

PREPARATION, CHARACTERIZATION AND ANTI-CORROSION PROPERTIES OF EPOXY-ORGANO-BENTONITE COMPOSITE ADDED ZnO SUBMICROMETER PARTICLES

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ABSTRACT

In this paper, an epoxy-based composite coating containing various combinations of zinc oxide submicrometer particles and organobentonite nanoparticles were prepared. Dispersion of zinc oxide particles with organobentonite nanoparticles within the composites were evaluated using XRD analyses. Hardness, adhesion, physical properties and corrosion resistance of composites were studied. The results showed that simultaneous use of low-loading fillers have a positive effect on the clay exfoliation behavior in resulting nanocomposites. Hardness, adhesion of novel composites containing zinc oxide particle slightly increased compared with neat-epoxy even though with epoxy-organobentonite composite. Corrosion performance of composites increased with addition of zinc oxide and organobentonite particles, due to improving barrier properties of the coating.

Keywords: anti-corrosion, epoxy-organobentonite composite coating, EIS, ZnO submicrometer particles.

1. INTRODUCTION

Nowadays, steel has become an important part of our life due to its extensively applications in automotive, household appliances, business machine and heavy construction such as marine and chemical industries. Low-carbon steel is selected for construction because of its mechanical properties and machine-ability at a low price [1]. It is known that when steel is exposed to a natural atmosphere or marine environment, rust is formed. Although the rusting of steel is usually termed as corrosion, the latter is a general term which is used to define the destructive interaction of a material with its environment. Corrosion usually refers to metals and causes enormous industrial losses with a depletion of our natural resources [2]. In this regard, polymeric coatings can provide protection either by a barrier action from the layer or from active corrosion inhibition supplied by pigments in the coating, which give protection to the underlying substrate [3]. However, in practice, all polymeric coatings are permeable to corrosive species too such as

oxygen, water and ions to some extent [4 - 7]. Water molecules at the steel/coating interface may reduce the coating adhesion, thus favouring corrosion of the metal underneath the film.

Attempts have been carried out to improve coating resistance against corrosive environments. Some type of pigments i.e. chromate, phosphate, micro sized metallic or metal oxides and organobentonite pigments have been utilized to improve corrosion resistance of the organic coatings [8, 9]. It has been shown that organic/inorganic pigments can significantly improve corrosion resistance of the organic coatings. Zhang et al. [10] showed that nano- TiO_2 particle can significantly improve corrosion resistance of the epoxy coating. Dhoke and Khanna [11, 12] revealed that nano sized ZnO particles can effectively improve corrosion resistance of the coatings. It pointed out that nano-ZnO is a non-toxic particle. Therefore, environmentally friendly coatings can be produced using these nanoparticles. Epoxy nanocomposites containing different contents of nano-ZnO particles were prepared by B. Ramezanzadeh, M. M Attar, the nanocomposites were exposed to 3.5 wt% NaCl solution, mechanical properties of the nanocomposites (before and after exposure to NaCl solution) were studied. Results showed that corrosion resistance of the epoxy coating was significantly improved using nanoparticles [13]. Nanoclay is also introduced into epoxy matrix and endowed epoxy/clay composite significantly improved physical and chemical properties [14, 15]. In previous studies [16 - 19], some types of epoxy-clay nanocomposite had prepared in order to produce anti-corrosive epoxy coating. Anti-corrosive properties of nanocomposite were often investigated using electrochemical impedance spectroscopy (EIS) methods. All of results showed an improvement in the barrier and anti-corrosive characteristics of new composite coating. But nano-pigments are a bit of high cost. Therefore, in the other way, some low-cost filler are considered as pigments.

In this study, it has been aimed to investigate the effects of combine organobentonite nanoparticles and ZnO sub-micrometer particles on the mechanical, physical properties and corrosion resistance of epoxy based coating on Q235 low-carbon steel substrate.

2. MATERIALS AND EXPERIMENTAL

2.1 Materials

This research has used low-cost epoxy "GCC135" - a low viscosity liquid epoxy resin. The GCC135 is a bisphenol A type epoxy resin, ethylene glycol diglycidyl ether. Appearance of GCC135 epoxy resin: transparent liquid, no mechanical impurities; epoxy value (eq/100 g): 0.54 - 0.6; viscosity (mPas) : 700 - 1100; density (g/cm^3): 1.13 - 1.17.

