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Spray pyrolysis of Low Carbon Steel by Polymer Matrix Composite

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Abstract

In this research study the effect of spray pyrolysis that coating low carbon steel with epoxy reduced corrosion rate by (89.4%), while coated low carbon steel with epoxy composite reinforcement at 2%wt of (Al,TiO_2, and Zn) was result the corrosion rate of coated specimens with epoxy composites reinforcement (Al, TiO_2, and Zn) are lower than coated specimens with epoxy by (73.42%,91.75%,97.9%) respectively. The weight loss of low carbon steel coated with epoxy at90ºand 30^0 impingement angles are lower than those of uncoated specimens of low carbon steel by (51.06%,43.2%) respectively ,while the weight loss at90ºand 30° of coated specimens with epoxy composites reinforcement (Al,TiO_2) lower than coated specimens with epoxy by (34.78%,17.39%)and (47.61%,23.80%) respectively. The weight loss of erosion characteristics at90ºand 30^0 of coated specimens with epoxy composites reinforced (Zn) are higher than coated specimens with epoxy by (34.78 %, 28.57%) respectively. The wettability determine by measuring the contact angle that are small than 70º signifies hydrophilic surfaces have high surface energies and good wettability. The adhesive strength of coated specimens with epoxy composite has low adhesive strength than that in coated specimens with. The used (AFM) to showed surface morphology and surface roughness of coated specimens with epoxy and epoxy composites .The Pore Size measurement of specimens surfaces coating by (SEM), signifies each type of coated specimens with epoxy composites decreased than with epoxy.

Keywords: steel bar corrosion, erosion ,spray pyrolysis, Adhesion test

Introduction

Steel bars deterioration due to corrosion is worldwide problem causing billions of dollars in repair and replacement and costing drastic failures in many infrastructures. Usually steel bars are protected in alkaline incubator combined with other protections such as coating, cover against exposure, and/or using inhibitors to mitigate their deterioration direction when exposed to harsh environment [1] figure 1 below show

Figure 1: Corrosion of steel bar [2]

Most of the times steel reinforcement is exposed to the atmosphere during transportation and storage in the building sites for a long period before their installation in the concrete structures. At any of those stages, steel bar can be contaminated by chloride ions from sea spray or the windy salt. This fact leads to the formation of corrosion products on their surface [2].The organic coatings have played an important role in corrosion protection of metals and have been used in a large scale in many industries these coatings form a protective layer over the metal substrates and prevent them from oxidation which could affect the function and appearance of the object [3].

Experimental part

The substrate used for applying the coating by spray pyrolysis was Low Carbon Steel 1022**.** The coating materials used Epoxy resin matrix was (Quickmast 105) which is a two components product composed of resin base and formulated amine hardener mixed at a ratio 3:1. And three types of reinforcement material powder by 2%weight percent aluminum (Al) type (Himedia India), TiO_2 (GCC England), and Zinc (BDH England).

Spray pyrolysis Technique:

One of the major techniques to deposit a wide variety of materials in thin film form. To obtaining good quality thin film is the optimization of preparative conditions such as, substrate temperature, spray rate, concentration of solution [4]. Advantage spray pyrolysis in general it's simple, cheap, and safe technique, its low cost of the equipment's and raw materials needed, it does not require high quality of targets and substrates, the homogeneity and film thickness can be controlled by changing spray parameters [5].Fig. 2 shows the apparatus used to Control of temperature, timer of spray, vacuum, Nozzles spray, base of substrate. The following procedure was adopted during specimens coating process:

- The specimen is fixed on the base with fixed at 90^o and the distance 10cm from substrate and the substrate was heated at $50C^0$.Used anhydrous ethanol alcohol was added to composite material as solvent. The time of deposition run was (5sec).
- The deposition runs were repeated until a certain thickness was deposit on the substrate (175±25) µm and then coating layer was dried in air for one day than the curing Process was carried out at $50C^0$ for 1hr.

Figure 2: Spray pyrolysis technique.

Corrosion Test:

The method of the measuring corrosion rate procedure is summarized as follows Carefully weigh the specimens of uncoated and coated low carbon steel with epoxy and epoxy composite for a known period of time.

1- The weight loss measurements were performed on the surface of hanging coated specimen in a glass vessel containing (6ml) of (3.5%) wt NaCl salt solution at room temperature , the immersion time was 90days .
2- The coated specimens and uncoated were withdrawn and then rinsed with distilled water and washed with acetone, dried and weighed.
3- The value of the weight loss was calculated.

