



# Composite Resin Coating for The Protection of Surfaces and Metals

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#### Abstract

The curing process of epoxy resin was investigated with some additives and fillers such as nano fumed silica with primary particle diameter of 56–58 nanometers, fine ferric oxide with an average particle diameter of 0.4 micrometer, fine calcium carbonate with an average particle diameter of 0.58 micrometer, and plasticizer, a new composite resin has been obtained. Based on present experimental results, the new composite resin shows high impact strength and hardness as well as the ability of the composite withstand to degradation from water way in. Additionally, curing time of composite was reduced to the minimum set time than epoxy resin at different temperature. This new composite resin was used appropriately for coating to protect surfaces and metals in marine ships from corrosion which meet the requirements for certain applications such vehicles body, crude oil tankers, petroleum pipelines services, and other applications.

**Keywords :** Epoxy resin, Additives, Hardener, Impact strength, Hardness strength, Curing time, Composite resin.

استخدام المتراكبات الراتينج في الطلاء لحماية الاسطح والمعادن هدى علي البنا<sup>1</sup>\*, نادية ابراهيم فخري<sup>1</sup>, نسرين علي حسين<sup>2</sup> <sup>1</sup> قسم الكيمياء , وزارة العلوم والتكنلوجيا ,بغداد, العراق <sup>2</sup> قسم الصناعات الكيمياوية , معهد التكنلوجيا بغداد , بغداد, العراق

#### الخلاصة

تم في هذا البحث تحوير و معالجة راتتج الايبوكسي مع بعض المواد المضافة والحشوات مثل السيليكا المتطايرة حيث يتراوح قطر الدقائق ما بين 56-58 نانوميتر , دقائق اوكسيد الحديديك بمعدل قطر 0.4 مايكروميتر , ملدن , دقائق كاربونات الكالسيوم بمعدل قطر 0.58 مايكروميتر لغرض الحصول على مادة مركبة راتنجية . ولقد أظهرت النتائج المختبرية ان المادة المركبة ذات قوة صدم و قوة صلادة عالية اضافة الى امكانيتها على مقاومة أنحلالها في الماء وكذلك تم تقليص زمن تصلب وتقسية المادة المركبة الراتنجية بمقدار زمني اقل بكثير من تقسية راتنج الايبوكسي بمختلف درجات الحرارة. وأعتمادا" للمواصفات التي تم الحصول عليها فان المادة المركبة الجديدة تم تطبيقها لاغراض طلاء الاسطح والقطع المعدنية المستخدمة في صناعة الزوارق والقطع البحرية لغرض حمايتها من التآكل , كذالك تنوع استخداماتها بما يتلائم مع بعض التطبيقات التي تدخل في طلاء الناقلات والانابيب النفطيه

## **1. Introduction**

Epoxy resins constitute a major class of thermosetting resins. However, some of the properties of the materials of epoxy resins are non-beneficial, such as the inherent brittleness due to the high cross-linking density formed in curing, which limits their many end-use applications [1]. A large number of studies have been conducted to improve the toughness of highly cross-linked epoxy resins. Epoxy resins are usually toughened by modification with liquid elastomers, thermoplastics, and inorganic particles [2,3]. The thermal and mechanical properties of epoxidized castor oilmodified epoxy resins have been studied by Park and Jin[4]. Also, the fabrication and mechanical properties of epoxidized soybean oil /epoxy composites were studied by Erhan et al, and Chandrashekhara et al. [5,6]. Gupta et al [7] have dealt with material systems that contain fibers and fillers simultaneously, whereas Srivastava et.al [8] has focused on the experimental aspects of the fly ash filled materials.

