50%–55% RH) for 48 h and then in vacuum oven (50 °C) for 24 h with a thickness of about 800 $\mu$m. The accelerated UV-aging tests were carried out based on the GBT14522–2008 standard in an accelerated weathering chamber (Q8, Hongzhan Instrument Co. LTD, Dongguan, China) to investigate the UV-aging mechanism and resistance of coating. The samples were exposed to Ultra-violet radiation lamp for 15d \cite{35} at 25 ± 1°C and the relative humidity was set at 50 ± 3% RH. UV lamps with wavelength of 313 nm were selected for irradiation with 0.89 W m$^{-2}$ intensity of irradiation.

Films were measured at 0 h and 360 h of aging to evaluate the UV-aging mechanism by ATR-FTIR and x-ray photoelectron spectroscopy (XPS, AXIS ULTRA, KRATOSANALYTICAL Ltd, Manchester, England,) with an Al-Ka monochromatic x-ray source (1486.6 eV) operated at 150 W.
The surface morphology of coating films was observed by field emission scanning electron microscope (SEM, JSM-7800F, Tokyo, Japan) at 3.0 kV accelerating potential measuring at 0 h and 360 h of aging.

The color change of coating films during UV-aging test at different time was measured by a CR-400 color difference meter (Konica Minolta, Tokyo, Japan). Total chromatic variations (ΔE) were calculated from CIE (L*a*b*) 1976 formula [36].

The contact angle of water of coating films during UV-aging test at different time was determined by static contact angle (SCA, JY-82, Chengde Dahua Testing Machine Co. LTD, China) at 25 °C and ± 0.5° instrument error.

2.2.2. Evaluation of application on painted muds
The painted muds samples are a laboratory simulated grotto mural painted muds. In a circular plastic petri dish with a diameter of 6.5 cm, a proportion of soil, sand, wheat straw and water (shown in table 1) are first mixed evenly, and then a layer of 1.5cm mud layer was laid as the lower layer. The lower layer mud was dry at room temperature. When the lower layer mud was semi-dry, the soil, sand, hemp and water in a proportion (shown in table 1) was mixed to form an upper layer with a thickness of 0.5 cm. Then the obtained mud samples were dried at room temperature for a week. And then, a layer of calcium carbonate mixed with 7% gelatin was brushed onto the obtained dried mud samples as a white bottom. After the muds coated with calcium carbonate were dried, they were divided into different areas and then painted with azurite, haematitum, mineral green and cinnabar pigments. The prepared painted muds were dried at room temperature for a week and shown in figure 2.

The fluorinated polymer coating (FP) was brushed onto the prepared painted muds samples and then the treated painted muds were dried under natural conditions until constant weight. The treated painted muds samples were put into an Q8 accelerated weathering chamber for UV-aging tests. Then, the chromatic variations...
(ΔE) and water contact angle of the samples were measured at different aging time to evaluate the protective efficacy for the FP coating. The research process is shown in the figure 3.

3. Results and discussion

3.1. The UV-aging mechanism

The ATR-FTIR spectrogram of FP film at aging 0h and 360h was shown in figure 4. It could be seen that the characteristic peaks of FP before and after UV-aging was basically not changed, which was shown that the chemical structure of FP coating was no change. But after aging, the intensity of the characteristic peaks of C=O at ~1724 cm⁻¹ and C–O–C at ~1135 cm⁻¹ in the ester group was greatly enhanced. At the same time, the intensity of the peaks of C–H at ~625 cm⁻¹ was also greatly enhanced. The enhancement of the intensity of these peaks indicated that the conformations of FP was changed presumably because of the microphase separation. Because the bond energy of C–F is very large, about 497 kJ mol⁻¹, and the energy of ultraviolet rays is 314 ~ 419 kJ/mol. Therefore, the C–F bond has excellent UV aging resistance. In our previous work [34], we found that the fluorine is mainly concentrated on the surface of the FP film, thus forming an anti-ultraviolet surface. Therefore, the FP has excellent anti-ultraviolet aging property and only a conformational change occurred after aging for 360 h. As the results of ATR-FTIR, it was believed that the UV-aging mechanism of FP coating was that its chemical structure does not change during the UV-aging, but only the conformation changes caused by microphase separation.