The value of the corrosion rate measurement is obtained using below.

$$\textit{Corrosion rate (mpy)} = 534 \frac{W}{DAT} \quad \text{.... (3-1)}$$

W= weight loss (gm)
D= density of specimen g/cm3
A= area of specimen ($in.^2$)
T= exposure time (hrs.)

Erosion Test:

The erosion test set up consists of an air compressor, a pressure gauge, a gun, air particle silica sand mixing chamber, accelerating tube. Schematic of erosion test is shown in Fig.3 with specimen fixed in erosion test. These particles impact the specimen which can be held at different angles

Figure 3: Schematic of erosion test.

Coating Thickness Gauge:

Coating Thickness Gauge type (QuaNix1500 Germany) .Fig.4 was used to measure the thickness of coating layer after spraying

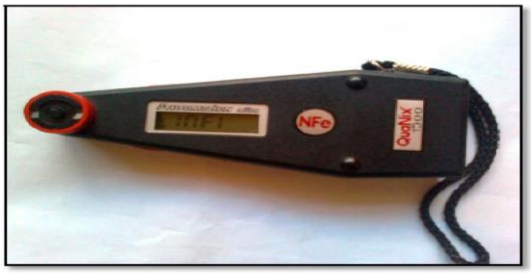

Figure 4: Coating thickness gage.

Contact Angle Measurement:

The contact angle was measured at the Department of Chemical Engineering, University of Technology using an equipment type (CAM 110) manufactured in Taiwan as shown in Fig. 5 according to ASTM D 5946 standards.

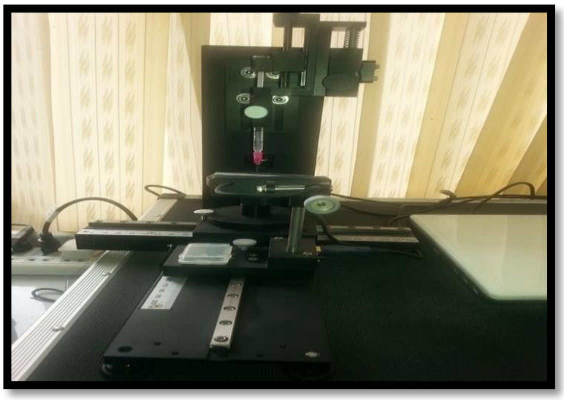

Figure 5: Contact Angle Measurement.

Shore D Durometer Hardness

Shore D Durometer Hardness instrument as shown in Fig.6 was used to carry out the hardness test using pointed dibbing tool following ASTM D2240 standards.

Figure 6: Durometer Hardness instrument of type (Shore D).

Adhesion Strength test

Adhesive examination for coating layer was made by using a tensile tester as shown in Fig.7 at the Institute of Oil/Oil Ministry. The adhesive (epoxy) type 105 was used for adhesion of pieces together (coated and uncoated).

Figure 7: Tension Machine for flat specimen

Porosity Measurement

The specimen was polished, etched and then the specimen was subjected to sputtering coating of (Au-pd) for two minutes. The Pore size measurement was conducted at the Ferdouesi

University /Mashhad Iran using scanning electron microscopy (SEM) as shown in Fig.8.

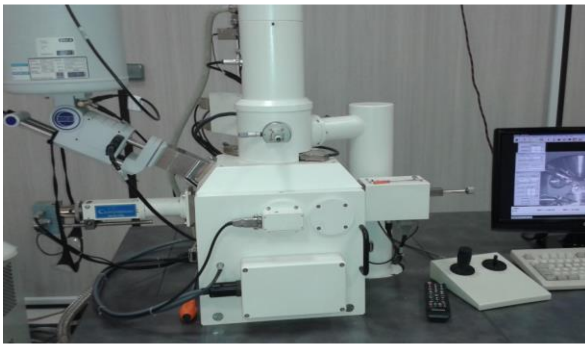

Figure 8: scanning electron microscopy.

Atomic Force Microscopy

AFMs were performed to reveal the surface morphology of the coatings at the micro and Nano level Fig.9 show of AFM at the College of Science / University of Baghdad.

Figure 9: Atomic force microscope (AFM).

