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Effect of Graphene Oxide on UV-Thermo-Humidity Degradation of Environmentally Friendly Alkyd Composite Coating



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ARTICLE INFO	ABSTRACT
Article history: Received 18 April 2022 Received in revised form 7 September 2022 Accepted 26 September 2022 Available online 30 November 2022	This article presents the degradation of environmentally friendly alkyd composite coating containing GO as an anti-UV additive under UV-thermo-humidity conditions. The coatings were exposed under accelerated weathering treatment for 100 cycles. FT-IR spectroscopy, TGA and SEM were used to assess coatings changes before and after accelerated weathering testing. Results showed that gloss loss, adhesion, flexural strength, impact resistance, and relative hardness of coatings were much improved with 1.2 weight percent (wt. %) of GO. FT-IR spectrums of initial and aged coatings without GO illustrated that the aging process led to the increased intensity of CH ₂ and C=O (ester) groups. The coating with GO exhibited peaks of CH ₂ , and ester groups had been changed slightly. SEM images indicated that the surface morphology of aged samples changed differently depending on the presence of GO. The surface of aged coating without GO was rougher than that of coating with GO after the aging process. Results also indicated that the presence of GO had enhanced the UV-thermo-humidity durability of environmentally friendly alkyd coating.
Keywords:	
Coating Degradation, GO,	
Environmentally Friendly Alkyd Coating,	
Paint, Polymer	

1. Introduction

Degradation of polymers in general or paint coating in particular under high temperature, oxygen, sunlight, UV radiations, and humidity leads to losing adhesion, blistering or peeling [1-5]. Jianwen Hu et al. [6] showed the degradation of acrylic polyurethane varnish coating under fluorescent UV/condensation and xenon lamp exposure. Results showed that the degradation rate in the UV condition was higher than in the Xenon condition. Ahmad Ghasemi-Kahrizsangi et al. [7] showed that epoxy coating with about 2.5 wt. % carbon black had no micro-cracks after 1000 h of UV exposure. Besides that, this coating generated much less carbonyl groups than epoxy coating. Jingjing Si et al.

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[8] investigated the effect of graphene oxide on the thermal stability of composite based on phenolic. Results showed that graphene oxide increased (about 14°C) of initial degradation temperature of the phenolic resin. Rama Layek et al. [9] used graphene oxide as an additive in methylcellulose composite; mechanical and gas barrier properties of the composite were examined. Results showed that GO had enhanced mechanical and gas barrier properties of methylcellulose coatings. Oxygen permeation of composite coating with GO decreased by about 98%. David G. Goodwin et al. [10] studied the degradation of waterborne polyurethane coating containing hydrophilic GO fillers under exposure to a complex of UV radiation and moisture. The authors also evaluated the effect of GO on coating degradability under UV- moisture and UV separately. Thickness loss and infrared spectroscopy measurements indicated GO slightly improved the durability of the coating under dry UV conditions but did not under UV humidity complex. Raman spectroscopy, scanning electron microscopy, and atomic force microscopy modulus measurements showed that GO accumulation occurred at and near polymer nanocomposite surface under both conditions but to a more rapid extent under UV humidity complex conditions. Other authors have studied degradation coatings under UV radiation. Zinc oxide (ZnO), titanium oxide (TiO₂), and cerium (IV) oxide (CeO₂) have been used to prevent the coating from degrading [11-14].

Alkyd paint is one of the most popular industrial paints. This can be used for many purposes, including indoor and outdoor [1, 15, 16]. In recent decades, environmentally friendly paints have been studied to reduce volatile components by using chitosan, lignin, starch, shellac, wool fibers, vegetable oils, etc., in paints or adding oil modified with silane-based diluents to alkyd paint [17-21]. Environmentally friendly alkyd paints have been developed, but very few papers mention the degradation of those paints.

In previous research, the author found that using polysaccharides to make "Emulsion Intermediate" before adding them to the alkyd paint manufacturing process led to reducing about 20% of solvents in the alkyd paint [16]. Meanwhile, with 1.2 wt.% of GO, adhesion, flexural strength, and relative hardness of the above environmentally friendly alkyd coating increased sharply; besides that, anti-corrosion protection and thermal oxidation resistance of environmentally friendly alkyd coating were also improved [21].

