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Suan Sunandha Science and Technology Journal
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Influence of Tinuvin 292 on Chlorinated Rubber Varnish/ Coating Properties under UV Radiation

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Received: *27 December 2022* / **Revised:** *13 February 2023* / **Accepted:** *28 February 2023*

Abstract

This article presents the effect of Tinuvin 292 (a light stabilizer based on liquid hindered amine) on properties of chlorinated rubber varnish/coating under 100 UV-thermo-humidity complex cycles. The properties included relative hardness, adhesion, flexural strength, impact resistance, and gloss loss. The results showed that 2.5 weight percent (wt%) of Tinuvin 292 had much improved physical properties of varnish coating such as relative hardness, adhesion, flexural strength, impact resistance, and gloss loss. Fourier-transform infrared spectroscopy (FT-IR), Thermogravimetric analysis (TGA), and Field Emission Scanning Electron Microscopy (FESEM) were used to evaluate the changes of aged coatings in comparison to the initial ones. FT-IR spectrums also showed that intensities of CH_2 and C=O (ester) groups changed strongly or slightly depending on the appearance of Tinuvin 292. SEM images indicated that the surface of aged coating with Tinuvin 292 was smoother than coating without Tinuvin 292. TGA also expressed that thermal oxidation stability of chlorinated rubber varnish/ coating had been enhanced with 2.5 wt% of Tinuvin 292.

Keywords: Chlorinated rubber paint, Aged coating, Polymer, Tinuvin 292

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1. Introduction

Carbon black, graphene oxide (GO), titanium oxide (TiO_2), zinc oxide (ZnO) have been added in compositions of coatings materials (Crescenzo, Zendri, Pons, Lospez, & Marco, 2014; Das, Pandey, Mohanty, & Nayak, 2017; Kızılkonca & Erim, 2019) to protect paint or varnish coatings from aging under UV light. However, the anti-UV additives agglomeration made them difficult to be well dispersed in liquid paints as well as losing their transparency (Cogulet, Blanchet, & Landry, 2019; Kahrizsangi, Neshati, Shariatpanahi, & Akbarinezhad, 2015; Nguyen et al., 2017; Pintus, Wei, & Schreiner, 2016). Chlorinated rubber paint is made from chlorinated rubber resin of 60-68% chlorine group and characterized by fast dry, inflammable coating. Chlorinated rubber coatings have been widely used in shipbuilding, water conservancy, petrochemical industry and it also performs as a good anti-corrosion coating (Ahmed, Emira, & Tawfik, 2013; Olabisi & Adewale, 2015; Sakhri, Perrin, Aragon, Lamouric, & Benaboura, 2010; Talbert, 2007). To improve performance and promote the development of weather resistance properties, chlorinated rubber paints have been modified with coal tar pitch, thermoplastic acrylic resins (Ismail & Harun, 2016; Liu, Xiong, Lv, & Zuo, 2009; Morsi, Emira, El-Sawy, Mohsen, & Khorshed, 2019). Bano and colleagues (Bano, Khan, & Kazmi, 2013) investigated the chlorinated rubber coating in which coatings were exposed at some places such as seaside, industrial and urban areas. Accelerated testing was performed with a salt spraying chamber. Visual examination results of blistering, rusting and SEM micrographs indicated that there was a little degradation of the coating surface at natural exposure testing sites or accelerated testing. The carbonyl compounds formation was observed by FTIR spectroscopy. Lang and colleagues (Lang et al., 2019) had tested a commercially available chlorinated rubber-based iron oxide red paint for 9 months, results showed that coating did not perform well, which could not resist the colonization of major fouling organisms. Also, Sakhri and colleagues (Sakhri et al., 2010) studied on effect of zinc phosphate and polyaniline pigments on chlorinated rubber paints to prevent corrosion on mild steel. The anticorrosion performance of coatings with presence of polyaniline emeraldine salt or zinc phosphate were compared by using salt spray in 3.5% NaCl solution immersion. The results showed that protection of polyaniline was better in comparison with zinc phosphate. The time to have the first rust spot in 3.5% NaCl solution of coating with 1.5 wt% polyaniline reached 960 hours, which was about six times higher than coating without polyaniline.

