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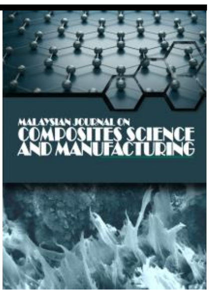

Preparation of Antifouling Composite Coating for Rubber. Part 1: Manufacturing and Laboratory Testing

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ABSTRACT

This article presents the results of investigations on an antifouling composite coating applied to a rubber substrate. Field emission scanning electron microscopy (FESEM), graphite furnace atomic absorption spectroscopy (GFAAS), energy-dispersive X-ray analysis (EDX), and other standard methods were employed to examine the results of these investigations. This study evaluates the effects of Cu_2O concentration on the release rate of the antifouling composite coating for rubber, the influence of paint fineness on its ability to release Cu_2O (where an increase in paint fineness reduces the Cu_2O release rate), and the impact of the interval between composite coating preparation and testing on the ability of Cu_2O release. Furthermore, the effects of anti-UV additives on the capability of composite coatings to withstand UV-thermo-humidity complexes were also examined. The results indicate that a Cu_2O concentration of 25 wt.%, a paint fineness of 75–80 µm, and a duration of approximately 5 days from composite coating preparation to use are optimal for achieving superior antifouling performance while preserving the mechanical properties of the composite coating. An anti-UV additive at 1 wt.% is deemed suitable for the antifouling composite coating for rubber.

Keywords:
Antifouling Paint, Composite Coating, Cu_2O, Polymer, UV-Thermo-Humidity Complex Stability

1. Introduction

Antifouling paints are commonly used to prevent oysters, mussels, and other marine species from being destroyed, causing corrosion to hulls. This brings economic benefits to ship owners, particularly by expanding the lifespan of ships [1-4]. Scientists have studied and manufactured

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antifouling paints based on acrylic resins, vinyl copolymers, silicone and used antifouling additives such as zinc, copper, Diuron (N'- (3,4-diclophenyl) -N, N-dimethyl-urea), Tween-20 (Polysorbate 20) [5-8]. In addition, a new generation of antifouling coatings using eco-friendly biocides, without bitumen or copper oxide, has also been studied [9-13]. Nanoparticles such as silver nanoparticles, carbon nanotubes, graphene, titanium dioxide (TiO2), and zinc oxide (ZnO) are promising novel approaches for biofouling agents. Nanocomposite coatings have good adhesion to the substrates, and they can express better protection of coating because they can eliminate the presence of holes in coatings and the interface between coatings and substrates [14]. Xiangping Hao *et al.* studied the capability of chitosan to change the swelling of capsules with amino protonation and deprotonation, depending on changes in pH conditions. Chitosan was combined with capsaicin to form nanocapsules. The results showed that pH values influenced the releasing behaviour of nanocapsules. Additionally, pH changes were also corresponding to bacteria reproduction. The release rate increased in the acidic environment and was lower in the alkaline environment [15]. Until now, studies and products of antifouling paints have primarily focused on steel ships, wooden ships, and concrete wharves [16 - 20]. In fact, besides steel, wood, and composite ships, there are also rubber-coated ships. However, publications on antifouling paints for rubber shells are hardly mentioned.

This article discusses a study on antifouling paint for ships with an outer shell made of neoprene rubber. Specifically, it examines an antifouling paint system consisting of a primer made from neoprene rubber, epoxy resin, resin, solvent, and an antifouling paint composed of vinyl copolymers, pine rosin, anti-UV additives, and antifouling agents such as Cu_2O and Diuron (80DF-Alligare), among others. The article examined the effects of Cu_2O at concentrations of 10, 15, 20, 25, 30% on Cu release rate of coating, the impacts of paint fineness (45- 90 µm) on the Cu release ability of coating, and the effects of 3 to 12 day period from sample preparation to the point of testing on Cu release. Additionally, the influence of Cu_2O content on some mechanical properties of the coating was also analyzed [2,3,6,8,11]. The effects of an anti-UV additive under a 45-cycle UV-thermo-humidity complex of the coating were also investigated to confirm the suitability of the studied paint as an antifouling coating for neoprene rubber shells.

