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Testing of Drinking Water Reservoirs Coating

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Abstract

The present study refers to a ready-made three components epoxy based paint made by the Modern Paints Industries Company (Al-Za'farania, Baghdad) subjected to several tests in order to improve its specifications by optimizing the application conditions. The paint components are under the trade names: Resin (D-5547), Hardener (H-1457) and Thinner (P-851). The paint is used for painting drinking water reservoirs from inside.

The main tests used in this study, for optimizing and comparing between formulations and conditions, is the resistance to water absorption and the hardness of the coated film.

Keywords: epoxy, coating, food grade coatings.

Introduction

Organic materials have been used in drinking water pipes and storage tanks for several years. During the last decade plastic materials are used for bottles and containers for water. Leaching of organic matter from the materials to the drinking water is proven. Water in contact with plastic pipes, surface coatings or other materials can be affected by migration of components that make the water quality unacceptable with respect to aesthetic effects or health [1].

Several complaints on drinking water quality on offshore installations are related to volatile organic matter (VOC) leaching from epoxy lining or paint. Typical VOCs from epoxy are alkyl benzenes and alkoxy compounds. In particular leaching of hydrocarbons from epoxy coated storage tanks offshore and on ships seems to be a problem, with a solvent (xylene/ethylbenzene) content up to 60-70 µg/l being measured. C3- and C4 cyclohexanes and other alicyclic hydrocarbons have also been identified as taste and odor compounds leaching from surface coatings. In a treated water basin for municipal water supply, organo-sulphide compounds were identified as the source for taste and odor, and the most likely source of the sulphides produced was microbiological activity due to degradation of organic compounds leaching from the epoxy lining [1].

Bisphenol A is the main compound used in the manufacture of epoxy resins based paints. They are widely used in the food industry and water management (drinking water reservoirs) due to its high quality instead of its higher price. These paints are based on bisphenol A diglycidyl ether (DGEBA), the monomer formed from epichlorohydrin and bisphenol A [2].

Although bisphenol A has weak toxicity to aquatic organisms, its estrogenic activity is well known since many years and it is probably one of the most studied endocrine disruptors. However, less information is available on other compounds present in the formulations of epoxy paints that could also have endocrine disruptor properties [2].

Experimental Work

Epoxy paint of three ready-made components was used. These components are: the resin D-5547 (referred by D), the hardener H-1457 (referred by H) and the thinner P-851 (referred by P).

Several problems were detected in the application of the above product when immersed in water (rusting) or by aging painted exterior surfaces (chalking). The target from the experiments in this part is to find the better conditions for the application of this three-component product. Therefore, the first set of experiments is focused on variation the most important factors which are the post curing of the coated film is depending on the curing schedules and thinner concentration. After finding the best thinner ratio, second set of experiments had been carried out by varying the hardener concentration.

Each formulation's sample had been prepared and then coated manually (by brush) in a single buildup, on a prepared metal and glass flat plates (1 x 3in.). Coated films are then cured utilizing one of two curing schedules a and b:

a. At room temperature (30°C) for 7 days.
b. At room temperature (30°C) for 6 hrs., then at 100°C for 1 hr.

Tests were carried out directly after the curing schedules. These tests included; the visual inspection, measuring the thickness, measuring the hardness and immersion in water to find the water absorption resistance, of the coated film.

Surface preparation

For all epoxy coatings, the best performance results are obtained by blasting metal surfaces to near-white or white metal. These surface finishes can be achieved by using commonly available abrasives such as sand, steel grit, aluminum oxide, garnet, etc. The most common blast grade specified is a near-white surface finish, which allows very light shadows or slight discoloration of the metal [3,4].

Thinner mixing ratio

Table 1 indicates the four suggestions for the thinner mixing ratio, with fixed resin to hardener ratio according to the data sheet of product.