Tinuvin 292 is a liquid hindered amine light stabilizer that is a pure mixture of Bis (1, 2, 2, 6, 6-pentamethyl-4-piperidyl) sebacate and Methyl 1, 2, 2, 6, 6-pentamethyl-4-piperidyl sebacate. Tinuvin 292 has been used in coatings materials to extend lifetime and protect them from UV rays (Selli, Duman, & Yagyemez, 2021; Shenoy & Marathe, 2007).

As mentioned before, many works have been done on chlorinated rubber paint for enhancing its properties but hardly any papers published on improving degradation of this varnish. In this article chlorinated rubber-based varnish coating was investigated on the effect of Tinuvin 292 to its degradation under UV-thermo-humidity condition. Changing in coating's surfaces, physical properties, gloss and resistance in thermal oxidation of coatings were also investigated.

2. Materials and Methods

2.1 Chemicals

Binders: (1) Chlorinated rubber resin, Pergut 20 (Germany) with its specifications as Viscosity at 23°C, 20% in xylene 16-24 mPa.s; Appearance: White or pale-yellow loose and soft powders; Chlorine content (Cl) > 64; Ash content □ 0.3%; Water content □ 0.5%. (2) Acrylic resin, Degalan 709 (Germany) with its specifications as Solid content: 40%; Flash point: 23°C; Glass transition temperature of 49°C.

Solvents: Xylene, acetone, butyl acetate: Industrial products (China).

Anti-UV additive: Tinuvin 292 was a product of Kremer Pigmente GmbH & Co. KG (Germany).

2.2 Vanish preparation

Raw materials were prepared as in Table 1. Chlorinated rubber resin and acrylic resin were dissolved in xylene, then acetone, butyl acetate was added. Varnish mixture was stirred well in about 10 minutes at the speed of 100 rounds per minute. Filtering - canning: a 100 hole/mm^2 mesh was used to remove impurities in the varnish, then switch to canning.

Table 1. Composition of varnish.

No.	Components	Content (wt%)
1	Chlorinated rubber resin	40
2	Acrylic resin	5
3	Xylene	30
4	Acetone	12-14
5	Butyl acetate	10.5
6	Tinuvin 292	0-2.5

2.3 Sample preparation

Physical properties measurement

Samples were prepared on steel panels (in accordance with ISO 1514:2016). Varnish coatings were painted on the 15x 10x 0.2 cm cleaned steel panels by a sprayer with pressure of 4 kg/cm^2. The steel panels were abraded with 400-grade abrasive papers and cleaned with distilled water and ethanol and then dried before covering.

These coatings were prepared and kept for 7 days at temperature of (25±2) °C and humidity of (50±5) % before testing. A minitest 600 Erichsen digital meter was used to measure the dried coating thickness.

2.4 Analysis methods

UV-thermo-humidity complex stability was tested according to ASTM D4587-11 under the UV light of UVB-313 fluorescent lamps in chamber Atlas UVCON UC-327-2. The aging cycle included 8 hours of UV light at 60°C and 4 hours at 50°C in dark water condensation. Chemical changes of coatings were analyzed by Fourier-transform infrared spectroscopy (FT-IR), FT-IR spectrometer Nicolet 8700. Morphology of coating surfaces were determined by SEM images, which were recorded by FESEM Hitachi S4800 machine. Relative hardness, Impact resistance, Flexural strength, and adhesion of coating were determined in accordance with ISO 1522:2006, ISO 6272-1:2011, ISO 1519:2011 and ISO 2409:2013, respectively. Gloss of coating was determined in accordance with ISO 2813:2014 at an angle of 60 degrees.

Thermal oxidation resistance was carried out in air condition by Thermogravimetric analysis (TGA) on NETZSCH TG 209F1 LIBRA machine with temperature raising rate of 10°C/minute from room temperature to 600°C.