The W93 type has used as hardener - a low viscosity liquid hardener. The W93 is a modified isophorone amine. Appearance of W93: colorless to pale yellow liquid; amine value (KOH/g): 550 - 600 mg; viscosity (mPas): 10 - 100;

Epoxy resin and Hardener were purchased from G. C. Chem Co. Kunshan, China.

Ethanol, acetone (AR) and others reagent for preparation of artificial seawater (ASW) were purchased from Sinopharm Chemical Reagent Co. Ltd, China.

TIKOGEL MP100 organobentonite was used as 1st filler which is a high organic bentonite. The MP100 was purchased from Shunde District of Foshan City, Qinghong Trade Co. Ltd, China. Performance of TIKOGEL MP100: Appearance: cream colored free flowing powder; density: about 1.4 g/cm^3 ; bulk density: 370 - 510 g/l ; moisture content: $\leq 3\%$; 90 microns sieve

residue: <15 %; full basic state: 1 - 5 μm , thickness of organobentonite plate about 1.4 nm.

Submicron sized ZnO powder was purchased from Sinopharm Chemical Reagent Co. Ltd, China with an average particle size of 120 - 260 nm.

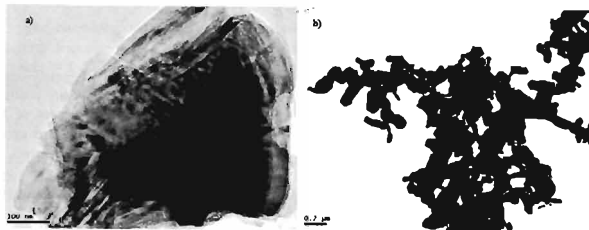


Figure 1. Transmission electron microscope images of: a) Tixogel MP100, b) submicron sized ZnO particles.

Q235 general structure steel (used as plain rebars in some cases) was purchased from Angang Iron and Steel Group, Xinyang Iron & Steel Co., Ltd, China. Chemical composition of Q235 steel in wt.% as C: 0.17, Si: 0.37, Mn: 0.08, P: 0.036, S: 0.039 and Fe: bal.

2.2. Preparation of epoxy-organobentonite nanocomposites added submicron size ZnO nanoparticles

In the first stage, GCC135 epoxy resin was heated at 45 °C to reduce viscosity by heating water bath, then, a desired quantity - 1.5 phr (phr - parts per hundred of total weight of epoxy resin and hardener) of organobentonite MP100 was added to the resin and hand stirred until all the fillers had been immersed. The mixture was stirred at 600 rpm for 15 min., 1200 rpm for next 15 min., 2600 rpm for next 1 h and 300 rpm for 30 min. using a variable speed mechanical stirrer fitted with a high shear impeller at heating condition. After that, the mixture had been self-degassed at 45 °C for 6 h. The desired quantity (0.75, 1 and 1.25 phr - parts per hundred of total epoxy resin and hardener) of ZnO submicron size articles was added to the mixture, the mixture was stirred at 900 rpm for next 15 min., 2000 rpm for next 1 h and 300 rpm for 30 min. After that, the mixture had been self-degassed at 45 °C for 6 h and degassed under vacuum in vacuum oven for next 2 h. At last, W93 hardener was added. Prior to curing, the mixture was cooled at ambient temperature (25 ± 2 °C). Curing was done in ambient condition at 24 h.

2.3. Electrodes preparation

A copper wire was electrically connected to one surface of each 10 mm \times 10 mm \times 0.5 mm Q235 steel piece, and then this surface and all the other surfaces except the one exposed to electrolyte for corrosion testing were sealed with a thick bulk E44 epoxy resin wrapped by PVC tube. After epoxy curing, the unsealed coupon surface was polished on silicon carbide (SiC) papers down to a grid size of 400. Then, the sample surface was rinsed with tap water, dried in air-flow of air-compressor machine.

2.4. Substrate preparation for adhesion test

The Q235 steel panels with 47 mm × 105 mm × 2 mm dimension were polished on silicon carbide (SiC) papers down to a grid size of 400. Then, the sample surface was rinsed with tap water, dried in air-flow of air-compressor machine.

2.5. Composite coatings preparation

The mixture (epoxy+hardener+organobentonite MP100 + ZnO) was degassed under vacuum for 10 min. at 25 °C, to obtain new composite material. Then the composite coatings were applied by wire-beam film applicator on steel substrate. Dry film thicknesses of the coatings were about 60 ± 2 μm. All samples were cured in ambient condition at 24 h. Pure epoxy and epoxy added organobentonite were prepared too for making comparison.