In this study, polymer-inorganic and organic various fillers, such fumed SiO<sub>2</sub> fine-CaCO<sub>3</sub>, and fine-Fe<sub>2</sub>O<sub>3</sub> have been added to epoxy resin in order to improve the properties of the material and second, to reduce the cost of component. Such attempt was not found in the literatures. Due to the large boundary surface created by the fumed fillers which can be added to the epoxy resin had a chance to have new properties. Nano Fumed Silica has unique properties and is commonly added to liquids/coatings to improve their properties where recommended for thickening and thixotropy [9,10]. Iron oxide is used as a pigment in epoxies and other coatings for anti corrosion on steel and metal surfaces [11]. Calcium Carbonate reduces the cost, saves resource and modifies physical property of resin; it obviously improves product tensile strength, and impact strength [12]. Corn oil is found to be suitable for application in polymers such as coatings, adhesives, foams, plasticizers, composite epoxy resins, and elastomers [13].

## 2. Experimental

#### **2.1Materials**

Diglycidylether of bisphenol-A (CY-223), an epoxy resin, used as 3 parts by weigh and 1 part by weigh (HY 953) diethylene triamine as a hardener manufactured by (Ciba-Giegy). The chemical structures of CY 223 and HY 953 are shown in Figure 1. Fumed silica (SiO<sub>2</sub>) with median particle diameter of 58-56 nanometers is used. The particles are non-porous which allow to be added to the thin films such as inks and coatings without affecting the gloss. Rust fine Iron oxide ( $Fe_2O_3$ ) is used as a pigment in epoxies and other coatings for anti-corrosion on metal surfaces. The fine pigment median particle diameter is 0.4 micrometer and has a bright color. Also the fine particle for easy mixing (CaCO<sub>3</sub>) has a median particle diameter of 0.58 micrometer, as shown in Figure 2. Corn oil or fatty acids had been used as plasticizer for preparing composite resin.



Diglycidylether of bisphenol- A (DGEBA)

н  $NH_2$  $H_2N$ Diethylenetriamine (DETA)

Figure 1- Chemical structures of the epoxy resin and hardener used.



Figure 2- The particle diameter measurements for SiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, CaCO<sub>3</sub>

#### 2.2 Synthesis Process

The stainless steel reactor used in this work is equipped with mechanical stirrer and speed regulator. About 69 wt% Liquid epoxy was introduced to the reactor,

2.5 wt% of fumed Silica was added slowly with continuous stirring at speed ranging 60-80 rpm. Then 9 wt% of fine Iron oxide, 19 wt% of fine Calcium Carbonate, and 0.5 wt% of Corn oil were added gradually. The mixture was stirred at slow speed to ensure good mixing and wetting all components in resin as well as to formation of large sticky mass and dead zones. The speeds of stirring were raised to 110 rpm and left it for 1 hour to reach specific density. The composite was then transferred to the moulds and was allowed to cure at room and different temperature. The composite resin mixture had an intermediate density so that the resin didn't flow out of the layup, especially on a vertical surface. It is an important to have the composite resin and the hardener as liquids at room temperature to facilitate mixing process since the amines are too reactive to be blended at elevated temperatures and to have easy cure at room temperature.

#### 2.3 Sample Preparation

Composite resin and hardener materials were mixed in a ratio of 5: 1 by weight. The designed amount was stirred and degassed in a vacuum oven to eliminate air bubbles before measuring. The bubble-free mixture was injected into a mold, placed in a convection oven and subjected to three curing phases at temperatures of 80, 120, and 150 °C respectively.

## 2.4 Characterization and Measurements

In the present study impact strength and hardness tests were carried out to study the mechanical behavior of the composite. The impact strength performed using an Izod impact tester according to ASTM D256. Specimens were cut to suitable dimensions for mechanical tests. The specimen size was 5mm×12.7mm×6.35 mm. hammer model (6545/000) is used. The hardness tests (Shore-D) pre load applications of 50 N forces were performed according to DIN 53505, ASTM D 2240, and ISO 808. Density determinations of liquid samples and displays the results directly as density, by portable density meter allow rapid measurement range of 0 to 3  $g/cm^3$  with resolution of 0.0001 g/cm<sup>3</sup>, accuracy of  $\pm$  $0.001 \text{ g/cm}^3$  and temperature range of 5-35°C. Particle diameter analyzer (SALD- 2101-WEA1: V1.20) laser diffraction by Shimadzu Company was used to measure median particle diameter.