Results and Discussions

Corrosion Test

The result indicate that coating low carbon steel with epoxy by spray pyrolysis reduced corrosion rate by (89.4%) ,while with epoxy composite reinforcement at 2%wt of (Al,TiO_2,Zn) by (73.42%,91.75%,97.9%) respectively . The additive of powder to epoxy bring the benefits it terms of reduced corrosion rate, the corrosion protection is attributed to polymer coating (physical barrier), and also the increase of the porosity increases the corrosion rate [7]

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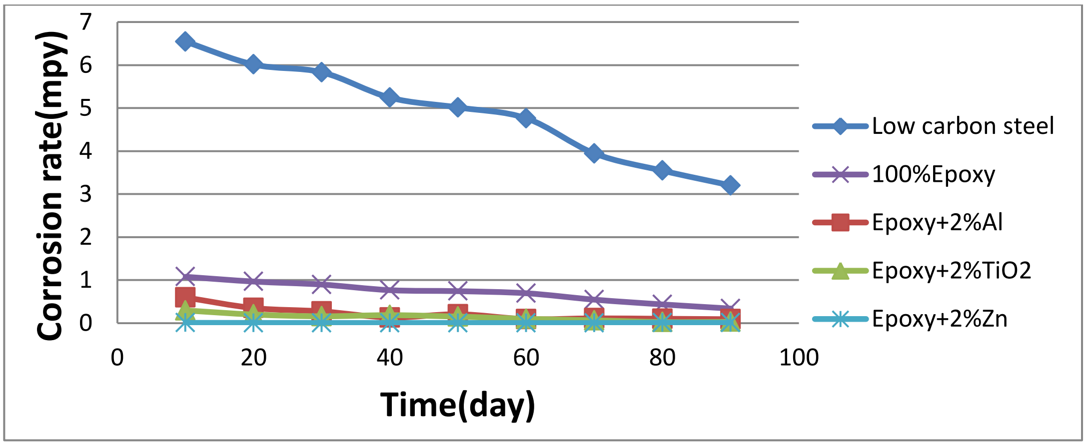

Figure 10: Corrosion Rate of Spray Pyrolysis.

Erosion Test

The weight loss of low carbon steel coated with epoxy at90ºand 30^0 impingement angles are lower than those of uncoated specimens of low carbon steel by (51.06%,43.2%) respectively ,while the weight loss of erosion characteristics at90ºand $30^0$of coated specimens with epoxy composites reinforcement 2% (Al, TiO_2) lower than coated specimens with epoxy by (34.78%,17.39%)and(47.61%,23.80%) respectively, and the weight loss of erosion characteristics at90º,30^0 in coated specimens with epoxy composites reinforced(Zn) are higher than coated specimens with epoxy by (34.78 % , 28.57%) respectively . The coated specimens with composite materials of (Al) has less erosion wear than other types of coated specimens, due to the shape of (Al) particles beings more regular than other which leads to increasing the bonding between the particles and polymer matrix show Fig. (11, 12).

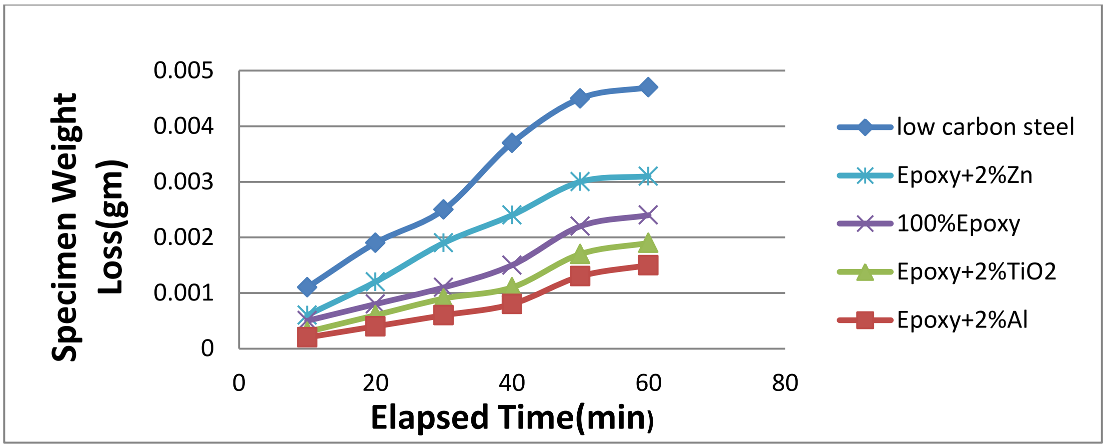

Figure 11: The weight loss with Elapsed time for low carbon steel and coated materials at 90° and impact velocity 30m/s by spray pyrolysis