This article introduces the effect of GO on the degradation of environmentally friendly alkyd composite coating under UV-thermo-humidity conditions. Surface morphology, gloss loss, mechanical properties changing, and thermal oxidation stability of environmentally friendly alkyd composite coating were also investigated.

2. Materials and Methods

2.1 Chemicals

Alkyd resin, QA 7812 (Taiwan) and Polysaccharide Resin, LPR 76 were supplied by Lorama Group Inc- Canada) and used as binders. Xylene, Industrial products (China) and kerosene were supplied by Petrolimex (Vietnam) and utilized as solvents. Several additives from Industrial products (China) were involved for different functions: dioctyl phthalate (DOP) as the plasticizer, bentonite as the thickener, and octoate cobalt as the drying agent. Meanwhile, TiO₂ (Rutile R-996) of Sichuan Lomon Corporation (China) and carbon black (N330) of Industrial products (China) were the pigments. The anti-UV additive was ACS Material Graphene Oxide S-Method (Staudenmaier Method)- GO, a product of ACS Material LLC (USA).



2.2 Paint Preparation

The paint preparation was conducted in this work according to the procedure suggested by the previous works [16, 21].

2.2.1. Emulsion Intermediate preparation

Table 1 summarizes the raw material composition. First, alkyd resin was stirred at a speed of 1200-1500 rpm. Second, kerosene and xylene were gradually added. The speed was then increased to 3000 rpm, and LPR 76 was added while stirring for 20 minutes. Finally, water was added and stirred for about 30 minutes at 3000 rpm to obtain a homogeneous emulsion intermediate solution.

	Table 1				
	Composition of Emulsion Intermediate (EI)				
No.	Components	Weight Percent (wt. %)			
1	Alkyd	18			
2	Polysaccharide	12			
3	Kerosene	11			
4	Xylene	7			
5	Water	52			

2.2.2. Paint Preparation

The raw materials were prepared as in Table 2.

For the preparation, there were two stages of grinding involved. (I) Primary grinding: 90% xylene was added along with all other ingredients and stirred for an hour at 20-40 rpm. After 24 hours, the mixture was moved to the second grinding stage. (II) Fine grinding: the mixture was ground at a speed of 1300- 1500 rpm until the paint fineness was \leq 30 μ m.

The remaining xylene was added and stirred for an hour. Any coarse particles or dirt with a 100-hole/mm² mesh was removed before canning for storage. A 100-hole/mm² mesh was used to remove any dirt or large particles in the paint. Finally, the canning step was performed for storage purposes.

Tabl	e 2	
Com	position of paint	
No.	Components	Weight Percent (wt. %)
1	Alkyd	15- 16.5
2	Kerosene	14
3	Xylene	20
4	TiO ₂	6
5	Bentonite	0.5
6	Octoate cobalt	1
7	N330	2
8	GO	0- 1.5
9	El solution	40

2.3. Sample preparation

Samples for testing were prepared on steel panels (ISO 1514:2016). Paint coatings were deposited on the cleaned panels by using a sprayer with a pressure of 4 kg/cm². The coatings were dried at a temperature of $(25\pm2)^{\circ}$ C and humidity of $(50\pm5)^{\circ}$ for 7 days before testing. The thickness of dried coatings was measured with Minitest 600 Erichsen digital meter.



2.4. Analysis methods

The coating's adhesion was tested following ISO 2409:2013. The relative hardness of the coating was determined using ISO 1522: 2006. The impact resistance and the coating's flexural strength were determined using ISO 6272-1:2011 and ISO 1519:2011, respectively. Meanwhile, the drying time of the coating was measured using ISO 9117-6:2012. ISO 1524: 2020 was used to determine the fineness of paints. The gloss of the coating was measured using ISO 2012. ISO 2813:2014 and a 60-degree angle.

The infrared spectroscopy (FT-IR) Fourier FTIR-8700 series converter was used for functional group determination. Thermal oxidation stability: Thermal gravimetric analysis (TGA) was performed in air using a NETZSCH TG 209F1 LIBRA at a heat rate of 10°C/minute from room temperature to 600°C. The morphology of coatings was examined using a FESEM Hitachi S4800 machine at 2,000 times magnification and 5 kV voltage. Following ASTM D4587-11, UV-thermo-humidity complex stability was tested in a UV/condensation weathering chamber Atlas UVCON UC-327-2 equipped with UVB-313 fluorescent lamps. It was operated under 8 hours of UV irradiation at 60°C followed by 4 hours of dark water condensation at 50°C.