3. Results and Discussion

3.1 Effect of Tinuvin 292 content on

properties tested coating under UV-thermo-humidity complex condition

Effect of Tinuvin 292 content on physical properties and gloss of aged coatings:

Samples with thickness of 30-40 µm were formulated with Tinuvin 292 contents of 0; 1.0; 2.0; 2.5 and 3.0 named as S0, S1, S2, S3 and S4, respectively were tested with 100 cycles UV-thermo-humidity complex. Results were shown in Table 2 and Table 3.

Table 2. Gloss and physical properties of initial coating.

Samples	Gloss at 60°	Physical properties of coating			
		Adhesion (Points)	**Flexural strength (mm)**	**Impact resistance (kg.cm)**	**Relative hardness**
S0	81	1	2	200	0.59
S1	81	1	2	200	0.59
S2	81	1	2	200	0.59
S3	80	1	2	200	0.59
S4	80	1	2	200	0.59

Table 3. Gloss and physical properties of aged coating.

Samples	Gloss at 60°	Physical properties of coating			
		Adhesion (Points)	**Flexural strength (mm)**	**Impact resistance (kg.cm)**	**Relative hardness**
S0	43	5	6	80	0.71
S1	49	4	5	140	0.68
S2	61	2	3	180	0.64
S3	76	1	2	200	0.60
S4	77	1	2	200	0.60

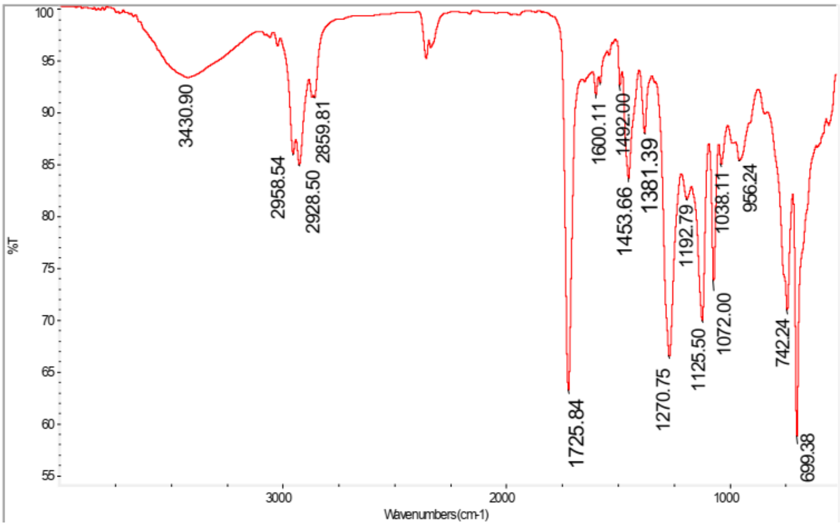
Table 2 showed that physical properties and gloss of coating were not affected by investigated Tivunin 292 contents. Table 3 showed that after 100 cycles of UV-thermo-humidity complex testing, content of Tivunin 292 increased, adhesion, flexural strength, impact resistance, gloss loss of coatings reduced or changed less. After testing, impact resistance of S0 (sample without Tinuvin 292) decreased sharply, from 200 kg.cm to 80 kg.cm and relative hardness increased from 0.59 to 0.71 or flexural strength increased to 6 mm from 2 mm. It can conclude that coating became more brittle and lost its adhesion. Meanwhile, in S3 (sample with 2.5 wt% of Tinuvin 292) or S4 (sample with 3 wt% of Tinuvin 292) the adhesion, flexural strength, impact resistance and gloss loss of coating changed slightly. results also showed that in Tinuvin 292 content more than 2.5 wt%, the relative hardness did not increase. This can be explained that Tinuvin 292 had worked as an anti-UV additive which prevented polymer chains from breaking down and cutting, so polymer aging by UV rays was limited (Kotnarowska, 2018; Selli et al., 2021; Shenoy & Marathe, 2007). Hence, with Tinuvin 292, physical properties and gloss of coating would be improved in comparison to those of coating without Tinuvin 292 after 100 cycles of UV-thermo-humidity complex testing (Nikafshar et al., 2017; Rus, Kemp, & Clark, 2008). Results also showed that Tinuvin 292 with content of 2.5 wt% and above, did not change the properties of coating after 100 UV-thermo-humidity complex cycles testing. It showed that S3 was the most suitable sample and therefore was chosen for further study.