2. Materials and Methods

2.1. Chemicals

Neoprene rubber (trademark SN231) was supplied by Nairit/United Chemical Products Ltd (China). Epoxy resin, Grade YD-115, was supplied by Kukdo Chemical Co., Ltd (Korea). Pine rosin was supplied by Tung Viet Chemicals Corporation (Vietnam). Acrylonitrile-butadiene rubber with acrylonitrile content 26 wt% (trademark CKH-26) was supplied by VF FGUP (Voronezh, Russia). Vinyl copolymer (trademark Laroflex MP 25) was supplied by BASF (USA). Cu_2O powder was supplied by American Chemet Corporation (USA). Biocide (trademark Diuron 80DF) was supplied by Alligare LLC (USA). Anti-UV additive (trademark UV-531) was supplied by Cytec (USA). Dioctyl phthalate (DOP) plasticizer, xylene, white spirit, bentonite, butyl acetate, and black coal N330 are industrial products made in China. $BaSO_4$ (particle size ≤ 30 µm) is also an industrial product made in China.

2.2. Sample Preparation

2.2.1 Fabrication of Primer Paint

Raw materials for primer paint were prepared as in Table 1. Primary grinding: Add 90% of xylene along with all neoprene rubber, epoxy, pine rosin, N330, bentonite, BaSO4, and other additives to the tank, then stir at 80-100 rpm for an hour. Let the mixture sit for 24 hours. Fine grinding: Grind at a speed of 1,300- 1,500 rpm until paint fineness is ≤ 20 µm. Preparation: Add the remaining xylene and stir for 2 hours. Collect paint samples for testing. Filtering - canning - storage: Use a 100 hole/mm^2 mesh to remove any coarse particles or dirt, then switch to canning for storage.

Table 1
Compositions of primer paint

No.	Components	Content (wt. %)
1	Neoprene rubber (30% in xylene)	35
2	Epoxy	5
3	Pine rosin	3
4	N330	3
5	DOP	2
6	Bentonite	1
7	$BaSO_4$	5
8	Xylene	20
9	Butyl axetate	10
10	White spirit	16

2.2.2. Fabrication of Antifouling Paint

Table 2
Compositions of antifouling paint

No.	Components	Content (wt. %)
1	Vinyl copolymer	20
2	Pine rosin	20
3	DOP	2
4	Cu_2O	10-30
5	Diuron 80DF	2
6	N330	4
7	UV531	0-1.2
8	Bentonite	1
9	Xylene	10
10	White spirit	10-30

The raw materials for antifouling paints were prepared as in Table 2. The 90% white spirit was added along with all Vinyl copolymer, Pine rosin, N330, bentonite, DOP, Cu_2O, Diuron 80DF, and other components into the tank and stirred at 80-100 rpm for an hour. The mixture was let to sit for 24 hours. Fine grinding: Grind at a speed of 1,300-1,500 rpm until paint fineness ≤ 90 µm. Preparation: Add the remaining xylene and stir for 2 hours. Collect the paint samples for testing. Filtering - canning - storage: Use a 100 hole/mm^2 mesh to remove any coarse particles or dirt, then switch to canning for storage.

2.3. Analysis Methods

After being made, the paint samples were processed according to ISO 15528: 2013 on sample sheets as per ISO 1514: 2004, including steel sheets for evaluating the mechanical properties of the composite coating and rubber sheets for assessing other properties of the composite coating. The gloss of composite coating was determined according to ISO 2813: 2014 with an angle of 60 degrees. The adhesion of composite coating was determined according to ISO 2409: 2013, the flexural strength of the coating was determined according to ISO 1519: 2011, and the impact resistance of the coating was determined according to ISO 6272-1:2011.

The morphology of the composite coating film was observed by the FESEM Hitachi S4800 machine (Japan) with a magnification of 2,000 times and a voltage of 15KV. X-ray diffraction analysis (XRD) was performed using D8 Advance Bruker (USA).

The 45-cycle UV-thermo-humidity stability testing was performed according to ASTM D4587-05 (each 12-hour cycle consisted of 8 hours of UV radiation at 60 °C followed by 4 hours of condensation at 50 °C). The testing device used was the ATLAS UVCON UC 327-2 (USA) with UVB-313 fluorescent lamps for UV exposure and moisture condensation.

The Cu release rate of the composite coating in the laboratory was determined by extracting Cu from it and analyzing it using graphite furnace atomic absorption spectroscopy (GFAAS). The paint was applied to sample sheets and left to dry naturally for four days before testing. Each sample was soaked in 1 liter of artificial seawater. Before each experiment, samples were removed, and the solution was analyzed for Cu content. Artificial seawater was prepared according to ASTM D1141. The copper release rate from antifouling coatings in substitute ocean water was determined according to the ASTM D6442-05 standard.

