



Preparation and Evaluation of Coating Have One and Two Shells (Part 2)

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CrossMark

MICRO-capsules are prepared with two basic forms, the 1st form is covered by one shell made of urea-formaldehyde, the 2nd one is covered by two shells (the first shell is urea-formaldehyde and the second shell made of poly-urethane).

Both the first and the second forms of the Micro-capsules are filled by two types which are corrosion inhibitors (corr A and corr B). Some tests were applied and discussed like: optical scanning microscope was used to confirm the formation of capsules, microcapsules were weighed in order to measure the shrinking rate, thermal gravimetric analysis was applied to find the rate of stability under heating of the micro-capsules; Infra-red spectroscopy was applied to make sure of the formation of shells of the micro-capsules and physical properties were monitored weekly to confirm the stability of the micro-capsules (these tests were applied and discussed in paper part 1 which sent under the number : 1809-1467). In this paper the self-healing properties are focused on as: micro-capsules of the one and two shells were incorporated with epoxy paint (which used as a protective coating for severe environmental conditions). Micro-capsules showed promising corrosion protection via performing self-healing properties as it focused on adding different anti-corrosive materials into capsules of one and two shells which opens the door for new uses and additions. The research results would be important for self-healing and anti-corrosive materials industry.

Keywords: Micro-capsules, Self-healing, One shell, Two shells.

Introduction

Metals including steel, magnesium, and aluminum, and their alloys are widely used in industrial and engineering structures due to their high strength and ductility [1]. Application of coatings is the most common strategy to protect metals from corrosion [2]. Generally, the coating consists of several layers, of which the most important ones are the pretreatment layer, primer and topcoat [3].

Anticorrosive coatings are often used as a mean for protection of steel structures against corrosion. Although the high performance of these materials can be now achieved showing high adhesion, good mechanical properties and durability, various mechanical, chemical, or thermal impacts, experienced by the products

during their service life, lead to formation of micro cracks and premature failure of the coating system [4-5]. Maintenance and repair of the damaged coatings on large industrially used steel structures like offshore oil rigs, sea ships, etc. are known to be very expensive. Costs of structural failures, subsequent environmental pollution and sometimes even human lives are dramatically higher than that. Looking for solutions to prolong service life of anticorrosive coatings, scientists referred to intrinsic and the most remarkable ability of biological materials to self-repair [6] and have developed a class of polymeric materials called self-healing. Most often self-repairing or self-healing coatings are understood as the coatings, which have ability to restore their structural integrity once a micro-damage

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Received 26/9/2018; Accepted 18/12/2018

DOI: 10.21608/EJCHEM.2018.5289.1468

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has happened. Healing occurs at the site of the micro-crack and prevents the crack from further propagation and diminishes penetration of water, oxygen and ions down to the substrate and, thereby protecting the metal substrate from corrosion. Meanwhile, another meaning of —self-healing coating can be found in the relevant literature. Thus, A. Yabuki *et al.* [7], A. Zheludkevich *et al.* [8], and G. Galiwoda-Porebska *et al.* [9] described self-healing efficiency of the fluoro-organic compound, 8-hydroxyquinoline and polypyrrole-containing coatings, in which release of inhibitor restores the protection function of the coatings, although the real healing of the coating fracture does not occur. So far, varieties of materials have been explored, such as anodized coatings [10], layered double hydroxide (LDH) inorganic conversion coatings [11], and hybrid organic–inorganic sol–gel films [12–13]. An alternative of “green” chitosan, a linear polysaccharide, is also reported as a potential candidate for pretreatment due to its excellent adhesion to metallic surfaces and to many organic polymers [14–15]. In the case of primers, conventional organic coatings or paints, including polyurethane [16], epoxy [17], alkyd primers [18], and polyvinyl butyral [19] are usually utilized considering their low cost, easy application, and aesthetic functionality.