3.2 Fourier-transform infrared spectroscopy (FT-IR) analysis

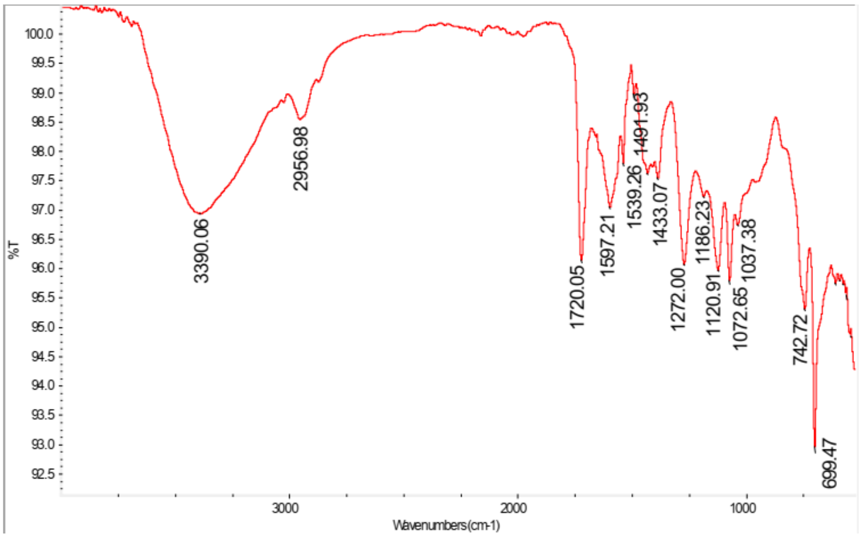
Generally, degradation of polymers may not be seen with their appearances but by chemical changes of their functional groups. In this research, FT-IR spectroscopy was used to determine chemical changes of coatings' functional groups before and after aging with 100 cycles of UV-thermo-humidity complex. S0 and S3 were chosen for testing. Results were shown in Figure 1 and Table 4.

Table 4. Selected measured IR bands of coating.

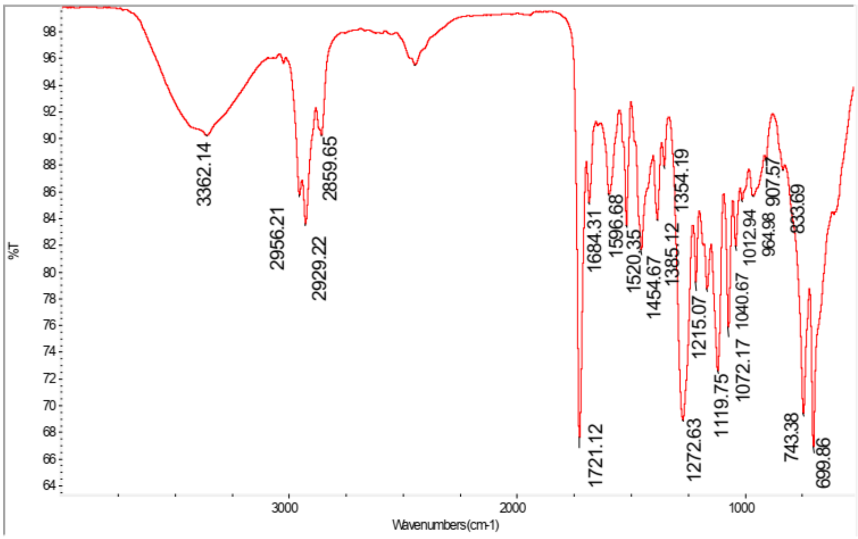
No.	Typical spectrum	Wavenumbers (cm^{-1})
1	νCH, stretch	3430
		3390
		3362
		3360
2	vas(CH$_{2=}$), asymmetry in vinyl group	2958
		2956
3	vas(CH$_{2=}$), asymmetry in carbon chain	2929
		2928
		2859
4		1725
	ν (CO) of fat acid	1721
		1720
5	ν (C=C) in vinyl group	1600
		1599
		1597
		1596
6	νC–O stretch, vibrations	1125
		1120
		1119
7	δ(CH), oscillation deformation of CH in aromatic ring	743
		742



