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Journal homepage:
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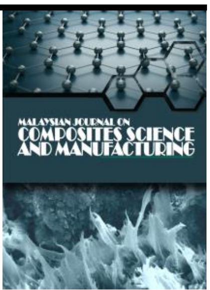
ISSN: 2716-6945



Improvement of Environmentally-Friendly Alkyd Composite Coating with Graphene Oxide

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ARTICLE INFO

Article history:
Received 22 December 2021
Received in revised form 8 March 2022
Accepted 16 March 2022
Available online 31 March 2022

Keywords:
Corrosion protection, graphene oxide, environmentally friendly alkyd coating, paint, polymer

ABSTRACT

In this article, environmentally-friendly alkyd coatings with different graphene oxide (GO) content of 0-1.5 weight percent (wt. %) were prepared for improving mechanical properties, corrosion protection and thermal oxidation resistance of coatings. GO in the coating was characterized by FT-IR. The corrosion resistance of environmentally-friendly alkyd coatings containing GO was investigated by salt fog testing. Effects of GO content on adhesion, flexural strength, relative hardness and thermal oxidation stability of alkyd coatings with and without GO were also examined. GO with the content of 1.2 wt. % significantly improved mechanical properties, corrosion resistance, thermal oxidation resistance of environmentally-friendly alkyd coatings.

1. Introduction

Improving the mechanical properties of coatings or composites with additives have been investigated by many authors [1-5]. Jian-Gong Wen et al. [6] had prepared modified graphene oxide (GO) using the isophorone diisocyanate (IPDI), and it was referred to as the IP-GO. Their results showed that IP-GO improved its dispersion and compatibility in the water polyurethane matrix compared with neat graphene oxide. Besides that, composite materials with 0.3 wt. % of IP-GO had the highest mechanical properties and the lowest water absorption. Besides, the anti-corrosion efficiency of the coating was significantly improved when adding GO and IP-GO.

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https://doi.org/10.37934/mjcsm.7.1.110

Bin Yu et al. [7] used functionalized graphene oxide (FGO) in polyurethane acrylate (PUA) cured by UV. Structure, morphology, thermal stability, mechanical properties of FGO/PUA nanocomposite coatings were examined. Results showed that FGO was well dispersed in the PUA matrix and made strong interfacial adhesion with PUA after curing. FGO strongly enhanced the thermal stability and mechanical properties of PUA coating. Initial degradation temperature of PUA composite with 1.0 wt. % of FGO increased up to 316 °C from 299 °C of neat PUA, respectively. Glass transition temperature (Tg) of FGO/PUA nanocomposite was slightly increased compared to PUA only. Zhengyan Gao et al. [8] prepared graphene-modified epoxy coating (G/OEP) to enhance the corrosion resistance of AZ31 magnesium alloy. Results showed that graphene content of 0.6 wt. % had significantly improved surface quality of coating and reduced defects. The coating could provide good corrosion protection for magnesium alloy. In comparison to the corrosion current density of magnesium alloy, G/OEP coating could strongly reduce corrosion current density. Due to the application of coating/ composite, some kinds of additives such as GO, CeO_2, TiO_2, modified nano carbon black have been used to improve mechanical properties or anti-corrosion [9-14].

Alkyd paint is made from alkyd resin and is one of the most popular paints. This kind of paint can be used for indoor and outdoor purposes [15-17]. Recently, environmentally-friendly paints have been studied using chitosan, lignin, polysaccharides, shellac, wool fibres, and vegetable oils to reduce volatile components. But, hardly any papers mentioned using GO as an additive for enhancing the performance mechanical properties of those kinds of paints [16-20].

In previous research, the author had studied manufacturing environmentally-friendly alkyd from alkyd and polysaccharides. Using polysaccharides to make "Emulsion Intermediate" before adding to alkyd paint reduced about 20 % of solvents in the alkyd paint [16]. Meanwhile, this article introduces the preliminary study of the effect of graphene oxide on mechanical properties, thermal oxidation stability, and salt mist stability of environmentally-friendly alkyd composite coating. It is to confirm that some coating properties have been improved with GO.