Encapsulation of functionally active materials in hollow microspheres is an attractive way of storing as well as protecting these from environment till required for fulfilling appropriate applications. Microencapsulated substances have been utilized for sustained drug release [20,21], electro rheological fluids [22], intumescent fire retarding powders [23,24], preservation of flavors [25,26], electro phoretic display applications [27], textiles [28], biotechnology [29,30] and inorganic metal salt catalyst [31], etc. Recently, there has been growing interest in use of microencapsulated materials for healing of cracks generated during service of a polymer based composite materials [32, 33]. Microcapsules containing dicyclopentadiene were incorporated in the composite matrix. These capsules rupture and release dicyclopentadiene during crack formation and reacts with Grubbs ruthenium catalyst present in the composites leading to crack repair to restore mechanical properties.

Paints are extensively used for modification of substrates either for aesthetic appearance or for corrosion protection. During its service life, the paint film undergoes changes in mechanical

properties leading to formation of micro-cracks which subsequently propagates and exposes substrate to atmospheric moisture and oxygen. This action results in accelerated dis-bonding of the paint and flake formation from the metal coating interface. Paint coatings can be considered as a special class of composite materials, comprising binders and pigments. Hence, the concept of self-healing of cracks, as reported for composites, can be adopted for coatings to provide longer durability. An attempt for healing of scratches on automotive coating using temperature dependent elastic properties of polymer has been reported [34.] Here, we report our work on development of self-healing coatings with microencapsulated drying oil. In this study, linseed oil along with driers has been selected as a healing agent due to its film forming ability by atmospheric oxidation. Microcapsules with urea–formaldehyde as a shell and drying oil as a core were synthesized by in situ polymerization [35]. Efficacy of these microcapsules in healing of cracks in an epoxy coating and corrosion protection has been demonstrated.

Materials used

One shell formation chemicals (All materials are supplied by international trade company- Cairo- Egypt) these include-:

Urea, formaldehyde, ammonium chloride, resorcinol, poly vinyl alcohol (PVA), sodium dodecyl sulphate as stabilizer, nonyl phenol poly ethylene glycol as surfactant, linseed oil, fumaric acid, di-ethanol amine, xylene and zinc oxide.

Materials for two shells formation chemicals (All materials are supplied by international trade company- Egypt) these include-:

Toluene 2,4- di-iso cyanate(TDI), di-ethylene triamine(DETA), micro-capsules obtained from first step would be used as a core material, NP-9 surfactant used as emulsifier added to sodium dodecyl sulphate, linseed oil, fumaric acid, di-ethanol amine, xylene and zinc oxide.

Preparation of corrosion inhibitor (A)

TABLE 1. Corrosion inhibitor (A) formation.

Material used	Weight, gm
Linseed oil fatty acid	55.2
Fumaric acid	18.3
Water	3.5
Diethanol amine	23
Total	100

Experimental and formation of micro-capsules

Fig. 1. Four necked glass kettle for preparation.

Procedure

Linseed oil fatty acid is charged to 4-necked flask and raises the temperature up to 180 °C. The Fumaric acid was added little by little with stirring. Temperature was raised up to 250°C with stirring for one hour, then check the solubility of the adduct in xylene, when solubility is complete, cool to 70 °C and power off.

Add little by little a mixture of water and diethanol amine and reflux to 110 °C for 2 hours with stirring.

Check the solubility in water (10 gm product/100 gm H₂O)

Preparation of corrosion inhibitor (B)

TABLE 2. Corrosion inhibitor (B) formation.

Materials used	Weight, gm
Diethanol amine	21.2
Linseed oil fatty acid	51.2
Xylene	27.4
Zinc oxide	0.2
Total	100.0

Procedure

Diethanol amine, linseed oil fatty acid, xylene and zinc oxide were added in 4-necked flask under mechanical stirrer, thermometer, nitrogen gas. The content was heated up to reflux (\approx 110°C) for 2 hours.

Dean and Stark trap apparatus was used and strip the water (0.2 mol) i.e. 3.6 ml (for 100 gm

materials \approx 6.3 ml). The flask was cooled and filtered.