Table 1 Formulations of the first set of experiments

Sample	*D, ppv*	*H, ppv*	*P, ppv*
1	10	4	2
2	10	4	4
3	10	4	6
4	10	4	8

D = Resin component D-5547, H = Hardener component H-1457
P = Thinner component P-851, ppv = parts per volume

Hardness test

Hardness of the coated film had been measured using Vickers micro hardness method according to ASTM E384 and utilizing a micro hardness tester (Micromet, Adolph I. Buehler Inc., USA) for each sample.

The micro hardness tester consists of a testing machine that supports the specimen and permits the indenter and specimen to be brought into contact gradually and smoothly under a predetermined load. No rocking or lateral movement of the indenter or specimen is permitted while the load is being applied or removed. A measuring microscope mounted on the machine in such a manner that the indentation may be readily located in the field of view.

The Vickers hardness numbers are given in special tables [5] for test loads (gf) and diagonal impression (µm). In this study the hardness values are obtained directly from these special tables.

Table 2 Hardness test results for the first set of experiments

Sample	*Thinner to resin ratio*	*HV**	*Film thickness, mm*
1_a	2 : 10	18.2_{50}	0.27~0.40
1_b	2 : 10	20.5_{50}	0.26~0.37
2_a	4 : 10	18.0_{50}	0.25~0.35
2_b	4 : 10	19.9_{50}	0.20~0.35
3_a	6 : 10	19.0_{50}	0.15~0.19
3_b	6 : 10	19.6_{50}	0.13~0.19
4_a	8 : 10	18.0_{50}	0.08~0.11
4_b	8 : 10	19.5_{50}	0.07~0.11

* HV: Vickers hardness and the subscript number referring to the test load in (g_f)

Water absorption resistance

The samples were tested for 2 hours by immersion in boiling water, according to ASTM D 570. The test specimen was in the form of sheet, 76.2 mm (3 in.) long by 25.4 mm (1 in.) wide. Coated film sheets were applied on glass plates, in a single buildup. After curing by utilizing one of the curing schedules (a) or (b), this film sheets were removed from the glass plates by the aid of a special release agent (polyvinyl alcohol 5% in water) had been previously coated and dried on the glass plates.

The specimens placed in a container of boiling distilled water, supported on edges and had been entirely immersed. At end of 120 ± 4 min, the specimens removed from water and cooled in distilled water maintained at room temperature. After 15 ± 1 min, the specimen removed from water, one at a time, all water on the surface removed with a dry cloth, and the specimens weighed to the nearest 0.000001 g immediately.

Table 3 Immersion test for the first set of experiments

Sample	*Thinner to resin ratio*	*Sample weight*, g*		*Change in weight,%*	*Visual notes ∮*
		Initial..	*Wet..*		
1_a	2 : 10	0.685654	0.675714	- 1.45	OK
1_b	2 : 10	0.546228	0.555241	+ 1.65	OK
2_a	4 : 10	0.721056	0.709685	- 1.58	OK
2_b	4 : 10	0.593129	0.599120	+ 1.01	OK
3_a	6 : 10	0.375558	0.380654	- 1.35	1L,1B
3_b	6 : 10	0.422448	0.426326	+ 0.92	1L,1B
4_a	8 : 10	0.261391	0.258801	- 0.99	2B
4_b	8 : 10	0.162339	0.167570	+ 3.22	2B

* *Sample weight* : Measured before and after 2 hours immersion in boiling water (ASTM D 570).

∮ *Visual notes after immersion:* 1. Slight, 2. Moderate, 3. Severe. B. Blistering, T. Tacky, C. Coloration, D. Dissolving, L. Lifting, OK. Unchanged, R. Rusting, S. Softening.

Hardener mixing ratio

Another set of experiments were carried out to get a better formulation by varying the hardener concentration (H) ratio. Table 4 indicates formulation's samples of the second set.

As it's indicated in this table, a constant thinner concentration (P) had been used in the second set of experiments. Sample no.1 had been used as a reference for this set.