Table 1. Compositions of epoxy based composites were synthesized in this study.

Sample	Organobentonite concentration (phr)	Sub-micronsize ZnO particles concentration (phr)
PE	-	-
EC	1.5	0
ECZa	1.5	0.75
ECZb	1.5	1
ECZc	1.5	1.25

2.6. Synthesis of artificial sea water

According to the Lyman and Fleming formula for artificial seawater [20], the artificial seawater with salinity 3.50 % is prepared. This solution was used for corrosive media to exposure samples.

2.7. Characterization

2.7.1. Density

The novel composites were casted in plastic formwork. A cylinder samples are measured a volumes and masses to obtain densities.

2.7.2. Measurement of weight change in solution

The solution uptake of cylinder samples immersed in artificial sea at ambient temperature using glass cups was measured by recording the amount of the solution absorbed within a fixed interval of immersion time. Before immersion, all samples were dried at 60 °C for 24 h in vacuum oven and their initial weights as well as dimensions were measured. The samples were periodically removed, wiped with filter paper to remove excess solution, and then their weights – wet condition – are taken. Solution content was determined using equation:

$$Mt[\%] = \frac{W_t - W_o}{W_o} \cdot 100 \quad (1)$$

where M_t , W_t and W_0 are the solution content at a given time, weight of the sample at the time of the measurement and initial weight, respectively.

2.7.3. XRD observations for nanocomposite morphology assessment

The nanocomposites were characterized by Rigaku X-ray diffractometer using Cu- α A radiation, measured at 40 kV/250 mA. The data was recorded in the range of $2\theta = 1^\circ - 8^\circ$, at the step size of 0.02° and the counting speed of $0.5^\circ/\text{min}$. These parameters were selected based on preliminary studies to give sufficient resolution in the acquired XRD data.

2.7.4. Ultraviolet-Visible analysis

The UV light absorption properties of new materials were tested by Hitachi U3010 UV-VIS spectrophotometer. Wave length region was between 200–800 nm. The UV absorption efficiency of new materials was significantly dependent on the transmittance of UV light through samples.

2.7.5. Mechanical properties

HV test: Hardness is a characteristic of a material, not a fundamental physical property. It is defined as the resistance to indentation, and determined by measuring the permanent depth of the indentation. More simply put, when using a fixed force (load) and a given indenter, the smaller the indentation, the harder the material. The Vickers hardness test method, also referred to as a microhardness test method, is mostly used for small parts, thin sections, or case depth work. The Vickers method is based on an optical measurement system. The microhardness test procedure, ASTM E-384, specifies a range of light loads using a diamond indenter to make an indentation which is measured and converted to a hardness value. It is very useful for testing on a wide type of materials as long as test samples are carefully prepared. A square base pyramid shaped diamond is used for testing in the Vickers scale. This microhardness method is used to test those novel composites - any type of material. The HV values of the novel nanocomposites had been carried out by MH-3 Everone HV test instrument with 0.2 kgf, dwell-time is 5 s.

Adhesion test: for assessing adhesion of new nanocomposites coating films to low-carbon steel substrate, ASTM D3359 – method B had been applied in this research by QFH comb-knife instruments.

2.8. Electrochemical measurements

Electrochemical measurements were conducted using a three-electrode system. The epoxy-coated steel samples served as the working electrode, while the counter electrode and the reference electrode used were a platinum grid and a saturated calomel electrode (SCE) respectively. The coatings evaluated in the electrochemical measurements had similar thickness as those used in the morphological study since they were prepared following the same procedures. The corrosive solutions tested were artificial seawater (ASW). Two methods were used to test the anticorrosive performance of these nanocomposite coatings: electrochemical impedance spectroscopy (EIS) and potentiodynamic weak polarization. Up to 56 days immersion of the coated steel, the EIS measurements were carried out periodically using a CHI660C electrochemical workstation. The steel was polarized at ± 10 mV around its open circuit potential (OCP) by an alternating current (AC) signal with its frequency ranging from 10

kHz to 10 mHz (12 points per decade). The polarization scans were conducted at 10 mV/s, and the scan range generally started at nearly -250 mV cathodic of the open circuit potential and terminated at nearly 250 mV noble of the open circuit potential. Corrosion current (I_{corr}), corrosion rate (CR) and corrosion potential (E_{corr}) were calculated automatically by CHI ver. 8.03 software.