In order to further confirm the above results of ATR-FTIR, the XPS test was performed on the FP film during the UV-aging. The results of XPS for FP film at aging 0h and 360h were shown in figure 5. The characteristic signal peaks of C, O and F elements for FP film were clearly seen before and after aging. While, the intensity of these peaks changed before and after aging. After 360h of UV-aging, the content of F element decreased from 27.3% to 18.6% and the content of C and O element increased from 54.2% and 18.5% to 59.3% and 22.1%,
respectively. The specific element contents were listed in Table 2. In our previous work, we found that fluorine-containing segments tend to migrate to the surface during membrane formation and were vertically distributed on the surface for a relatively ordered structure. Therefore, the FP film had a high fluorine content on the surface before aging. During UV-aging, part of fluorinated segment migrates to the lower layer, while part of methyl methacrylate segment migrates to the surface, which just coincided with the above results. Even if the fluorine content on the surface of the FP is reduced during the aging process, it still has a sufficiently high content, so FP coating still has sufficient UV-resistance. The schematic diagram of the aging process of FP film was shown in Figure 6. The XPS results further proved that FP film only suffered microphase separation during UV-aging and it was coincided with the results of ATR-FTIR spectrum.

3.2. Analysis of UV-aging resistance

3.2.1. Surface morphology analysis

The changes of surface morphology for FP film were measured by SEM to evaluate the UV-aging resistance during UV-aging. The SEM results of FP film before and after UV-aging were shown in Figures 7(a), (b). Before

![Figure 6. Schematic diagram of the aging process of FP film.](image)

![Figure 7. SEM images for FP film (a), 0 h; (b), 360 h during UV-aging.](image)

| Ageing time/h | F (%) | C (%) | O (%) |
|--------------|-------|-------|-------|
| 0            | 27.3  | 54.2  | 18.5  |
| 360          | 18.6  | 59.3  | 22.1  |

![Table 2. The element contents of FP film during UV-aging.](image)

![Figure 8. Chromatic variations result of FP film during UV-aging.](image)
aging, the surface of the FP film was smooth and flat (figure 7(a)). However, after aging, the surface of FP film had some bumps (figure 7(b)). The fluorine-containing chains migrated to film surface and the fluoride side chains formed a disordered structure on the film surface, which resulted a smooth surface. After 360 h of aging, part of the ordered structure containing fluorine on the surface is destroyed, and some tiny protrusions appear on the surface. This result shown that FP film only had microphase separation, which was consistent with the aging mechanism discussed above. The SEM results shown that FP film had excellent UV-aging resistance.

3.2.2. Chromatic variations analysis
The chromatic variations (ΔE) were obtained by diffuse reflectance spectroscopy using the CR-400 color difference meter to quantitatively describe the color change of the FP coating during UV-aging. The chromatic variations with time for FP film was shown in figure 8 during UV-aging. The ΔE of FP film was gradually increased with the aging time. The ΔE of FP film was 0.46 after 360h of aging. The color change of the FP film could be regarded as no difference [36] (ΔE < 0.5). Due to the anti-ultraviolet surface formed by the fluorine-containing segments, the color of the FP surface is less affected by ultraviolet light, and shows a small color change. This result indicated that FP film had excellent UV-aging resistance and could be used as a protective coating.