3. Results and Discussion

3.1. Effect of Copper (I) Oxide Content on Cu Release Rate of Antifouling Composite Coating

The toxic release rate of antifouling composite coating depends on the solubility of binders and the concentration of antifouling additives in the composition of paints. This study used Cu_2O as an antifouling additive, and tests were carried out with Cu_2O content of 10, 15, 20, 25, and 30 % corresponding to samples M1, M2, M3, M4, and M5, as shown in Table 3. Neoprene sample sheets were first painted with primer, then coated with 150 µm of antifouling paint and left to dry naturally for four days before being soaked in artificial seawater at 25 °C. The investigation results are shown in Figure 1.

Table 3
Compositions of antifouling paint for Cu releasing rate testing

Components (Wt. %)	Samples				
	M1	M2	M3	M4	M5
Vinyl copolymer	20	20	20	20	20
Pine rosin	20	20	20	20	20
DOP	2	2	2	2	2
Cu_2O	10	15	20	25	30
Diuron 80DF	2	2	2	2	2
N330	4	4	4	4	4
UV531	1	1	1	1	1
Bentonite	1	1	1	1	1
Xylene	10	10	10	10	10
White spirit	30	25	20	15	10

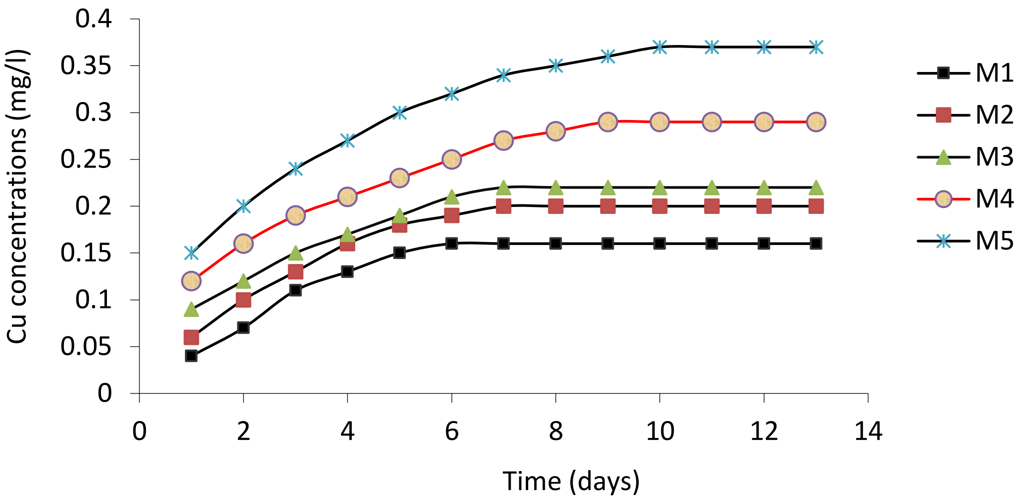

Fig.1. Cu concentration release by time

Figure 1 shows that during the initial days of the testing process (the 7th day for M1 and the 10th day for M5), Cu concentrations increased rapidly from 0.04 up to 0.16 mg/l for M1 and from 0.15 up to 0.37 mg/l for M5. After this period, the Cu concentration in the testing solution increased very slowly. This could be explained by the fact that, in the first days, the Cu released mainly comes from Cu_2O accumulated on the coating surface [8, 9, 12, 19]. Thus, if the binder is low or has no solubility, the antifouling effect will be poor because the antifouling additives within the coating cannot escape.

3.2. *Effect of Paint Fineness on Ability of Cu Concentration Release*

Tests were conducted using the M4 sample to study the effects of paint fineness on Cu release from the composite coating. The paints were tested at different grinding times corresponding to different levels of fineness. Neoprene sample sheets were painted with primer paint first, then covered with a thickness of 150 µm antifouling paint and left to dry naturally for 4 days before being tested. Tests were conducted in artificial seawater, and samples were rotated at 150 rpm at 25 °C for 10 hours. The results are shown in Table 4.

Table 4
Effects of paint fineness on Cu releasing

No.	Paint fineness (µm)	Cu release (mg/l)
1	45-50	0.07
2	55-60	0.15
3	65-70	0.28
4	75- 80	0.37
5	85-90	0.39

As shown in Table 4, as the fineness of the paint increased, the speed of Cu release decreased. This can be explained by the fact that the finer the paint, the more tightly the Cu_2O particles are encapsulated, reducing the paint's Cu_2O release ability. However, low paint fineness would affect the mechanical properties of the composite coating [5, 7, 20]. Therefore, paint with a 75–80 µm fineness was chosen for further studies.