3. Results and Discussion

3.1. Effect of GO content on gloss and mechanical properties of initial and aged coating

To investigate the effect of GO content on adhesion, flexural strength, and relative hardness of aged environmentally friendly alkyd coatings, samples with GO content of 0.5; 0.8; 1.2; 1.5 wt.% were prepared, as shown in Table 2. The samples were coded as M1, M2, M3, and M4. Meanwhile, M0 was used to represent samples that did not contain GO. The prepared paint was applied to ISO 1514:2016 standard panels with a thickness of (50±3) μ m and left at room temperature for 7 days before testing 100 cycles of the UV-thermo-humidity complex. The results are listed in Table 3.

	Table 3				
	Gloss and	mechanical p	roperties of coating befor	e and after 100 cycles te	esting
		Gloss an	d mechanical properties of o	coating before testing	
Mechanical properties of the coating					
Samples	Gloss at 60°C	Adhesion (Points)	Flexural strength (mm)	Impact resistance (Kg.cm)	Relative hardness
M0	75	1	2	200	0.39
M1	73	1	2	200	0.43
M2	72	1	2	200	0.45
M3	70	1	2	200	0.48
M4	65	3	4	160	0.53
	Glos	s and mechanic	al properties of the coating	after 100 cycles testing	
		Mechanical properties of the coating			
Samples	60°C	Adhesion (Points)	Flexural strength (mm)	Impact resistance (Kg.cm)	Relative hardness
M0	31	4	5	100	0.60
M1	47	3	3	160	0.55
M2	58	2	2	180	0.51
M3	67	1	2	200	0.49
M4	64	4	5	160	0.57



Table 3 shows that after 100 cycles of UV-thermo-humidity complex testing, the more GO content, the less adhesion, flexural strength, impact resistance, and gloss loss reduction. Meanwhile, when GO content increased, relative hardness had not increased respectively. It can be explained that GO had acted as an anti-UV additive which prevented polymer chains from breaking and cutting, which led to the limitation of polymer aging by UV rays [22, 23]. With GO, the mechanical properties of environmentally friendly alkyd coating would be better than those of coating without GO after 100 cycles of UV-thermo-humidity complex. That was also the same with gloss coating. Results also showed when GO content increased from 0.5-1.2 wt.%, and relative hardness increased; meanwhile, adhesion and flexural strength were the best. Still, when GO content was 1.5 wt.%, the hardness increased slightly, but adhesion and flexural strength coating decreased strongly [2, 3, 8, 21]. These characteristics can be explained by the fact that GO particles are much harder than polymer matrix, so the more GO content, the harder the relative hardness of the coating. However, if GO content increases continuously (to 1.5 wt.%), GO particles tend to agglomerate into larger particles to release surface energy and adhesion and flexural would decrease [24, 25]. M3 expressed the best properties of environmentally friendly alkyd composite coating, so it was chosen for further study.

3.2. Infrared spectroscopy (FT-IR) analysis

Degradation of coatings may not be observed obviously with their appearance but by chemical changes of functional groups. FT-IR spectroscopy was conducted on M0 and M3 samples to investigate the changes in initial and aged composite coatings. The results are shown in Figure 1.



Fig. 1. IR spectra of (a) M0, (b) aged M0, (c) M3, and (d) aged M3

Figure 1(a) and 1(b) showed that [26] peak intensity of CH_2 (at 2925 cm⁻¹) refer to Figure 1(a)) decreased sharply and changed to 2932 cm⁻¹ (Figure 1(b)), this indicated that polymer molecular chain had been cut and molecular length much shorter. At that time, intensities of ester group peaks (1720 cm⁻¹ of C=O and 1254 cm⁻¹ of C-O in Figure 1(a)) decreased, changed to 1729 cm⁻¹ and 1258



cm⁻¹, respectively (Figure 1(b)) ester bond was destroyed to form new bonds such as ketones due to the oxidation process. Figures 1(c) and 1(d) showed that environmentally friendly alkyd composite coating with GO, the peak intensity of CH₂ (at 2925 cm⁻¹ Figure 1(c)) slightly changed to 2926 cm⁻¹ (Figure 1(d)) and ester group peaks (1720 cm⁻¹ of C=O and 1254 cm⁻¹ of C-O) also almost unchanged but intensities of these peaks reduced insignificantly, compared to the sample before UV-thermo-humidity complex testing. These observations meant that GO had prevented polymer chains from breaking chemical bonds, which protected environmentally friendly composite coating from degradation by UV rays [21, 25, 27].