Using the same procedure of the first set, samples in the second set had prepared and tested. Results are listed in Table 5 for hardness test and in Table 6 for water absorption resistance test.

Table 4 Formulations of the second set of experiments

Sample	*D, ppv*	*H, ppv*	*P, ppv*
5	10	3.4	2
6	10	3.6	2
7	10	3.8	2
8	10	4.2	2
1	10	4.0	2

D = Resin component D-5547, H = Hardener component H-1457
P = Thinner component P-851, ppv = parts per volume

Table 5 Hardness test results for the second set of experiments

Sample	*Hardener to Resin Ratio*	*HV**	*Film Thickness, mm*
5_a	3.4 : 10	18.0_{50}	0.28~0.39
5_b	3.4 : 10	20.0_{50}	0.25~0.35
6_a	3.6 : 10	18.5_{50}	0.28~0.41
6_b	3.6 : 10	20.5_{50}	0.28~0.39
7_a	3.8 : 10	18.5_{50}	0.32~0.45
7_b	3.8 : 10	19.0_{50}	0.28~0.38
1_a	4.0 : 10	18.2_{50}	0.27~0.40
1_b	4.0 : 10	20.5_{50}	0.26~0.37
8_a	4.2 : 10	18.0_{50}	0.27~0.42
8_b	4.2 : 10	20.1_{50}	0.26~0.32

* HV: Vickers hardness, and the subscript number referring to the test load in (gf).

Table 3 Immersion test for the second set of experiments

Sample	*Hardener to resin ratio*	*Sample weight*, g*		*Change in weight,%*	*Visual notes*[§]
		Initial..	*Wet..*		
5_a	3.4 : 10	0.325187	0.321131	- 1.25	OK
5_b	3.4 : 10	0.322925	0.330510	+ 2.35	1B
6_a	3.6 : 10	0.316066	0.313879	- 0.69	OK
6_b	3.6 : 10	0.383267	0.392034	+ 2.29	1B
7_a	3.8 : 10	0.254371	0.251740	- 1.03	OK
7_b	3.8 : 10	0.244318	0.250618	+ 2.58	OK
1_a	4.0 : 10	0.685654	0.675714	- 1.45	OK
1_b	4.0 : 10	0.546228	0.555241	+ 1.65	OK
8_a	4.2 : 10	0.422025	0.416732	- 1.25	OK
8_b	4.2 : 10	0.407759	0.417649	+ 2.43	2B

* *Sample weight* : Measured before and after 2 hours immersion in boiling water (ASTM D 570).

[§] *Visual notes after immersion:* 1. Slight, 2. Moderate, 3. Severe. B. Blistering, T. Tacky, C. Discoloration, D. Dissolving, L. Lifting, OK. Unchanged, R. Rusting, S. Softening.

Volatile organic matter

Four samples having the same formulations of sample no.1 (Table 1) were cured by the schedule (a), and then cured by schedule (b). Samples weighing at several temperatures had been carried out. Table 7 shows the results of this experiment.

Another experiment carried out to find how much volatile materials (solvents) are incorporated with the original product components: the Resin D-5547 and the Hardener H-1457. Table 8 shows the results for this experiment.

Total loss in weight by heating the three components product, were calculated in Table 9.

An immersion test (2 hrs. boiling distilled water) had been carried out for the four samples above. Results are listed in Table 10.

Long term immersion

Here a final long-term immersion test had carried out with formulation's sample no.1. This test had been done according to ASTM D570, by using similar procedure to the procedure mentioned above. Results of the long-term distilled water immersion test are listed in Table 11.