Experimental synthesis of microcapsules (36)

Microcapsules were prepared by in situ polymerization in oil in- water emulsion. At room temperature, 260 ml of de-ionized water, 10 ml of 5 wt% aqueous solution of polyvinyl alcohol (PVA) and 0.5 gm of sodium dodecyl sulphate were mixed in 1000 ml beaker. Under agitation 5 g urea, 0.5 g ammonium chloride and 0.5 g resorcinol were dissolved in solution. The pH was adjusted to approximately 3.5 by using 5wt% solution of hydrochloric acid in de-ionized water. One to two drops of octanol was added as an antifoaming agent. 30 ml of linseed oil was added slowly to form an emulsion and allowed to stabilize for 10min under agitation. After stabilization, 12.67 g of 37 wt.% aqueous solution of formaldehyde was added. The emulsion was covered and slowly heated and maintained at 55°C under stirring at 200rpm for 4 h.

Contents were cooled to ambient temperature. Microcapsules from the suspension were recovered by filtration under vacuum. These were rinsed with water, washed with xylene to remove suspended oil. The capsules were dried under vacuum.

Preparation of two shells microcapsules (23)

The micro-capsulation was carried out in three necked flask round bottom flask equipped with mechanical stirrer. Prior to encapsulation (nonylphenol ethoxylate) (2.5 gm) was added

to 80 ml water. An organic solution of cyclohexane (10 ml) added to TDI (3 gm) was prepared. The organic solution was added to the surfactant solution and the mixture was stirred with rate between 400-500 rpm to form oil in water emulsion. After 3 minutes of stirring,

DETA was diluted by distilled water (20 ml) and added slowly to start the interfacial condensation reaction continued to 90 minutes at 60 centigrade.

Resultant micro-capsules were filtered and then washed with water to remove the excess of

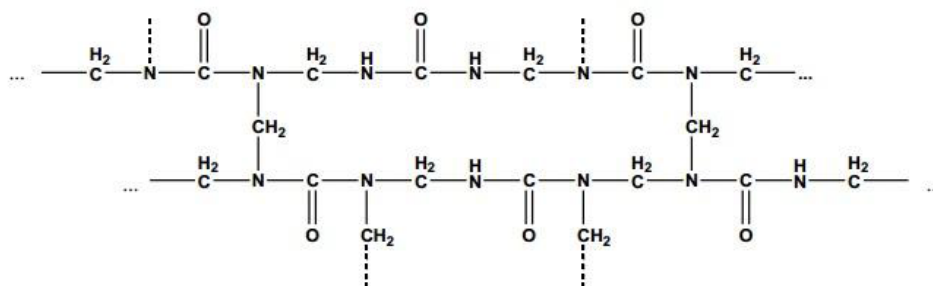


Fig. 2. Formula of cross-linked poly-urea formaldehyde capsules shell.

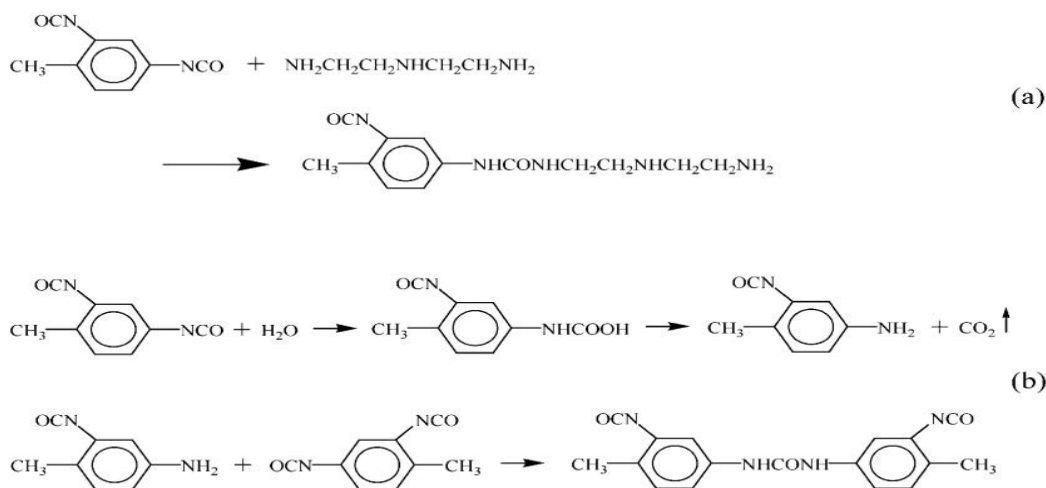


Fig. 3. Formula of poly-urethane micro-capsules shell.