3. RESULTS AND DISCUSSION

3.1. Physical and Mechanical properties of samples

3.1.1. Densities

Densities of those new composites are shown in table 2. It pointed out that density is increased linearly on fraction of fillers and if using those composites to make a coating, 1 kg of composites can be applied for about 14 m² with 60 μ m thickness.

Table 2. Densities of epoxy-based composites.

Samples	Density ρ (g/cm ³)
PE	1.171
EC	1.182
ECZa	1.188
ECZb	1.191
ECZc	1.217

3.1.2. Measurement of weight change

The solution uptake of cylinder samples immersed in artificial seawater at ambient temperature is shown as figure 2. It can be observed that ECZb sample has low and stability solution contents; it means that highest dense and lowest degradation.

3.1.3. XRD observations

Among the methods of determining the dispersion characteristics of fillers in polymers, X-ray diffraction (XRD) and transmission-electron microscopy (TEM) are widely used. In the present study, XRD is given preference over TEM because transmission-electron microscopy observes the structure of the material in a very small area, which is less than 0.2 μ m in length and width at 10⁶ magnification. The structure observed in such a small area may not be representative of the large batches. Therefore, XRD is used for characterization because it uses relatively large specimen size and sample selection will have a much smaller effect on the results. The XRD spectra for the synthesized composites are shown in figure 3.

Based on this figure, it can be observed that all samples have no exhibit any peak in their spectra, which indicates that fillers have exfoliated.

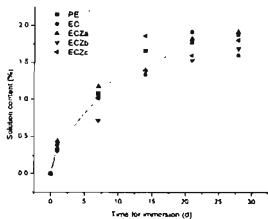


Figure 2. Solution content of samples in ASW solution.

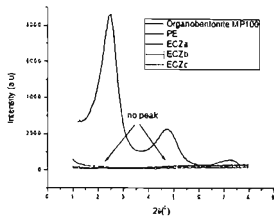


Figure 3. XRD spectrum of the samples.

3.1.4. Ultraviolet-Visible analysis

UV-VIS spectroscopic measurements confirmed that ZnO filler is extremely efficient UV absorber since 3 mm thick plates. ECZc sample absorbs more than 95% of the incident UV light (Figure 4). At the same time more than 85 % of the incident UV light was absorbed by ECZa, ECZb samples. Furthermore, only approximately 30 % visible light is transmitted through the ECZ samples. It means that new ECZ composites have a good UV absorption properties. Hence, those new materials have a potential application as UV stabilized materials for various outdoor applications [21].

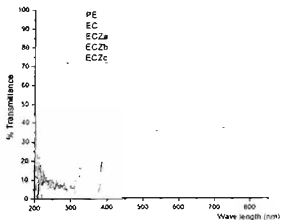


Figure 4. UV-VIS spectra of samples.

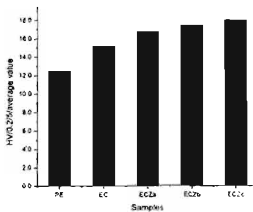


Figure 5. HV0.2/5 results of of samples.

3.1.5. The microhardness (HV) and adhesion grade

HV results of 5 types samples are shown in figure 5. It pointed out that added ZnO/organobentonite fillers increasing hardness of composites effectively.

Adhesion of coating films on steel substrates was also investigated. The results revealed that the adhesion does not vary strongly with increase in filler concentration. PE sample was up to 4B grade and others were also up to 5B grade of adhesion.

3.2. Effect of filler particles on the corrosion resistance of the coated Q235 steel

EIS plots for all samples are presented in the Bode plot format as a function of exposure time. According to the literature, the impedance modulus at the low frequency ($|Z|$ at 0.01 Hz) is a useful parameter to characterize the corrosion protection of coatings. The open circuit potential was obtained before making AC impedent test also. The electrochemical analysis of one bare Q235 steel after 56 days of immersion in ASW was carried out for comparison.

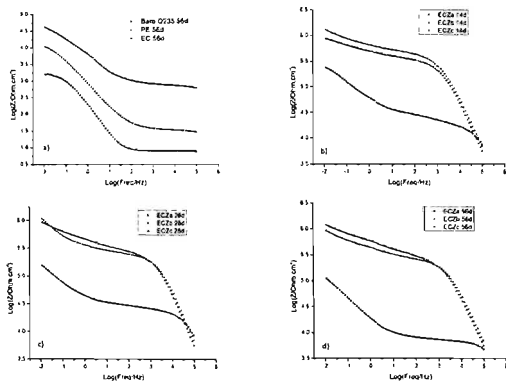


Figure 6. Bode plots of: a) Bare Q235 steel, PE, EC after 56 days, b) ECZ samples after 14 days, c) ECZ samples after 28 days and d) ECZ samples after 56 days of immersion in ASW.