3.3. Effect of Time Interval Between Sample Preparation and Testing on Cu Release

M4 with a fineness of 75–80 µm was used to study the effects of the period since sample preparation on Cu_2O release from the composite coating. Neoprene sample sheets were painted with primer paint first, then covered with a thickness of 150 µm antifouling paint and left to dry naturally for 3, 4, 5, 6, 7, 8, 9, 10, 11 and 12 days before testing for ability of Cu_2O release. Tests were done in artificial seawater, and samples were rotated at 150 rpm, 25 °C for 10 hours. The results are shown in Figure 2.

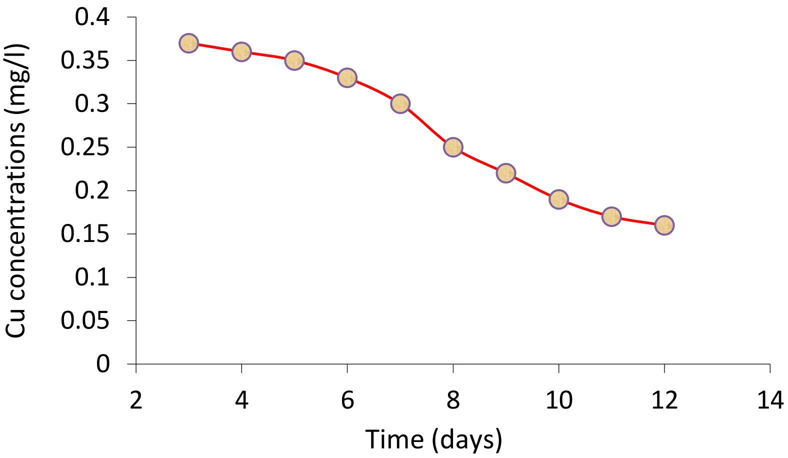

Fig. 2. Effects of time interval since sample preparation on Cu release

Figure 2 shows that the ability to release Cu was much higher after 3 to 5 days of sample preparation compared to 10 to 12 days. This can be explained by the fact that the antifouling mechanism of the composite coating is based on its solubility. The higher the solubility of the coating, the better its antifouling performance. Figure 2 showed that after 3 to 5 days of preparation, the composite coating had not completely dried, making it easier to dissolve compared to coatings that had been painted for a longer period, which may have been fully dried by that time [8, 12, 19]. However, after 3 days of composite coating preparation, the mechanical properties of the coating are still poor, making it easy to peel off. Therefore, after approximately 5 days of preparation, it is necessary to launch the ship to ensure the high-release ability of the composite coating while still maintaining its mechanical properties.

3.4. Effects of Cu_2O Content on the Mechanical Properties of Composite Coating

To investigate effects of Cu_2O content on mechanical properties of coating, samples were prepared using steel sheets and left to dry naturally for 5 days before testing. The results are shown in Table 5.

Table 5 shows that when the Cu_2O content was ≤ 25% (M1 to M4), it had little effect on the adhesion of the composite coating and did not affect its impact strength. However, when the Cu_2O content exceeded 25%, the impact resistance of the coating decreased. Similarly, when the Cu_2O content was ≤ 25%, it did not affect the flexural strength of the coating. However, when the Cu_2O content reached 30%, both the flexural strength and adhesion of the coating decreased significantly.

Cu_2O acts as the antifouling additive in the paint, so it is necessary to use the highest possible concentration while still ensuring the mechanical properties of the composite coating [21, 22].

Table 5
Effects of Cu_2O content on the mechanical properties of composite coating

Samples	Adhesion (points)	Impact resistance (Kg.cm)	Flexural strength (mm)
M1	1	200	2
M2	1	200	2
M3	1	200	2
M4	2	180	2
M5	3	160	4

3.5. X-Ray Diffraction Analysis of Antifouling Composite Coating

To analyze the appearance of Cu_2O in antifouling composite coating, X-ray diffraction analysis (XRD) was used. The result is shown in Figure 3. It shows that the solids in the antifouling paint had strong reflection peaks at 2θ angles such as 36.38°, 42.29°, 61.52°, and 73.76°, which represent cuprite-copper (I) oxide (Cu_2O) crystals. This confirms that Cu_2O was used in the antifouling paint [1,9].