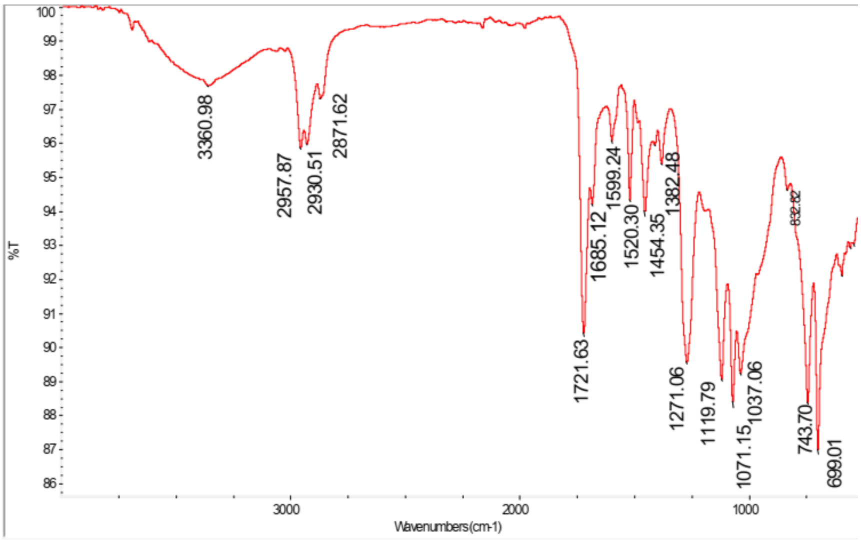
(a)



(b)



(c)



(d)

Figure 1. IR of initial S0 (a), aged S0 (b), initial S3 (c) and aged S3 (d).

Figures 1a and 1b showed (Moore, 2017) that peak intensity of CH_2 (2958) was strongly decreased, peaks 2928 and 2859 in Figure 1a could not be seen in Figure 1b. This might be explained that polymer molecular chains had been broken leading to the shorter molecular length. At that time, ester group intensities peaks (1725 of C=O or 1125 of C-O) fell off and ester bond was destructed to formulate new bonds of ketones as optical oxidation or C=O and C-O groups were stretched and shaked (Nikafshar et al., 2017). As shown in Figures 1c and 1d, there was little difference between initial and aged samples' intensities. These figures showed that intensities of CH_2 (2956, 2929, 2859) or ester groups (1721 of C=O and 1119 of C-O) of Tinuvin 292 aged coating was also stretched but vibrations or intensities of these peaks decreased insignificantly in comparison to the initial coating. The changes in intensities of ester groups or methylene after UV-thermo-humidity complex cycles testing was the same with reported by Thanh (2022a) or for 168 h for exposure to fluorescent UV radiation, heat, and water simulating weathering effects by Kızılkonca and Erim (2019).