Figure 6 provides the Bode plots data of bare and composite-coated steel in ASW solutions, as a function of filler particles concentration and exposure duration. The values of OCP and impedance $|Z|$ at 10 mHz of all samples exposed to ASW at different immersion times are shown in Table 3. It should be cautioned that the OCP reading of the coated steel was contributed both by the corrosion potential of the steel itself and by the electrical resistance of the coating layer.

As are shown in Fig. 6 and Table 3, because GCC135 is a low viscosity liquid epoxy resin, 60 μm thickness PE coating had poor protective effect. After 56 days of immersion, it has the $|Z|$ value at 10 mHz nearly 7 times of bare Q235 steel and the OCP of PE coating sample is linear increment following immersion time. The incorporation of a small amount of organobentonite (1.5 % by total weight of resin and hardener) into the pure epoxy, the EC composite was obtained, EC coating's corrosion resistance is more effective than PE coating, the $|Z|$ value at 10 mHz is more stability in corrosive solution but still at low value.

Incorporating low-loading organobentonite nanoparticles and ZnO submicron size particles together into epoxy coatings, in the initial period of immersion (after 14 days), the ECZb coating possessed the highest $|Z|$ value at 10 mHz, followed by the ECZc and ECZa just had a lowest $|Z|$ value at 10 mHz. After 28 days of immersion, the value of $|Z|$ at a low frequency of 10 mHz gradually decreased, at the same time, OCP decreased, which indicated that corrosion had occurred. Until the end of immersion process, the value of $|Z|$ at a low frequency of 10 mHz was still maintained at around 10^6 Ohm.cm^2 . Because the amount of ZnO submicron size particles was not enough to improve barrier properties of ECZa strongly, so the value of $|Z|$ at a low frequency of 10 mHz and OCP gradually decreased.

With highest amount of ZnO submicron size particles, the ECZc coating had the value of $|Z|$ at a low frequency of 10 mHz, which was below 10^6 Ohm.cm^2 after 14 days of immersion, then increased slightly after 28 days until the end of immersion. It proved that in this case, some ZnO submicron size particles were not well dispersed into epoxy resin and MP100 mixture, the number of pore in ECZc coating was much more than ECZb coating, some corrosion products sealed the pores of the coating. Therefore, with suitable loading of fillers, a ECZb coating showed good protective effect.

Table 3. The values of OCP and impedance $|Z|$ at 10 mHz of all samples exposed to ASW.

Samples	OCP (V/vs SCE)			$ Z $ at 10 mHz (Ohm.cm^2)		
	14 days	28 days	56 days	14 days	28 days	56 days
Bare Q235	-	-	-0.711	-	-	1.554E+3
PE	-0.623	-0.657	-0.670	4.452E+3	5.078E+3	1.071E+4
EC	-0.623	-0.634	-0.649	4.425E+4	4.042E+4	4.121E+4
ECZa	-0.615	-0.609	-0.62	2.381E+5	1.555E+5	1.101E+5
ECZb	-0.434	-0.487	-0.542	1.313E+6	1.082E+6	1.006E+6
ECZc	-0.496	-0.538	-0.558	8.765E+5	9.329E+5	1.203E+6

According to the other studies [3, 10], from Tafel polarization curves, it can be observed that the anticorrosion properties of different coatings were further revealed clearly and directly. It can be seen from Fig. 7 and table 4, once again, that the ECZb coatings indicated the highest corrosion potential ($E_{\text{corr}} = -0.631 \text{ V}$). Then, the pure epoxy coating showed the lowest corrosion potential. However, the sequence of the corrosion current (I_{corr}) is just the opposite. The corrosion current is an important factor to characterize the anticorrosion performance. The pure epoxy coatings had the highest corrosion current. Then, the ECZc coating had the lowest corrosion current.

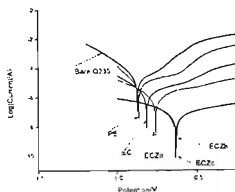


Figure 7. Tafel polarization curve of all samples after 56 days immersion in ASW.