DETA and dried in vacuum oven.

Test applied on micro-capsules

Self-healing properties: monitoring the change in self-healing properties of coating with different samples and different components.

Results and Discussion

Formation of micro-capsules under scanning electron microscope:

From Fig. 4 and 5:

It is clear that micro-capsules were already formed and as the magnification increases, the capsules are clear to be noticed. Capsules are formed and get closer to each other like a cloudy

shape which eases the self-healing process after rapture.

Self-healing properties:

The process of crack healing, on a coated surface, was carried out by incorporating micro-capsules into a solvent free anticorrosive paint. This paint having solid content 76wt%, density 1.7g/cm³ was used with the hardener. Anti-corrosion filled microcapsules were mixed in paint to produce contrast in the crack when observed under optical microscope or even under naked eye to record the healing process. A tinned panel, 15cm×20cm×0.4mm was coated with single coat to produce a dry film thickness (film thickness measured and was between 0.013 to 0.025 inches

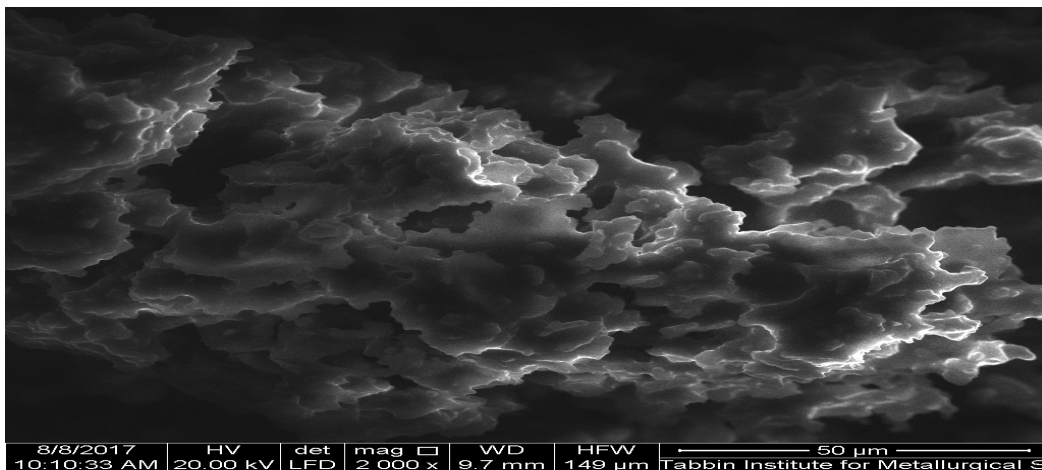


Fig. 4. SEM shot of formation of two shells micro-capsules under 2000X magnification lens.