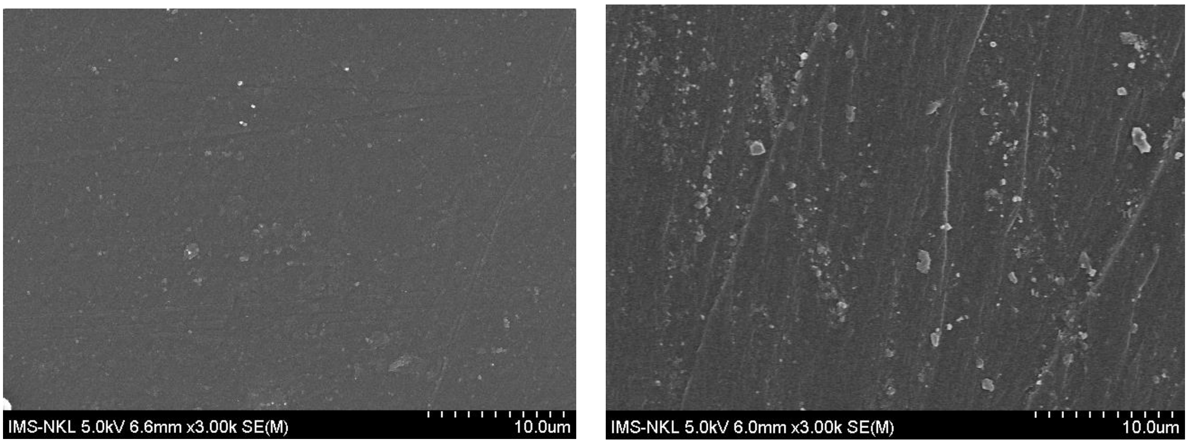
Besides that, degradation of polymers under UV rays condition may occur due to chain cutting or disproportionate macroradicals and bivalent bonds terminated (Kotnarowska, 2018; Nikafshar et al., 2017; Rus et al., 2008). Coating with Tinuvin 292 in the (S3) condition had very little difference with initial and aged samples, macroradicals occurred to the Tinuvin 292 conjugated devalent bonds. Tinuvin

292 played as an UV rays absorbent which led to reduce the varnish coating degradation (Kozak, 2015; Moore, 2017; Rus et al., 2008; Thanh, 2022b; Yu, Li, Zhong, & Xu, 2004).

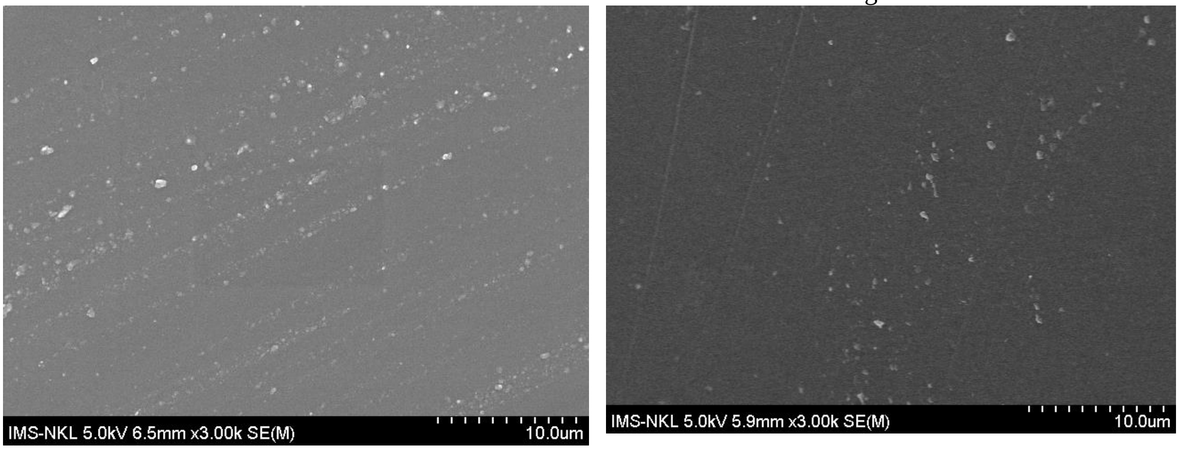
3.3 Morphology varnish coatings' surface

To investigate the effects of UV-thermo-humidity complex on coatings' surface, S0 and S3 were carried out with 100 UV-thermo-humidity complex cycles. Results were shown in Figure 2.