2. Materials and Methods

2.1 Chemicals

Binders: Alkyd resin, QA 7812 (Taiwan) and Polysaccharide Resin, LPR 76 were supplied by Lorama Group Inc- Canada). *Pigments:* TiO2 (Rutile R-996): was a product of Sichuan Lomon Corporation (China), and carbon black (N330): Industrial products (China). *Solvents:* Xylene, Industrial products (China) and kerosine were supplied by Petrolimex (Vietnam). *Additives:* Dioctyl phthalate (DOP) as a plasticizer, bentonite as a thickener, octoate cobalt as a drying agent: Industrial products (China). ACS Material Graphene Oxide S-Method (Staudenmaier Method)- GO was a product of ACS MATERIAL LLC (USA).

2.2 Sample preparation and Methodology

The sample preparation, testing and analyses are mostly similar to the one reported in [16] and consist of the following steps:

2.2.1 Emulsion Intermediate preparation

The composition of raw materials as in Table 1 was prepared. The alkyd resin was first stirred at 1,200- 1,500 rpm before the kerosine and xylene were gradually added. Then, the speed was increased up to 3,000 rpm before LPR 76 was added and continued stirring for 20 minutes. Lastly, the water was added and stirred at 3,000 rpm for about 30 minutes to achieve a homogeneous emulsion intermediate solution.

Table 1
Composition of Emulsion Intermediate (EI)

No.	Components	Weight percent (wt. %)
1	Alkyd	17
2	Polysaccharide	13
3	Kerosine	10.5
4	Xylene	7
5	Water	52.5

2.2.2 Paint preparation

Raw materials, as in Table 2 were prepared. There were two stages of grinding involved, 1) *Primary grinding:* 90% of xylene were added with all other materials and stirred at 20- 40 rpm for an hour. The mixture was kept to be conditioned for 24 hours. 2) *Fine grinding*: Then, the mixture was subjected to fine grinding at a speed of 1,300- 1,500 rpm to reach the paint fineness of ≤ 30 μm.

Preparation: Then, the rest of the xylene (another 10%) was added and stirred for an hour. After that, the paint samples for testing were acquired through filtering before proceeding with the canning step. A 100 hole/mm^2 mesh was used to remove any coarse particles or dirt in this stage. Then, it was followed by the canning step for storing purposes. Then, the paints were formed into coatings and subjected to further testing and analyses.

Table 2
Formulation of paint

No.	Components	Weight percent (wt. %)
1	Alkyd	15- 16.5
2	Kerosine	12
3	Xylene	10
4	TiO_2	17
5	Bentonite	0.5
6	Octoate cobalt	1
7	N330	3
8	GO	0- 1.5
9	EI solution	40

2.3 Analysis methods

The coatings of the paints were tested for several criteria and properties, as follows:

- The adhesion of the coating was determined according to ISO 2409:2013.
- The flexural strength of the coating was determined according to ISO 1519:2011.
- The impact resistance of the coating was determined according to ISO 6272-1:2011.
- The relative hardness of the coating was determined according to ISO 1522: 2006.
- The drying time of coating was determined according to ISO 9117-6:2012.

Compositional: Infrared spectroscopy (FT-IR) was conducted on the Fourier FTIR-8700 series converter. *Thermal oxidation stability:* Thermal gravimetric analysis (TGA) was analyzed by NETZSCH

TG 209F1 LIBRA in the air with a heating rate of 10 °C/minute from room temperature to 600 °C. *Morphological characteristics:* Morphology of coating film was observed by the FESEM Hitachi S4800 machine with a magnification of 1,000 times and voltage of 5 kV. Salt mist, cyclic testing (5% NaCl) was determined by IEC 60068-2-52: 2017 with 04 cycles. Each cycle included 2 hours of spraying (at (35 ± 2) °C), keeping humidity condition for seven days at (40± 2) °C, with a relative humidity of (93 ± 2) %.

3. Results and discussion

3.1 Effect of GO content on mechanical properties of the coating

The samples were prepared as the formulation in Table 2 to investigate the effect of GO content on adhesion, flexural strength and relative hardness of the environmentally-friendly alkyd coating. The GO contents were varied at 0.5, 0.8, 1.2, and 1.5 wt. %. Samples were named M1, M2, M3, M4, and M0 was for the sample without GO. The prepared paints were applied on standard panels according to the ISO 1514:2016 with a thickness of 30 µm and kept at room temperature for seven days before testing. Results are shown in Table 3.