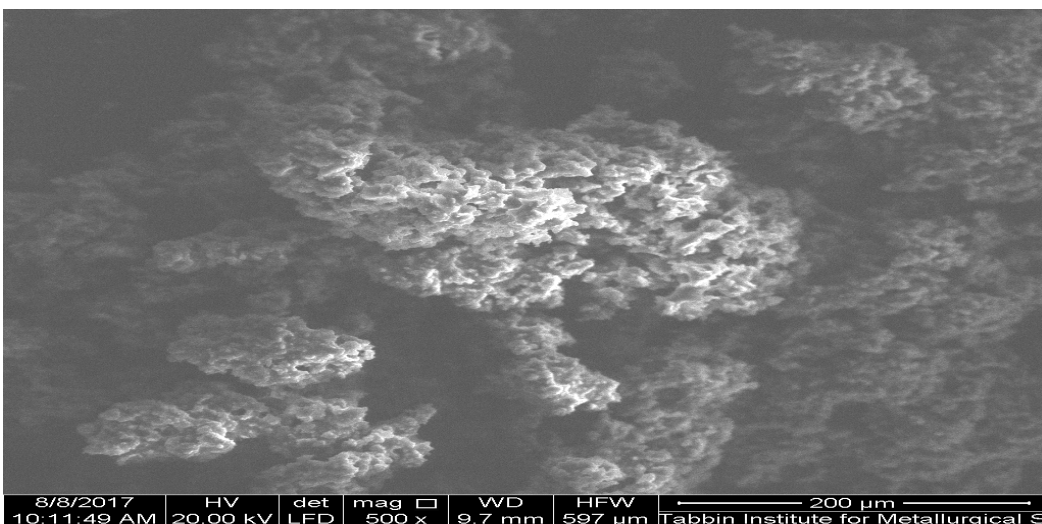


Fig. 5. SEM shot of formation of two shells micro-capsules under 500X magnification lens.

TABLE 3. Samples prepared for self- healing test.

Sample	Formation
1	Normal sample-Chemapoxy
2	Linseed oil as the core and UF as the shell
3	(Linseed in two shells(urea formaldehyde and poly-urethane
4	(Corrosion inhibitor A in one shell(urea formaldehyde
5	(Corrosion inhibitor A in two shells(urea formaldehyde and poly-urethane
6	(Corrosion inhibitor B in one shell(urea formaldehyde
7	(Corrosion inhibitor B in two shells(urea formaldehyde and poly-urethane

for all samples). After 7 days of curing a crack was generated in the coated panel. Panel was immediately kept under observing to record healing process initiated due to release of anti-corrosive materials from ruptured microcapsules.

Seven samples were prepared to perform the physical properties:

- First sample is normal chema-poxy 131, a commercial paint used for protection and made by Modern Building Chemicals Company (MBC).
- Second sample is normal micro-capsules of one shell of urea formaldehyde which contains linseed inside mixed in chema-poxy 131.
- Third sample is normal micro-capsules of two shells of urea formaldehyde as 1st shell and polyurethane as 2nd shell mixed in chema-poxy 131.
- Fourth sample is corrosion inhibitor A micro-capsules of one shell of urea formaldehyde mixed in chema-poxy 131.
- Fifth sample is corrosion inhibitor A micro-capsules of two shells of urea formaldehyde as 1st shell and polyurethane as 2nd shell mixed in chema-poxy 131.
- Sixth sample is corrosion inhibitor B micro-capsules of one shell of urea formaldehyde mixed in chema-poxy 131.
- Seventh sample is corrosion inhibitor B micro-capsules of two shells of urea formaldehyde as 1st shell and polyurethane as 2nd shell mixed in chema-poxy 131.

After preparation of samples, samples from 2 to 7 are mixed with chema-poxy 60 and applied to metallic plates and let for 24 hours for complete hardening. After complete dryness the samples would suffer scratching and let in normal atmosphere for 24 hours. Then samples are immersed in 10% sulfuric acid for 48 hours and then observing the steel panels.

Note that: all samples color changes to brown which indicates the micro-capsules rupture and the corrosion inhibitor flowing.

1. From Fig. 6 and 7 which show the difference of the same sample (1) before and after immersing in 10% sulfuric acid, it is noticed that the corrosion is started to appear beside the removal of some parts of the paint, this indicates that paint is not tightly adhere the metallic plate and it was influenced deeply by the acid which caused the corrosion to start, Fig. 8 shows the sample after one year and the corrosion continues to spread.
2. From Fig. 9 and 10 which show the difference of the same sample (2) before and after immersing in 10% sulfuric acid, it is noticed that the corrosion started to appear and there is no removal of the paint, this indicates that the paint tightly adheres the metallic plate while the corrosion is shown in the scratched area, so it's not suitable to be used as a self-healing material, Fig. 11 shows the sample after one year and the corrosion continues to spread with a slow rate.



Fig. 6. Normal chema-poxy sample.



Fig. 7. Chema-poxy sample after scratching and immersing in 10% sulfuric acid for 48 hours.



Fig. 8. Chema-poxy sample after scratching and immersing in 10% sulfuric acid after one year.



Fig. 9. Linseed sample with one shell after scratching – before immersing in 10% sulfuric acid.



Fig. 10. linseed sample with one shell after scratching – after immersing in 10% sulfuric acid for 48 hours.