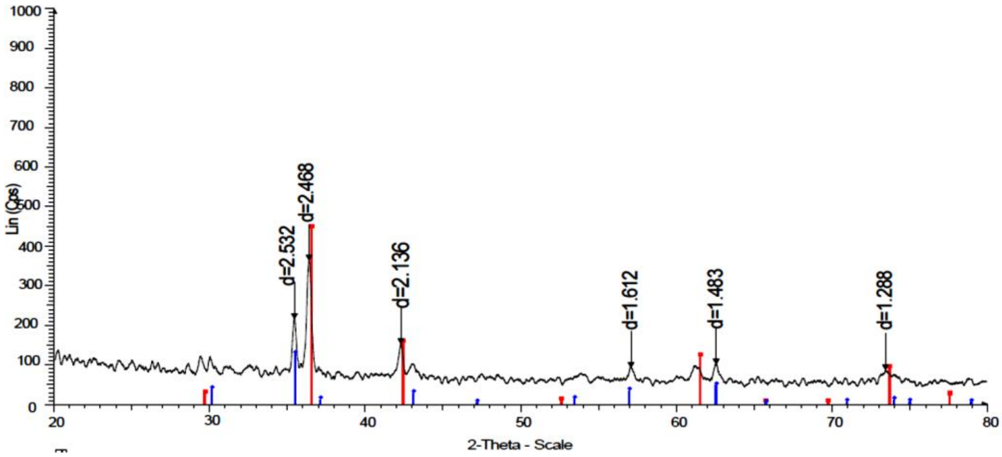

Fig. 3. XRD of antifouling composite coating

3.6. Effect of Anti-UV Additive on Composite Coating UV-Thermo-Humidity Complex Stability

To investigate the effects of anti-UV additive content on UV-thermo-humidity complex stability of composite coating, samples with the compositions shown in Table 6 were used.

The investigation was conducted over 45 cycles according to ASTM D4587-05 (one cycle consisting of 8 hours of UVB irradiation at 60 °C, followed by 4 hours of condensation at 50 °C). The results are shown in Table 8 and Figure 4. Before the test, the gloss of the samples was checked, and the results are shown in Table 7.

Table 6
Compositions of samples for investigating UV-thermo-humidity complex stability

Components (%)	Samples				
	M6	M7	M8	M9	M10
Vinyl copolymer	20	20	20	20	20
Pine rosin	20	20	20	20	20
DOP	2	2	2	2	2
Cu_2O	25	25	25	25	25
Diuron 80DF	2	2	2	2	2
N330	4	4	4	4	4
UV531	0	0.4	0.8	1	1.2
Bentonite	1	1	1	1	1
Xylene	10	10	10	10	10
White spirit	16	15.6	15.2	15	14.8

Table 7
Gloss of composite coating

No.	Samples	Gloss at 60°
1	M6	76
2	M7	76
3	M8	75
4	M9	75
5	M10	74

Table 8
Effect of anti-UV additive on coating UV-thermo-humidity complex stability

Samples	UV-thermo-humidity complex stability	Gloss at 60°
M6	- Surface: crack, peeling - Color loss	25
M7	- Surface: crack, blistering - Gloss loss, fade	31
M8	- Surface: no crack, no peeling, no blistering - Gloss and color changed a little	42
M9	- Surface: unchanged - Gloss and color changed very little	70
M10	- Surface: unchanged - Gloss and color: unchanged	72

Table 8 and Figure 4 illustrate that an increase in UV additive content enhances the UV-thermo-humidity complex stability of the coating. However, when the anti-UV additive content reaches 1 wt.% or higher, the composite coating's resistance to the UV-thermo-humidity complex stabilizes, showing no further improvement [23-25].