Table 3
Effect of GO content on adhesion, flexural strength and relative hardness of the coating

Sample	GO content (wt.%)	Adhesion (Points)	Flexural strength (mm)	Relative hardness
M0	0	1	2	0.38
M1	0.5	1	2	0.42
M2	0.8	1	2	0.45
M3	1.2	1	2	0.49
M4	1.5	3	4	0.54

Table 3 shows as the GO content increases from 0.5-1.2 wt. %, the relative hardness increases. The adhesion, flexural strength and relative hardness were optimum when the GO content was 1.2 wt%. Meanwhile, the adhesion point and flexural strength were greatly affected- at GO content of 1.5 wt.%, the hardness increased slightly, but adhesion and flexural strength coating decreased strongly. It can be explained that GO particles have much higher hardness than the environmentally-friendly alkyd matrix. So, the more GO content in the coating, the harder the relative hardness. However, if GO content increases continuously, those particles tend to agglomerate together, forming larger particles. The agglomeration releases surface energy; hence, coating adhesion and flexural strength would decrease [21-23].

3.2. Infrared spectroscopy (FT-IR) analysis

In order to investigate the appearance of GO in environmentally-friendly alkyd composite coating, infrared spectroscopy (IR) of coating with and without GO were conducted by Fourier FTIR-8700 series transformers. Results are shown in Table 4 and Figure 1.

Figures 1a and 1b show that peaks 2928 cm^{-1}, 2923 cm^{-1}, 2853 cm^{-1} and 2852 cm^{-1} as CH group of aliphatic hydrogens [24]. Peaks characteristic of C=O vibrations were observed at 1720 cm^{-1} and 1724 cm^{-1}. The spectrum of GO exhibited C-OH, and C-O group vibrations can be seen at 1225 cm^{-1} and 1067 cm^{-1}, respectively (Figure 1b). There had some new peaks corresponding to the GO in Figure 1b, in which peak 1225 cm^{-1} represented for vibration of C-OH bond, and peaks 602 cm^{-1} and 647 cm^{-1} were typical for the vibration of C= C (out of plane ring bending).

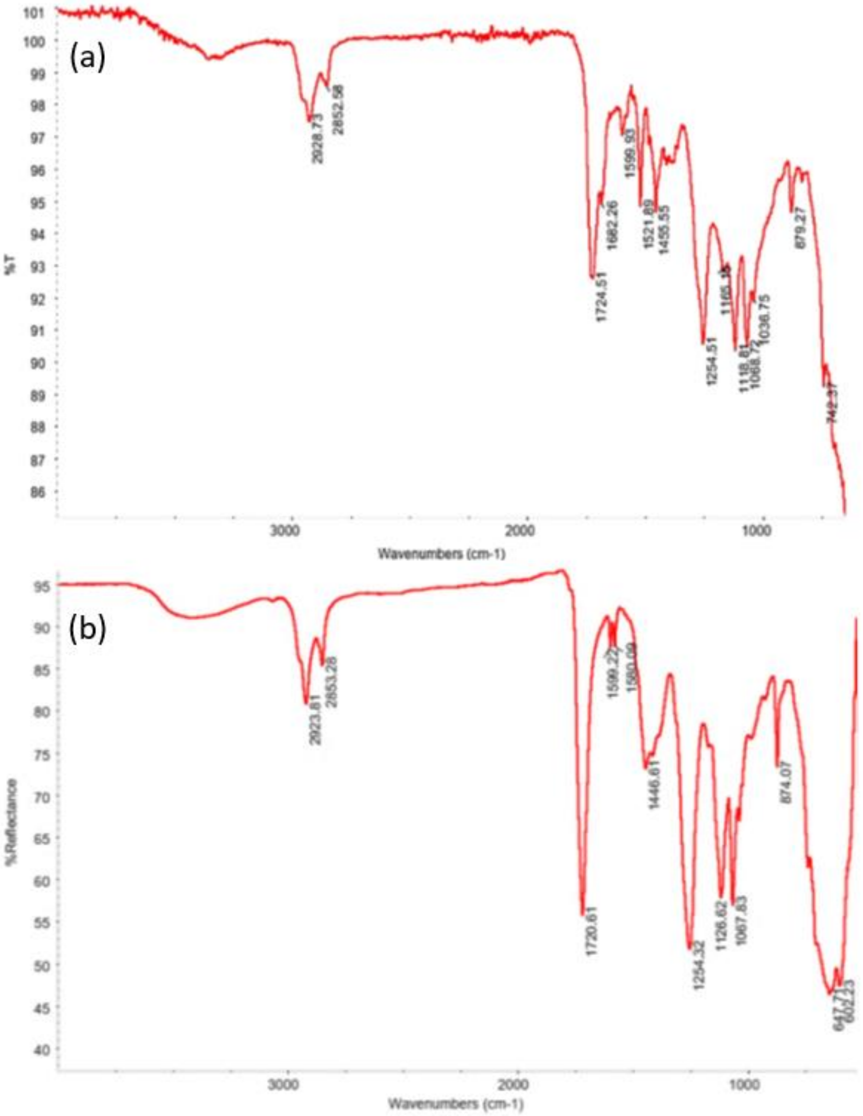

Fig. 1. IR of environmentally-friendly alkyd coating, (a) without GO and (b) with GO