As were shown in Table 4, after 56 days of immersion, the filler particles reduced the corrosion rate of composite coated Q235 steel by 2.3 times with pure epoxy, by 10 times with organobentonite only, by 23 times with ECZa, by around 356 and 410 times with organobentonite and ZnO submicron size particles together in ECZb and ECZc.

Table 4. Fitting values of Tafel plot of different coatings after 56 days immersion in ASW.

Samples	Fitting values of Tafel plot		
	E _{corr} (V)	I _{corr} (A.cm ⁻²)	CR (mm/year)
Bare Q235	-0.869	1.910E-5	2.218E-1
PE	-0.868	8.217E-6	9.543E-2
EC	-0.816	1.849E-6	2.147E-2
ECZa	-0.761	8.187E-7	9.500E-3
ECZb	-0.631	5.370E-8	6.236E-4
ECZc	-0.635	4.659E-8	5.410E-4

It pointed out that with suitable loading; the resistance values were much higher than those of pure epoxy coating. Furthermore, this indicated effectiveness of ZnO particles for improving barrier properties of coating layer.

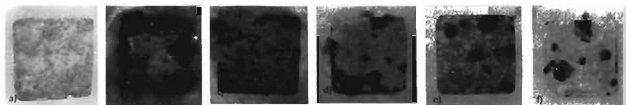


Figure 8 Digital photograph of composite-coated Q235 steel exposed to ASW after 56 days: a) bare Q235 steel, b) PE coating, c) EC coating, d) ECZa coating, e) ECZb coating, d) ECZc coating.

As were shown at Fig. 8, there is a thick rust layer on the surface bare steel samples, there is a rust layer around perimeter of PE sample and there are some rust dots on the surface of EC

sample. But on the surface of the ECZ samples, there are some colored dots only. It means that solution diffused into interaction layer between coating and steel but no rust yet. This suggests that filler particles improved the quality of the cured epoxy coating, reduced the porosity of the coating matrix, and zigzagged the diffusion path available by deleterious species, leading to improved barrier performance of the epoxy coating. Besides that, filler particles improved the adherence of the cured epoxy coating to the underlying substrate and altered the physiochemical properties of the coating-steel interface.

4. CONCLUSION

Incorporating low-loading organobentonite nanoparticles and ZnO submicron size particles together into low-cost organic coatings improves the both anti-corrosive properties and mechanical properties of coatings. Based on those results, the best performance of coatings was obtained at 1phr ZnO submicron size particles concentrations (ECZb sample). It is suggestion for economical consideration. By using mechanical mixing method or general machine, this type of coating can be applied for protection of rebar, general low-carbon steel structures in corrosive environment such as seashore in-field. This processing procedure can be applied for other type of epoxy resin.

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TÓM TẮT

CHẾ TẠO, ĐẶC TÍNH VÀ TÍNH CHẤT CHỐNG ẪN MÒN CỦA COMPOSITE NỀN EPOXY CHỨA BENTONITE HỮU CƠ, BỘT ZnO CÓ CỠ HẠT DƯỚI MICROMETER

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Bài báo này trình bày quá trình chế tạo lớp phủ composite nền epoxy được tăng cường bằng bentonite hữu cơ và bột ZnO có cỡ hạt dưới micrometer với các hàm lượng khác nhau. Sử dụng phương pháp nhiễu xạ tia X để đánh giá khả năng phân tán bentonite và hạt ZnO vào nền epoxy. Nghiên cứu độ cứng, độ bám dính, tính chất vật lý cơ bản và khả năng chống ăn mòn của vật liệu chế tạo được. Kết quả nghiên cứu cho thấy rằng với hàm lượng thấp, bentonite hữu cơ phân tán tốt vào nền epoxy và tạo ra loại composite tách lớp. Khi tăng cường thêm thành phần bột ZnO, độ cứng và độ bám dính của vật liệu chế tạo được tăng nhẹ so với epoxy và epoxy có gia cường bentonite hữu cơ. Khả năng chống ăn mòn của màng phủ làm bằng vật liệu mới cũng được gia tăng do khi tăng cường đồng thời bentonite hữu cơ và bột ZnO vào nền epoxy thì khả năng cản thấm của vật liệu cũng được cải thiện.

Từ khóa: chống ăn mòn, sơn tổng hợp epoxy-organobentonite, EIS, hạt submicrometer ZnO.