3.3. Morphology coatings' surfaces before and after testing

To investigate the effect of UV-thermo-humidity complex testing on the surface of composite coatings, M0 and M3 were tested with 100 UV-thermo-humidity complex cycles. Results are shown in Figure 2.



Fig. 2. SEM images of of (a) M0, (b) aged M0, (c) M3, and (d) aged M3

Figure 2 shows that after testing, there were no cracks, blisters or surface changes on the surface of M3. Compared to the surface before testing, 100 UV-thermo-humidity complex cycles aging surface of M3 was rougher. For M0, compared to the initial sample, the aged sample surface was rougher, and pinholes appeared. It may occur through oxidation and lead to micro-cavities and micro-holes at the coating surface [9, 14, 21, 28]. This can be explained by the fact that the anti-UV mechanism of GO could be recognized as physical barriers, UV absorbers and screeners, radical scavengers, and



quenchers. GO has worked as an anti-UV additive to protect the composite coating from UV radiation [10, 28]. Results indicated that GO with the content of 1.2 wt. % was suitable for protecting environmentally friendly alkyd composite coating from UV-thermo-humidity complex for 100 cycles.

3.4. Effects of GO on the thermal oxidation resistance of the coating

Thermogravimetric analysis (TGA) was used to study GO's effect on the thermal oxidation resistance of the coating. MO and M3 were tested with 100 UV-thermo-humidity complex cycles before taking for Thermogravimetric analysis. Results are shown in Table 4 and Figure 3.

Table 4

Effect of GO on the thermal oxidation resistance of the coating

Complex	Weight loss (%)		
Samples	300 °C	400 °C	500 °C
Initial M0	24.25	53.45	70.55
Aged M0	33.65	57.12	68.18
Initial M3	22.06	49.61	68.33
Aged M3	20.17	46.23	66.17



Table 4 and Figures 3(a), 3(b), 3(c), and 3(d) showed that different samples had different curve slopes. The TGA curve slope of aged M0 was the highest. At different temperatures, the weight loss of each sample was varied. Results showed that GO had remarkably improved the thermal stability of the environmentally friendly alkyd coating [29-31]. At elevated temperatures from room temperature to 400°C, the decomposition process has happened to low molecular substances, solvents, and residual functional groups of polymer chains. At this stage, the tested M0 experienced the highest decomposition, where their polymeric chains had been broken more severely than M3.



Meanwhile, for M3 (sample with GO), the GO had prevented polymer chains from easily breaking, so it had lower decomposition [25, 31, 32]. Results also showed that environmentally friendly alkyd composite coating without GO (MO) had an ash content of 29.45%; with GO, the ash content of the coating was higher, up to 31.67%. It can be explained that under high-temperature oxygen conditions, polymer chains were cut, and oxidation happened to organic substances. Oxygen promoted the formation of free radicals, and the polymer would be degraded sharply, leading to lower molecular compounds. Environmentally friendly alkyd composite coating with GO, these particles prevented penetration of heat and oxygen into the structure of alkyd chains. In addition, the thermal decomposition of GO would be coked to form ceramic, so the thermal oxidation resistance of composite coating with GO was better than that of coating without GO [16, 21, 32, 33].

4. Conclusions

The adhesion, flexural strength, impact resistance, relative hardness, and gloss loss of coating after 100 UV-thermo-humidity complex cycles highly depend on GO content. The suitable content of GO is 1.2 wt.%. Under UV irradiation, bonds of group \sim CH₂ and ester groups of samples without GO decrease very strongly. With GO, those functional groups of environmentally friendly alkyd coating have been prevented from easily breaking. Environmentally friendly alkyd composite coating with 1.2 wt. % of GO could suffer from 100 UV-thermo-humidity complex cycles with no cracks, blisters or surface changes slightly. The thermal oxidation resistance of the environmentally friendly alkyd composite coating with GO is much higher than that of the sample without GO. The decomposition from room temperature to 400 °C, of the sample with GO, is 22.06%, and the value is 24.25% of the sample without GO, respectively.

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