Figure 2 showed that after testing 100 UV-thermo-humidity complex cycles, the surface of the S3 sample was almost unchanged, there were no cracks, blisters or peeling on it. Meanwhile, in comparison with the initial coating surface, the aged surface of S0 was rougher and pinholes were observed on the sample surface. It can be explained that the oxidation process occurred and the surface of the sample destroyed by UV rays to generate micro-cavities and micro-holes on it (Queant, Blanchet, Landry, & Schorr, 2018; Yu et al., 2004). The pinholes also happened on the coating's surface of acrylic without TiO_2, ZnO under 800-hour aging in a UV/condensation weathering chamber (Nguyen et al., 2017). Those results showed that 2.5 wt% of Tinuvin 292 could protect chlorinated rubber varnish coating for UV-thermo-humidity complex 100 cycles.



Initial S0 Aged S0



Initial S3 Aged S3

Figure 2. SEM of initial and aged coatings' surfaces.

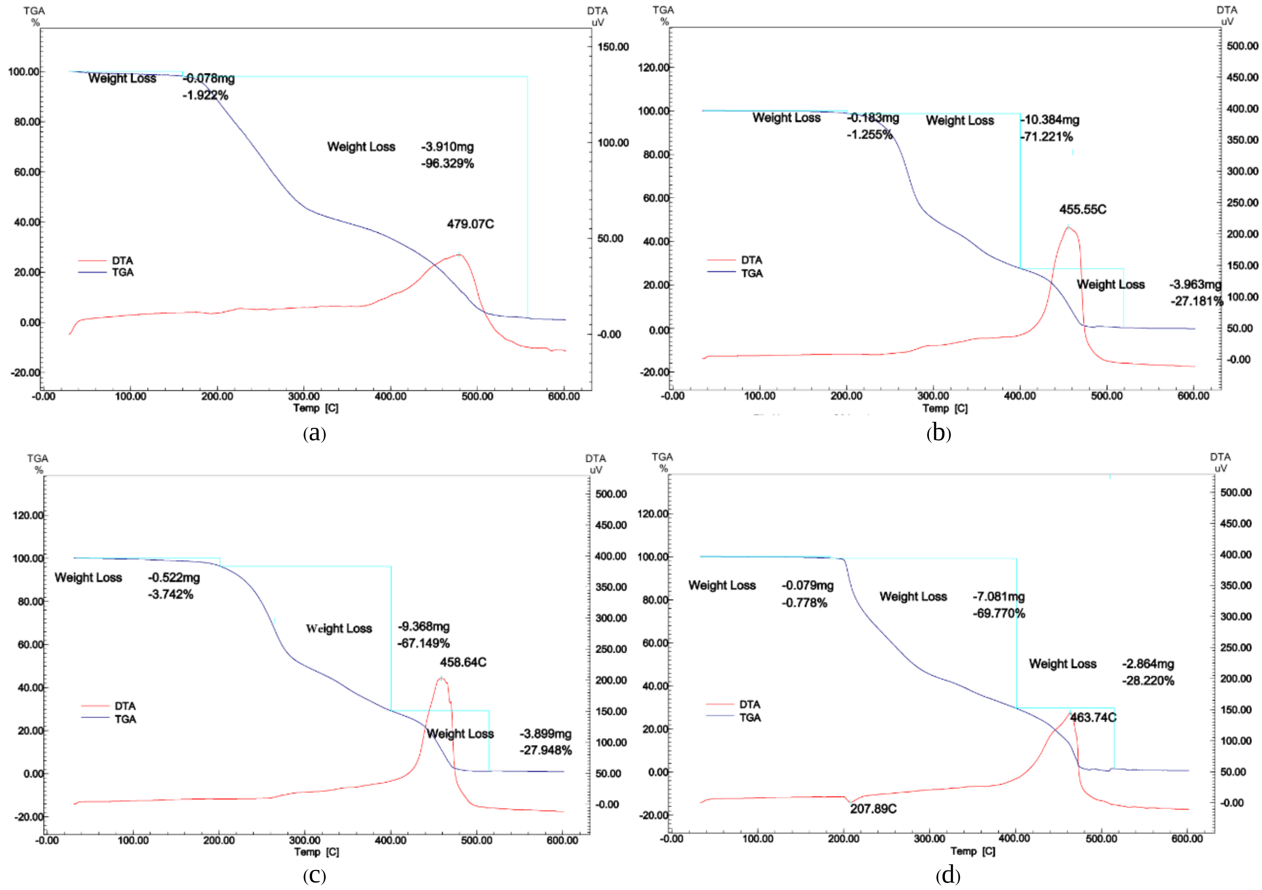
3.4 Thermal oxidation stability of varnish coating with and without Tinuvin 292