Table 4
Selected measured IR bands of coating

No.	Typical spectrum	Wavenumbers (cm^{-1})
1	vCH (Aliphatic hydrogens)	2928
2		2923
3		2853
4		2852
5	vC=O vibration	1724
6		1720
7	vC-OH	1225
8	vaC-O-C (asymmetry)	1068
9	va C-O-C (symmetry)	1067
10	vCH (bending)	879
11		874

3.3 Salt mist resistance of the coatings

The M0 (without GO) and M3 (with GO) coating samples were tested for salt mist resistance. It was to investigate the effect of the GO presence towards salt mist on the protection of the coatings. After making, the samples were kept at room temperature for seven days before testing in a salt mist chamber with 5% NaCl for four cycles. The optical photographs of the coatings' surfaces were examined by cross-cut test (cross-cut surface of coating was scratched and cleaned carefully) before and after salt mist cyclic testing. The SEM images were also taken at the magnifications of 1,000 times. It was to observe the morphological surfaces of the coatings before and after testing. The results are shown in Figure 2 and Figure 3.

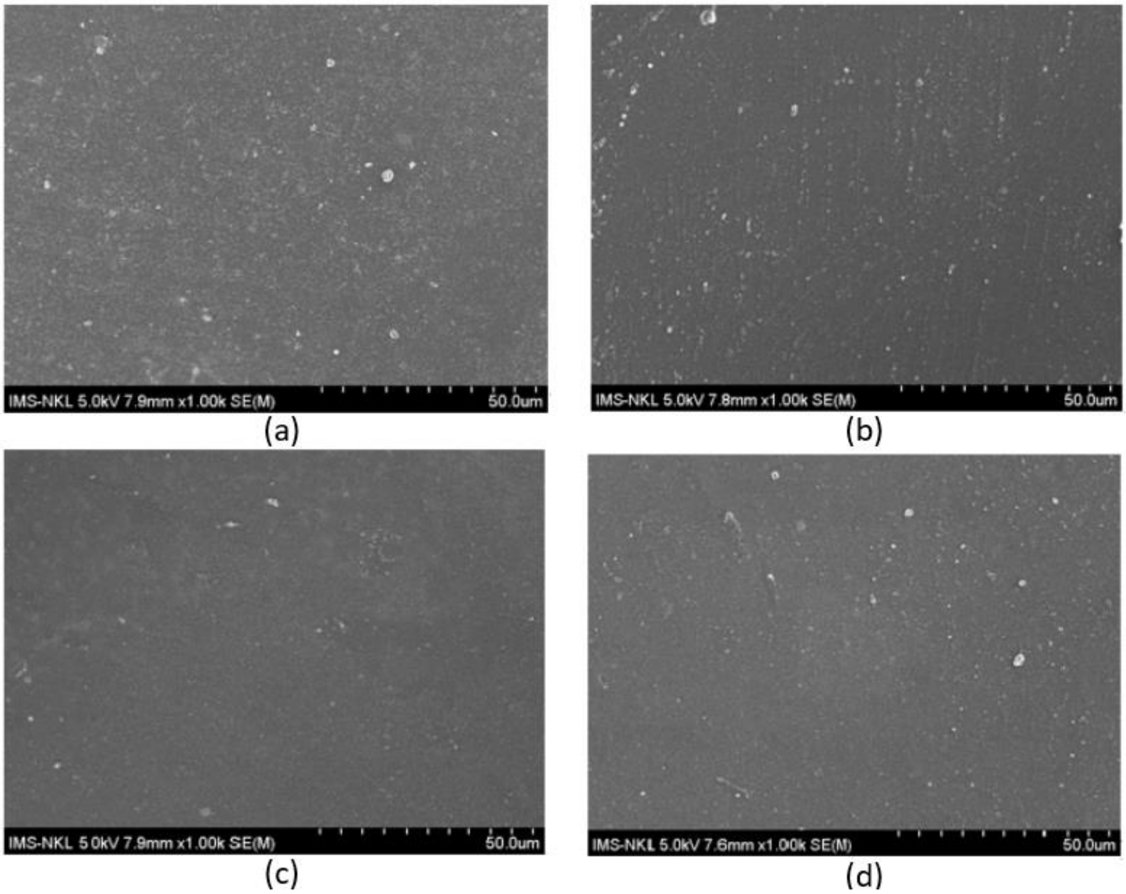

Fig. 2. SEM micrographs of (a) M0 before salt mist testing, (b) M0 after salt mist testing,(c) M3 before salt mist testing and (d) M3 after salt mist testing