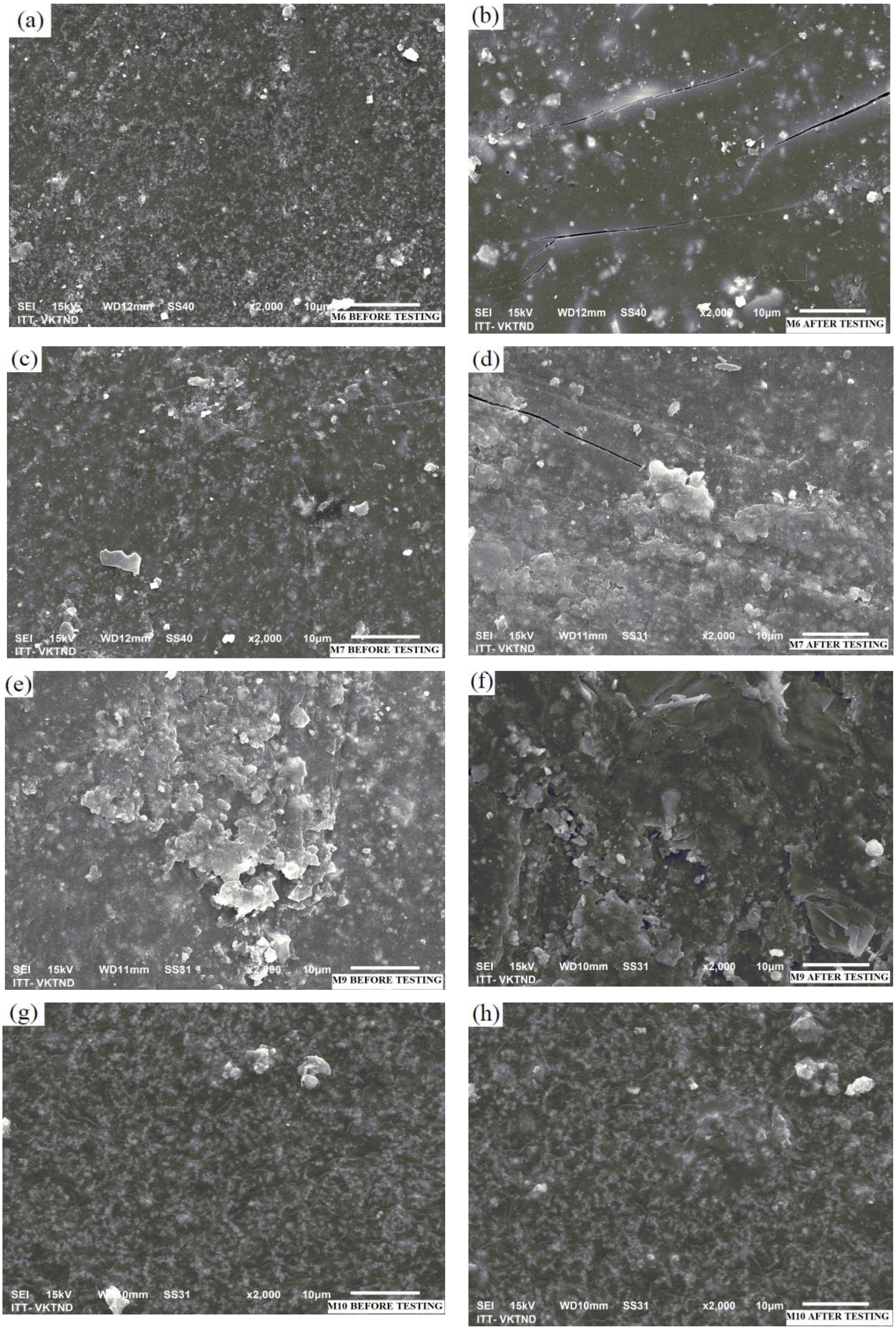

Fig. 4. SEM images of samples before (left) and after (right) 45-cycle UV-thermo-humidity complex: (a-b) M6, (c-d) M7, (e-f) M9 and (g-h) M10

4. Conclusions

After a testing period of 7 to 10 days, the Cu concentration increased rapidly from 0.04 to 0.16 mg/L for samples with a Cu_2O content of 10% and from 0.15 to 0.37 mg/L for samples with a Cu_2O content of 30 wt.%. As the fineness of the paint increases, the Cu release speed decreases. A fineness of approximately 75–80 µm balances Cu release speed and the mechanical properties of the coating. A period of approximately 5 days after sample preparation provides the highest antifouling effect while still ensuring the mechanical properties of the composite coating. A Cu_2O content of 25 wt.% is suitable for making antifouling paints, as it ensures both antifouling ability and the mechanical properties of the coating. As the anti-UV additive content increases from 0 to 1.2 wt.%, the UV-thermo-humidity complex stability of the composite coating improves. However, above 1 wt.%, the coating's ability to resist the UV-thermo-humidity complex remains unchanged. Thus, an anti-UV additive content of 1 wt.% is suitable for rubber-based antifouling composite coatings.

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