Table 7 Weight loss during post curing

Sample	*Schedule a sample weight in grams (at 30°C)*							
	Initially	1 day	2 days	3 days	4 days	5 days	6 days	1 week
9	0.574094	0.441807	0.438911	0.437424	0.436524	0.435920	0.435401	0.435056
10	0.523162	0.399040	0.395848	0.395708	0.394948	0.394609	0.394176	0.393852
Mean wt.	0.548628	0.418828	0.417380	0.416566	0.415736	0.415265	0.414789	0.414454
Lost wt.	0%	23.66%	23.92%	24.07%	24.22%	24.31%	24.40%	24.46%

Sample .	*Sample weight in grams at different temperatures*					
	30°C 1 week	50°C	75°C	100°C	100°C after 1 hr	100°C after 2 hr
11	0.540754	0.538590	0.535337	0.528261	0.514656	
12	0.324457	0.321443	0.318845	0.313313	0.310251	
13	0.293812	0.290420	0.287795	0.283455		0.280698
14	0.392082	0.388747	0.384678	0.379728		0.372608
Mean wt.	0.387776	0.387800	0.381664	0.376189		
Lost wt.	0%	0.77%	1.58%	2.99%	4.66%	4.75%
Total lost wt.	24.46%	25.23%	26.04%	27.45%	29.12%	29.21%

All samples above having the same formulation of sample no. 1. Heating rate between 30°C to 100°C was 6°C/min. Coated films thickness were varying between 0.17mm to 0.21 mm

Table 8 Weight loss during heating for the product components

Component	Initial wt. g	After 1hr. at 100°C		After 2hrs. at 100°C	
		Wt. g	Wt. loss	Wt.g	Wt.loss
Resin D-5547	8.46	7.03	16.9 %	6.70	20.8 %
Hardener H-1457	5.13	3.05	40.5 %	2.91	43.3 %
Thinner P-851	9.00	0	100 %	0	100 %

Table 9 Total weight loss during heating the product components

Component	ppv	Density g/ml	ppw	Wt %	Lost wt.% after	
					1 hr	2 hr
Resin D-5547	10	1.41	14.1	73.5	16.9	20.8
Hardener H-1457	4	0.86	3.44	17.9	40.5	43.3
Thinner P-851	2	0.82	1.64	8.6	100	100
Total wt. loss for the 3 components after heating to 100°C					28.3	31.6

Table 10 Immersion test results for the third set of experiments

No.	Curing schedule	Sample weight, g		weight increase %
		Initial	Wet	
11	1-week 30°C, 1hr. 100°C	0.514656	0.526451	+ 2.29
12	1-week 30°C, 1hr. 100°C	0.310251	0.318748	+ 2.74
13	1-week 30°C, 2hrs. 100°C	0.280698	0.289188	+ 3.02
14	1-week 30°C, 2hrs. 100°C	0.372608	0.383866	+ 3.02

Table 11 Long-term immersion test results

Time, day	Formulation no.1_a		Formulation no.1_b	
	Sample weight, g	Increase in weight, %	Sample weight, g	Increase in weight, %
0	0.486221	0	0.425918	0
1	0.486377	0.03	0.426293	0.09
7	0.487787	0.32	0.427912	0.47
14	0.488079	0.38	0.428338	0.57
28	0.488127	0.39	0.428721	0.66
42	0.488419	0.45	0.429232	0.78
56	0.488905	0.55	0.429828	0.92
70	0.488954	0.56	0.429956	0.95
84	0.489343	0.64	0.430127	0.99
98	0.489392	0.65	0.430212	1.01

- Immersion in distilled water at 28°C (ASTM D570).
- All samples were unaffected at end of the test.

Induction time

UV light long-term exposure test had been carried out with normal atmospheric conditions and by varying the induction time to find out if there is any effect of this property (induction time) on chalking of the coated film surface. Table 12 shows the results of this test. This test had been carried out by exposure of the samples to direct sunlight (~11 hrs. per day) at ~40oC.

Finally, Table 13 gives a summary of the final formulation specifications.

Pot life had been measured by using a rotary viscometer (Haake, Germany). Pot life generally ends at a viscosity of about 5000 centipoises [6]. Spread rate and volume mixing ratio values were cited from product data sheet.