#### 2.5 Degradation

The ability of resin to withstand degradation from water ingress was investigated by aging the newly prepared cured composite polymer in saline solution.

One gram of the sample was kept in 50 ml of normal saline solution. The weight% loss was monitored for a month with an interval of 5 days.

### **3.Results and Discussion**

The new composite resin mixture has an intermediate density appearance with red color. The reaction is low exothermic through the curing process. Results of impact and hardness tests of these materials are given in Table 1. Remarkable difference in the impact strength of the material can be shown with the addition of small quantity of additive and filler where the value of impact strength has increased by more than 200%, resulting in an improved bonding strength in the polymer composite blends so the composite resin has much higher impact strength compared to the epoxy resin. The hardness test reflects tensile strength and Young modulus. Therefore, shore hardness is used here to evaluate the mechanical property of composite resin. It was assumed that the composite resin specimens would have higher hardness compared with low cross-link density epoxy resin, as given in Table 1.

The curing of composite resin: hardener by weight ratio of 5: 1 is run for 6 hr for the first cure and 24 hr for complete cure at room temperature. Minimum curing time data are given in Table 2. The best balance of the degree of curing is necessary for good coating performance. Curing the agent with composite resin is significantly faster than liquid epoxy resin. In dry time tests at room temperature, the set time was reduced from 48 hr to 6 hr at 25°C and reduced from 10 hr to 2.3 hr at 80°C. Figure 3 shows the comparative dry times and cure temperature degree. An important property of any resin, particularly in a marine environment, is its ability to withstand degradation from water ingress. The effect of saline solution on the composite resin was evaluated, the weight% loss increases slightly with time less than 3%. Such weight% loss suggests that this sample is relatively more crossed-linked. This is reflected in the weight% loss in saline after 35 days of aging; such change is shown in Figure 4. The composite polymer resin can be used for painting. It has an intermediate density and easily brushed to wet the surface, but not run off the surface and has the ability to adhesion with other surfaces and easy to molding for different applicants. It does not have noxious volatiles and can be worked in an open situation.

 
 Table 1- Mechanical and Physical properties for resin with and without filler

Samples	Hardness test Shore D	Impact strength kJ/m <sup>2</sup>	Density gm/cm <sup>3</sup>
Composite resin specimen (1)	80	70	2.2-2.4
Composite resin specimen (2)	82	70	2.2-2.4
Epoxy resin	65	35.3	1.15

#### Table 2- The curing time

Temperature	Average	Epoxy
$c^{\circ}$	Composite	Resin Cure
-	Resin	(hr)
	Specimens	
	Cure (hr)	
25	6	48
80	2.3	10
120	1	4
150	1	2



Figure 3- The dry time as a function of cure temperature for the present new composite resin compared with pure epoxy resin.



Figure 4- Effect of saline solution on the percentage of weight loss.

#### 4. Conclusions

Addition of additive and filler of nanoscale particles of Fumed silica, fine Calcium Carbonate and fine Iron oxide in small weight fractions to low density epoxy resin led to increased impact strength and hardness considerably of the composite resin. The composite resin has a backbone that has been shown to excellent water resistance, and the ability to withstand degradation from water ingress. Curing agent with composite resin behavior is significantly faster than liquid epoxy resin where reduce the curing to the minimum set time at different temperatures. The composite resin goes through an intermediate density and made the surfaces bright.

This new composite resin have been used and applied in the field of coating marine ships to protect surfaces and metals from corrosion, it also needs to be subjected to more investigation in the field of certain applications like vehicles body, crude oil tankers, petroleum pipelines and boats, such suggested research will show the application potential of this new composite resin.

#### Acknowledgements

This paper is based on work partially supported by the marine leadership Office of Scientific Research managed by Dr. Motheher Sadeq Iraq.

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