Thermogravimetric analysis (TGA) was used to study effects of Tinuvin 292 on coatings' thermal oxidation stability. S0 and S3 of before and after testing were conducted on TGA. Results were shown in Figure 3 and Table 5.

Table 5 and Figure 3 showed the different curve slopes of different samples. Weight loss of samples was also different at different temperatures. Results showed that Tinuvin 292 had significantly improved thermostability of coating, too. TGA curve slope of aged S0 was the highest (Kotnarowska, 2018; Queant et al., 2018; Talbert, 2007; Thanh, 2020). It might be explained that under 200ºC, decomposition mainly took place to low molecular matters or remaining solvents in coatings. at this period the decomposition volume was of 2-4% At this temperature, decomposition volume was a bout 2-6% of epoxy cured with toluene diisocyanate (Thanh, 2022b). Up to 400ºC, the decomposition happened with low molecular substances or residual functional groups in polymer branches. The decomposition of initial S0 and initial S3 was the same. Meanwhile, aged S0 was decomposed higher than aged S3 and strongest of all. This can be explained that after aging, polymer chains S0 had been broken sharply and S3 had lower decomposition than S0 because Tinuvin 292 had worked as an anti-oxidation agent to protect polymer chains from breaking (Hu, Li, Gao, & Zhao, 2009; Moore, 2017; Yu et al., 2004). Besides, polymer chemical bonds had been protected from breaking with Tinuvin 292 and reducing incisions in polymer chains which would prevent oxygen permeation into coating. Thus thermal oxidation stability of coating had been enhanced. at 500ºC and above organic components had been combusted or broken down, meanwhile, non-degradable parts of samples were equivalent, so the decomposition of samples was not much different at that temperature (Queant et al., 2018; Thanh, 2022b; Yousif & Haddad, 2013).

Table 5. Effect of Tinuvin 292 on thermal oxidation stability of coatings.

Samples	**Weight loss (%)**		
	300ºC	**400ºC**	**500ºC**
Initial S0	53.24	65.17	96.33
Aged S0	59.68	69.85	98.4
Initial S3	49.85	66.73	95.10
Aged S3	54.15	67.95	97.99



(a) (b)

(c) (d)

Figure 3. TGA of initial S0 (a), aged S0 (b), initial S3 (c) and aged S3 (d).

4. Conclusions

In the range of 3 wt%, Tinuvin 292 content caused increasing the adhesion, flexural strength, impact resistance, relative hardness, gloss loss of chlorinated rubber varnish coating that would protect better under 100 UV-thermo-humidity complex cycles testing condition. The suitable content of Tinuvin 292 for chlorinated rubber coating is 2.5 wt%. Chlorinated rubber varnish coating with 2.5 wt% of Tinuvin 292 could suffer from 100 UV-thermo-humidity complex cycles without cracks, blisters or surface changing. Tinuvin 292 has improved thermal oxidation resistance of varnish coating. at 300°C, decomposition of aged sample with and without Tinuvin 292 were 54.15 and 59.68, respectively.

Conflict of Interest

The authors declare that there is no conflict of interest regarding the publication of this article.

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