Table 12 Exposure to uv light test at ~40°C

Sample	Induction period	Notes after exposure time..			
		1 month	5 weeks	6 weeks	7 weeks
1_a	0 minutes	OK	OK	LC	SC
1_b	0 minutes	OK	OK	LC	LC
1_a	5 minutes	OK	OK	OK	LC
1_b	5 minutes	OK	OK	OK	LC
1_a	½ hour	OK	OK	OK	OK
1_b	½ hour	OK	OK	OK	OK
1_a	1 hour	OK	OK	OK	OK
1_b	1 hour	OK	OK	OK	OK

OK: not affected, LC: chalking appears, SC: sever chalking.

Table 13 Specifications of the selected formulation (no.1)

Components	Concentration, ppv
Resin D-5547	10
Hardener H-1457	4
Thinner P-851	2
Specifications	Value
Pot life, 200g at 30°C, hours	8
Hardness, Vickers;	
Curing: 7 days (30°C)	18.2_{50}
Curing: 1 hr. (100°C)	20.5_{50}
Distilled water absorption, %	
After 2 hrs. boiling	
Curing: 7 days (30°C)	- 1.45
Curing: 1 hr. (100°C)	+ 1.65
Curing: 7 days (30°C), 1 hr. (100°C)	+ 2.52
Curing: 7 days (30°C), 2 hrs. (100°C)	+ 3.02
After 14 weeks at 28°C	
Curing: 7 days (30°C)	+ 0.65
Curing: 1 hr. (100°C)	+ 1.01
Spread rate, m²/l at 0.035 mm thickness:	
Concrete surfaces	4
Metal surfaces	7
Specific gravity at 30°C:	
Resin D-5547	1.41
Hardener H-1457	0.86
Thinner P-851	0.82
Volume Mixing ratio (Resin : Hardener)	10:4

Results and Discussion

Effect of thinner mixing ratio

By varying the thinner mixing ratio, four samples (Table 1) were prepared and tested. Hardness test results (Table 2) had plotted in Fig. 1. Results of the immersion test (two hours in boiling distilled water) as listed in Table 3 had plotted in Fig. 2.

The results of the first set of experiments above indicate the following:

1. From hardness test results, it is noted that increasing the thinner mixing ratio will results in lowering the

hardness value of the coated films after utilizing same curing schedule.

2. Better hardness values had been obtained by utilizing curing schedule (b), rather than curing schedule (a).
3. Cured coated film thickness after utilizing schedule (a) were always more than thickness after utilizing schedule (b).
4. Immersion test improves that reducing of thinner mixing ratio produces better water absorption resistance, especially for samples cured by utilizing schedule (b).
5. Samples cured utilizing curing schedule (a) had an unexpected phenomena, its weight reduces after immersion in boiled water.

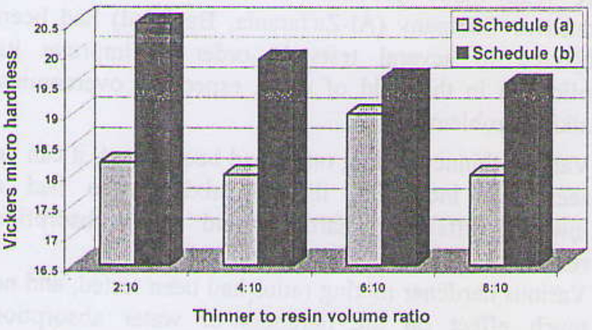

Fig. 1 Hardness results for the first set of experiments

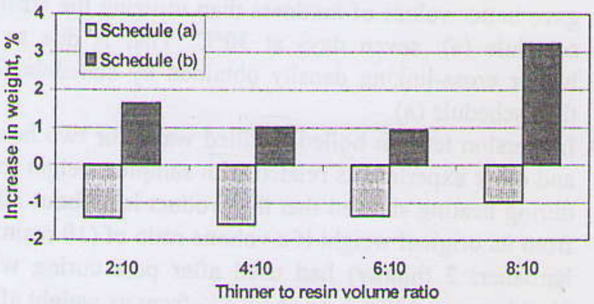

Fig. 2 Immersion test results for the first set of experiments (2 hr boiling in distilled water)