Figures 2a, 2b, 2c, and 2d show no apparent phenomenon on the coating surfaces after the salt mist testing, indicating that coating surfaces had not been much affected after four testing cycles. Meanwhile, Figure 3 shows that the rust spots appeared at the cutting boundary after four cycles of salt mist testing. The coating sample without GO had larger blistering, coating with GO had some smaller blistering spots. It proves that the steel surface had been corroded and reduced the adhesion to the coating. Besides, it was observed that the environmentally-friendly alkyd coating with GO could protect steel substrate better than the coating without GO. The GO played a vital role in good adhesion between coating steel substrates. This might result from the C-OH groups (peak 1225 cm^{-1}) on the surface of GO, which would create physical bonding of the coating to the steel substrate. This bonding guarantees the long-term protection of environmentally-friendly alkyd composite coating with GO. The process happened electrochemically, so SEM images could not clearly express the differences between samples [25-28].

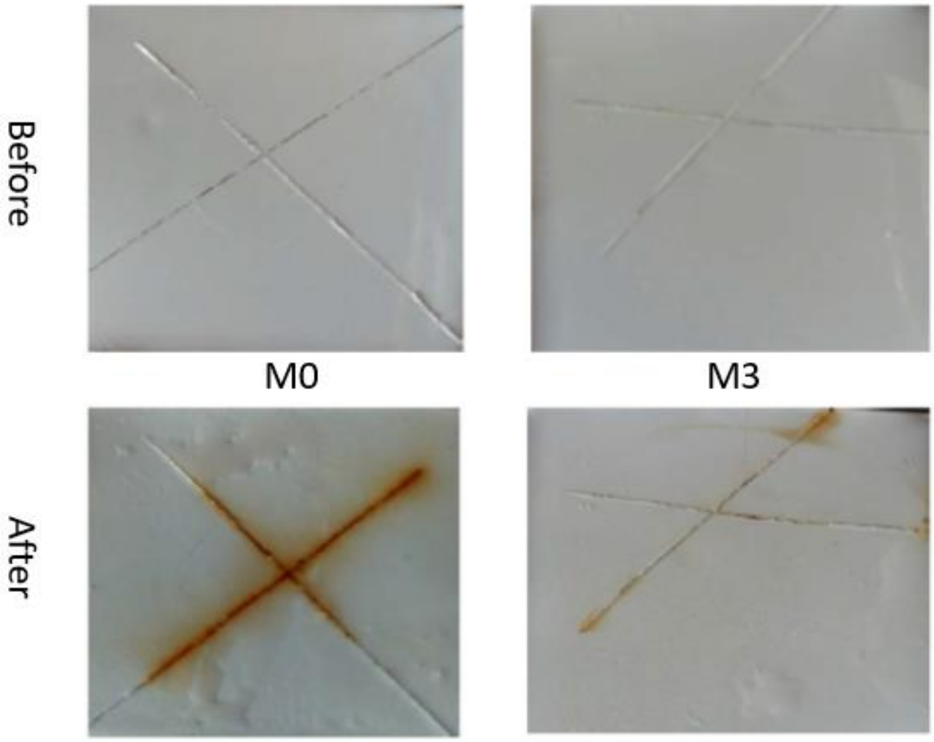

Fig. 3. Optical photographs on the surface of coatings examined before and after salt mist testing

3.4. Effect of GO content on coating's thermal oxidation resistance

The thermogravimetric analysis (TGA) was performed on M0 and M3 samples to study the effect of GO content on the thermal oxidation resistance of environmentally-friendly alkyd coating. Results are shown in Table 5 and Figure 4.