Fig. 11. linseed sample with one shell after scratching – after immersing in 10% sulfuric acid after one year.

3. From Fig. 12 and 13 which show the difference of the same sample (3) before and after immersing in 10% sulfuric acid, it's noticed that no corrosion is shown to appear and there is no removal of the paint beside

decreasing in scratched area, this indicates that the paint is adherent to the metallic plate and self-healing is demonstrated, so it's suitable to be used as a self-healing material, Fig. 14 shows the sample after one year and then there is a small area of corrosion present.



Fig. 12. linseed sample with two shells after scratching – before immersing in 10% sulfuric acid.



Fig. 13. linseed sample with two shells after scratching – after immersing in 10% sulfuric acid for 48 hours.



Fig. 14. linseed sample with two shells after scratching – after immersing in 10% sulfuric acid after one year.

4. From Fig. 15 and 16 which show the difference of the same sample (4) before and after immersing in 10% sulfuric acid, it is noticed that no corrosion started to appear and there is no removal of the paint beside decreasing in scratched area, this indicates

that paint is tightly adhere the metallic plate and self-healing demonstrated, so it is suitable to be used as a self-healing material, Fig. 17 shows the sample after one year and there is no corrosion presents..



Fig. 15. Corrosion inhibitor A sample with one shell after scratching – before immersing in 10% sulfuric acid.



Fig. 16. Corrosion inhibitor A sample with one shell after scratching – after immersing in 10% sulfuric acid.



Fig. 17. Corrosion inhibitor A sample with one shell after scratching – after immersing in 10% sulfuric acid after one year.

5. From Fig. 18 and 19 which show the difference of the same sample (5) before and after immersing in 10% sulfuric acid, it's noticed that the corrosion started to appear and there is no removal of the paint, that indicates the paint is tightly adhere the

metallic plate while the corrosion started in the scratched area, so it's not suitable to be used as a self-healing material, Fig. 20 shows the sample after one year and the corrosion spreads with a slow rate.



Fig. 18. Corrosion inhibitor A sample with two shells after scratching –before immersing in 10% sulfuric acid.

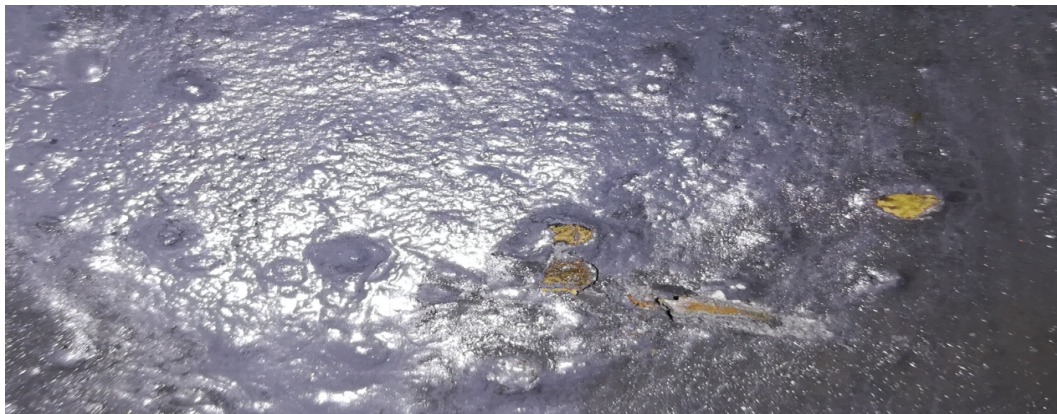


Fig. 19. Corrosion inhibitor A sample with two shells after scratching – after immersing in 10% sulfuric acid.



Fig. 20. Corrosion inhibitor A sample with two shells after scratching – after immersing in 10% sulfuric acid after one year

6. From Fig. 21 and 22 which show the difference of the same sample (6) before and after immersing in 10% sulfuric acid, its noticed that no corrosion started to appear and there is no removal of the paint beside decreasing in scratched area, this indicates

that paint is tightly adhere the metallic plate and self-healing started, so it is suitable to be used as a self- healing material, from Fig. 23 shows the sample after one year and there is no corrosion presents.