Effect of hardener mixing ratio

In this set of experiments, various hardener volume mixing-ratios were used (Table 4). Hardness test results (Table 5) had plotted in Fig. 3, while immersion test results (Table 6) had plotted in Fig. 4. It can be found that:

1. Hardener concentration, in the range of above experiments, had no much effect on the hardness or water absorption resistance of the cured coated films.
2. Utilizing curing schedule (b) gives better results in hardness than utilizing curing schedule (a).
3. Coated film thickness by utilizing curing schedule (a) were always more than the thickness by utilizing curing schedule (b).
4. Curing by schedule (a) still produces the same reducing effect on the weight of the cured coated film after immersion in boiled water for 2 hours.

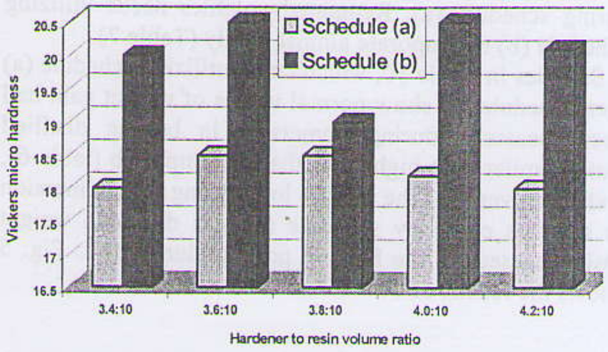

Fig. 3 Hardness results for the second set of experiments

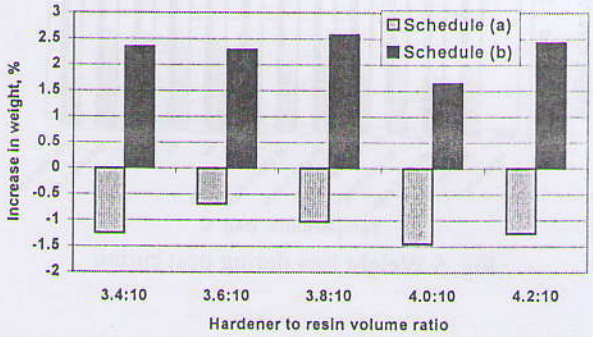

Fig. 2 Immersion test results for the second set of experiments (2 hr boiling in distilled water)

Volatile component matter

The reason behind the weight loss in the cured coated films that utilize schedule (a) after two hours immersion in boiled water. As it's seen in the first and second sets of experiments, varying of thinner and hardener mixing ratios had no much effect on volatile organic matter. Therefore another experiments were done to find the effect of post cure schedule on the weight of the coated film samples.

Table 7 indicates that the coated film undergoes a weight losing (due to solvent vaporization) during and after one or two hours of heating to 100oC, even after utilizing schedule (a).

Tables 8 and 9 show the quantity of volatile materials (solvents) that incorporated with the original components (the Resin D-5547 and the Hardener H-1457). These tables indicate that about 20.8% and 43.3% are volatile materials in the original resin and hardener compositions, respectively. Totally, 28.3 to 31.6% of the product weight is volatile materials, after incorporation of thinner in the volume mixing ratio D/H/P=10/4/2.

This high quantity of volatile materials in the product, which is chemically inert materials, evaporates to about 24.46% from the total weight of product after utilizing curing schedule (a). Remained volatiles needs utilizing schedule (b) to evaporate almost totally (Table 7).

Samples in Table 10, which cured utilizing schedule (a) then schedule (b) show normal values of weight gain had been measured during immersion in boiling distilled water similar (but higher) to that of sample 1b (table 6), and improves that the weight loss during this immersion in samples cured by schedule (a) was due to a weight losing caused by the heating process during test. Fig. 5 shows the results of Table 7 in a chart.