3.2.3. Contact angle analysis
The water contact angle with time for FP film was shown in figure 9 during UV-aging. The contact angles of FP film decreased with the aging time. The FP film had adequate hydrophilicity (112.0°) at 0 h. After 360 h of aging, the film still retained sufficient hydrophobicity (101.0°), which was because that the FP film only undergone microphase separation and there was still a large amount of fluorine on the film surface (shown in table 1) to keep an adequate hydrophobicity during UV-aging. The contact angle decreased by only 9.8% after 360 h of aging due to the strong hydrophobic effect of the fluorinated chain segment. The result of contact angle shown that the FP film could maintain sufficient hydrophobicity for a long time and had excellent UV-aging resistance.
3.3. Analysis of application on painted muds

Color is an important information carried by painted cultural relics. Therefore, it is required that the surface coating materials should not change or rarely change the color of the painted cultural relics. The color change can be expressed by chromatic variations (ΔE). The larger the ΔE, the more obvious the color change. The chromatic variations with aging time for treated painted muds samples were shown in figure 10. As can be seen from the figure 10, the ΔE of all pigments increased with the aging time, but the ΔE of calcium carbonate and haematitum was less than 1.5 belong to just noticeable difference [36], while the ΔE of azurite, mineral green and cinnabar were more than 4.0 belong to fairly large difference [36]. From the analysis of the chromatic variations results, it can be obtained that the protective effect of protective materials on azurite, mineral green and cinnabar was generally not ideal. Because the main chemical components of these pigments are CuCO₃·Cu(OH)₂, CuCO₃·Cu(OH)₂ and HgS, they are easy to decompose and change color under ultraviolet light. Due to its own stability and the interaction with FP, calcium carbonate and haematitum had good resistance to ultraviolet rays after FP treatment. This result indicated that FP coating could provide adequate protective efficacy for calcium carbonate and haematitum painted muds, but the protection efficacy for the azurite, mineral green and cinnabar painted muds was not ideal.

The contact angle with aging time for the painted muds samples was shown in figure 11. The contact angle for all pigments decreased with aging time, but they still maintained a certain hydrophobicity (>90°). While, the calcium carbonate had the highest contact angle (112.0°) at the end of aging. Due to FP coating’s excellent hydrophobicity, the surface of these pigments after FP treatment has sufficient hydrophobicity. This result indicated that all the pigment surface treated by FP coating could maintain sufficient hydrophobicity and durability. However, combined with the results of ΔE, FP coating was suitable as a protective coating for calcium carbonate and haematitum, but not for azurite, mineral green and cinnabar.

4. Conclusions

In this paper, we conducted a UV-aging tests on synthetic fluorinated polymer coating (poly (methyl methacrylate)-poly (dodecafluorohexyl methacrylate), FP). The UV-aging mechanism of FP was characterized by ATR-FI-IR and XPS. The UV-aging resistance for FP coating was evaluated by measuring the changes in surface morphology, chromatic variations and contact angle under UV radiation. On the other hand, we also evaluated the protective efficiency of the PF coating after being applied to the painted muds samples with calcium carbonate, azurite, haematitum, mineral green and cinnabar pigments by measuring the chromatic variations (ΔE) and water contact angles of samples after treatment. The results of this studies were summarized as follows:

- The chemical structure of FP had no change and only microphase separation occurred after 360h of UV-aging.
- After UV-aging, there were just some bumps on the FP film surface and it further proved the phenomenon of microphase separation; the color change of the FP film could be regarded as no difference (ΔE = 0.46); the FP film could retain adequate hydrophobicity (100.7°).
- The color change of the calcium carbonate and haematitum was just noticeable difference (ΔE = 0.80 ~ 1.49), while the color change of the azurite, mineral green and cinnabar was fairly large.
difference ($\Delta E = 4.4 \sim 6.3$) after the painted muds samples were treated by FP coating. The contact angles of samples were reduced, but they could be kept above 90.0°. The calcium carbonate had the highest contact angle (112.0°) at the end of aging.

- This study demonstrated that the FP coating had excellent UV-aging resistance and could provide adequate protective efficacy for calcium carbonate and haematitum painted muds, but the protection efficacy for the azurite, mineral green and cinnabar painted muds was not ideal. Thus, the fluoropolymer polymer (FP) coating can be suggested for use as a suitable UV resistant protective coating for calcium carbonate and haematitum painted muds. The aging details of painted muds remains to be further studied.

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