Fig. 21. Corrosion inhibitor B sample with one shell after scratching – before immersing in 10% sulfuric acid.



Fig. 22. Corrosion inhibitor B sample with one shell after scratching – after immersing in 10% sulfuric acid.



Fig. 23. Corrosion inhibitor B sample with one shell after scratching – after immersing in 10% sulfuric acid after one year.

7. From Fig. 24 and 25 which show the difference of the same sample (7) before and after immersing in 10% sulfuric acid, it's noticed that the corrosion started to appear and there is no removal of the paint, that indicates the paint is tightly adhere the metallic plate while the corrosion started in the scratched area, so it's not suitable to be used as a self-healing material, Fig. 26 shows the sample after one year and the corrosion continues to spread with a slow rate.

To summarize that, it is shown that sample 3, 4 and 6 have the ability to make self-healing for paint more than the other samples.

It's due to the rapid breaking of the shells in 4

and 6 samples as they have only one shell which is a good characteristic for early self-healing of paints while the two shells micro-capsules might be used for long term protection.

In sample 3, shells were broken early as linseed made self-reaction with the first shell (urea formaldehyde) which made the shells weak and easily broken, it must be noticed that the rate of self-healing is high as a polymer material was formed quickly on the metallic surface but not as strong as the polymer formed in sample 4 and 6.

So it is recommended to use sample 4 and 6 as self-healing material more than using sample 3 for short term protection, while sample 5 and 7 could be used for long term protection.



Fig. 24. Corrosion inhibitor B sample with two shells after scratching – before immersing in 10% sulfuric acid.



Fig. 25. Corrosion inhibitor B sample with two shells after scratching – after immersing in 10% sulfuric acid.



Fig. 26. Corrosion inhibitor B sample with two shells after scratching – after immersing in 10% sulfuric acid after one year.

Conclusion

Smart paints now have many important uses and addition to the chemical industry and in the core of it the paints industry. By making micro-encapsulation for some materials which used as anti-corrosive and self-healing materials, it could

give greater results.

In this paper the anti-corrosive material (A) and (B) were encapsulated in one shell of urea-formaldehyde and in then in two shells of urea-formaldehyde and poly-urethane. Samples were tested and the results were promising after

monitoring the self-healing rate and results. According to self-healing test, results showed that corrosion inhibitor (B) gave better results than corrosion inhibitor (A) and two shells type lasts more than one shell type.

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تحضير وتوصيف الكبسولات الميكرونية ذات الطبقة والطبقتين (الجزء الثاني)

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يوجد نوعان من الكبسولات الميكرونية، النوع الاول مغلف بطبقة واحدة من اليوريا فورمالدهيد، بينما النوع الثانى مغلف بطبقتان: اولاهما من اليوريافورمالدهيد والثانية من البولى يوريثان.

كلا النوعان مملوء بمادة مضادة للتآكل: الاولى هي (كور أ) والثانية (كور ب).

يتم عمل عدة اختبارات للتأكد من التركيب وهى: الفحص بالميكروسكوب الضوئي، وزن الكبسولات لتقييم مدى الانكماش تحت تأثير الحرارة، اختبار الأشعة تحت الحمراء للتأكد من تركيب الكبسولات، توصيف الخواص الفيزيائية للتأكد من استقرار الكبسولات الميكرونية (تم مناقشة هذه الاختبارات تفصيلاً فى الجزء الاول). فى الجزء الثانى يتم استكمال الدراسة عن طريق شرح خاصية الالتئام الذاتى لمقاومة التآكل.

يتم دمج الكبسولات الميكرونية ذات القشرة والقشرتين بعد تحضيرها مع دهان الايبوكسي ودراسة الفارق ما بين استخدام الايبوكسي وحده وما بعد الدمج مع الكبسولات. يتم ملاحظة النتائج بعد مرور يومين من غمر العينة فى حامض الكبريتيك المركز ١٠ ٪ لمدة يومين وبعد مرور سنة من ترك العينة فى الهواء الجوى. اظهرت العينة نتائج مميزة وواحدة اثناء عملية اعادة الالتئام.