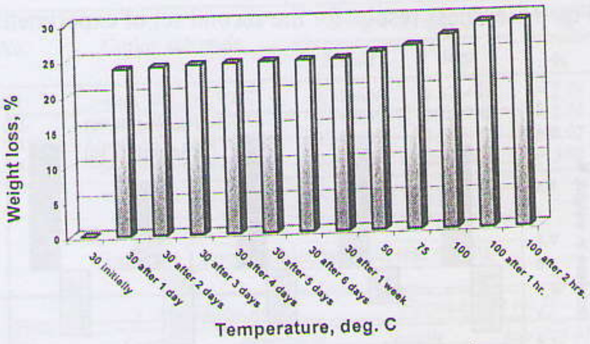

Fig. 5 Weight loss during post curing

Long-term immersion

Results of the long-term distilled water immersion test, which listed in Table 11, had been plotted in Fig. 6. These results show normal behavior for the coated film during immersion. Utilizing curing schedule (a) gives lower values of weight gain than schedule (b).

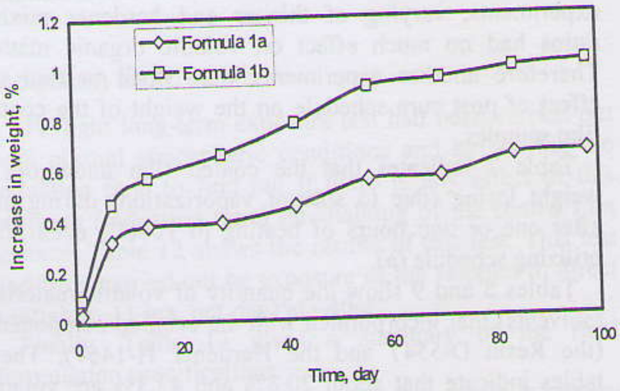

Fig. 6 Long-term immersion test (distilled water at 28°C)

Effect of induction time

UV light long-term exposure test had been carried out with normal atmospheric conditions and by varying the induction time to find out if there is any effect of this property (induction time) on chalking of the coated film surface. Table 12 shows the results of this test. Results showed that the induction time (the time between end of mixing of product components to the beginning of its application on surface) is an effective property on chalking occurness at coated film surface.

Conclusions

A ready-made product from the Modern Paints Industries Company (Al-Za'farania, Baghdad) had been subjected to several tests in order to improve its application in the field of work, especially overcoming chalking problem.

1. Various thinner mixing ratios had been tested, it can be seen that increasing thinner mixing ratio had a lowering effect on hardness and water absorption resistance.
2. Various hardener mixing ratios had been tested, and no much effect on the hardness or water absorption resistance of the cured coated films was observed within the range of the conducted experiments.
3. Utilizing the curing schedule (b): one hour at 100oC, gave better values of hardness than utilizing the curing schedule (a): seven days at 30°C. That is due to a higher cross-linking density obtained by schedule (b) than schedule (a).
4. Immersion tests in boiled distilled water for two hours and other experiments related with samples weight loss during heating showed that the product lost about 30% from its original weight if a volume ratio of (10 resin: 4 hardener: 2 thinner) had used after post curing with (1~2 hours at 100°C), or about 5% from its weight after utilizing curing schedule (a). This weight losing probably due to the evaporation of high boiling solvents.
5. Lose the unknown volatile organic matter leached from epoxy paint in boiled water immersion test (5% from total weight) had proven.
6. Long-term distilled water immersion test at 30°C indicates that samples cured utilizing schedule (a) had better resistance to water absorption than these cured utilizing schedule (b).
7. Ultra violate (UV) light exposure to samples with different induction times showed that chalking problem is caused mainly by insufficient induction time after mixing the product.

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