Table 5
Effect of GO on the thermal oxidation resistance of environmentally-friendly alkyd coating

Samples	Weight loss (%)		
	300 °C	350 °C	500 °C
M0	21.65	45.98	64.82
M3	18.73	36.45	61.67

Table 5 and Figures 4a and 4b show that the slopes of TG curves of M0 and M3 samples are almost identical, but their decompositions at different temperatures were dissimilar. There were two decomposition steps: 1) Under 200 °C: decomposition of low molecular substances and residual solvents. 2) about 300 °C and above: decomposition occurred with residual functional groups in polymer branches, low molecular substances. Results also showed that environmentally-friendly alkyd coating had an ash content of 35.18 %. Meanwhile, with GO, the ash content of the coating was higher, up to 38.33 %. In high-temperature conditions with oxygen, polymer chains were broken down, and oxidation had taken placed on the organic substances. Oxygen had promoted the formation of free radicals, and polymer would be degraded strongly, leading to the formation of lower molecular compounds. For environmentally-friendly alkyd 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. Thus, GO has improved the thermal oxidation resistance of the environmentally-friendly alkyd coating [29-31].

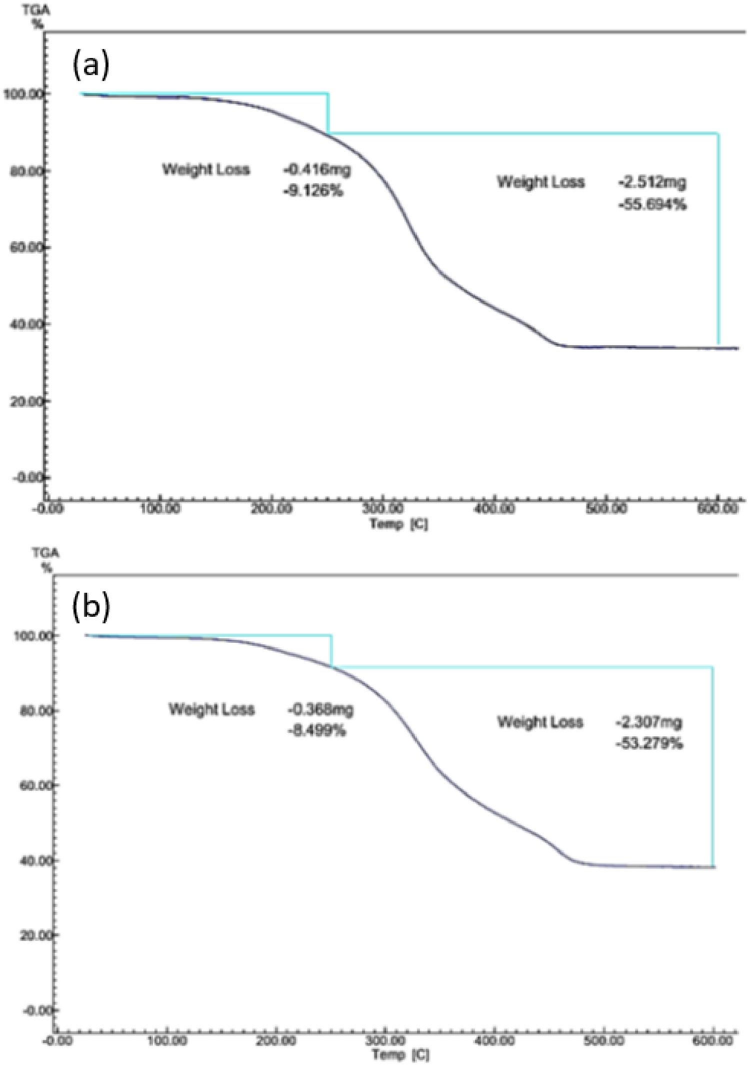

Fig. 4. TGA thermogram of (a) M0 and (b) M3

4. Conclusions

As the GO content of the environmentally-friendly alkyd coating increases from 0 to 1.2 wt%, the adhesion and flexural strength of the coating improves. Together, the relative hardness increases from 0.38 to 0.49. The coating's mechanical characteristics are optimum when the GO content is 1.2 wt%. From four cycles of salt mist testing, GO increases the anti-corrosion protection of an environmentally-friendly alkyd coating. Coating with 1.2 wt% GO protects the substrate much better than coating without it. The thermal oxidation resistance of a coating with GO is higher than that of the environmentally-friendly alkyd coating, which has a thermal oxidation resistance of 35.18 percent but a thermal oxidation resistance of 38.33 percent.

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