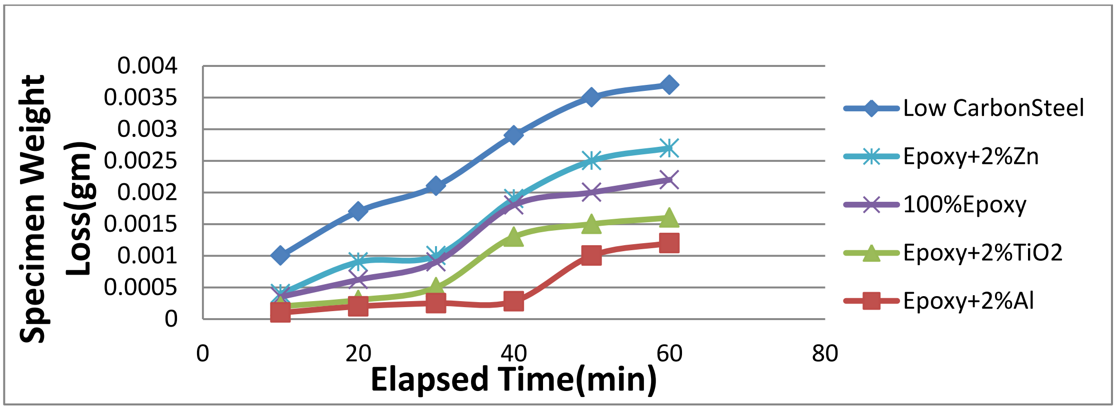

Figure 12: The weight loss with Elapsed time for low carbon steel and coated materials at 30° and impact velocity 30m/s by spray pyrolysis.

A statistical model (SPSS) for the prediction of the coating properties was created by regression function in SPSS software from the training data set

Table 1: Training data for weight loss of spray coating

No	Time (min)	Angle	Measured A_o	Predicted A_o	Measured A_1	Predicted A_1	Measured A_2	Predicted A_2	Measured A_3	Predicted A_3	Measured A_4	Predicted A_4
1	10	90	0.001	0.00111	0.0002	0.00005	0.0003	0.00027	0.0006	0.00072	0.0005	0.00039
2	30	90	0.002	0.00218	0.0006	0.00064	0.0009	0.00092	0.0019	0.00174	0.0011	0.0012
3	40	90	0.0028	0.00272	0.0008	0.00093	0.0011	0.00125	0.0023	0.00226	0.0015	0.0016
4	50	90	0.0035	0.00326	0.0012	0.00122	0.0017	0.00157	0.003	0.00277	0.0022	0.002
5	10	30	0.001	0.00104	0.0003	0.00035	0.0002	0.00007	0.0004	0.00029	0.0003	0.00029
6	20	30	0.0017	0.00158	0.0006	0.00064	0.0003	0.00039	0.0009	0.0008	0.0006	0.0007
7	30	30	0.002	0.00211	0.0009	0.00094	0.0005	0.00072	0.001	0.00131	0.0009	0.0011
8	50	30	0.0034	0.00319	0.0017	0.00152	0.0015	0.00137	0.0025	0.00233	0.002	0.0019
9	60	30	0.0034	0.00373	0.0019	0.00182	0.0016	0.0017	0.0027	0.00285	0.0022	0.00231

Table 2: Testing data for weight loss of spray coating

No	Time (min)	Angle	Measured A_o	Predicted A_o	Measured A_1	Predicted A_1	Measured A_2	Predicted A_2	Measured A_3	Predicted A_3	Measured A_4	Predicted A_4
1	20	90	0.0017	0.00164	0.0004	0.00034	0.0006	0.00059	0.0011	0.00123	0.0008	0.0008
2	60	90	0.0037	0.00379	0.0015	0.00152	0.0019	0.0019	0.0031	0.00328	0.0023	0.00241
3	40	30	0.0028	0.00265	0.0011	0.00123	0.0012	0.00105	0.0019	0.00182	0.0018	0.0015

The values of the multiple correlation coefficients R, that tells how strongly the multiple independent variables are related to the dependent variable, were (0.984, 0.984, 0.979, 0.984, and 0.980) .The figures (13, 14, 15, 16, and 17) show the comparison between the predicted and measured values of two angle 90º and 30º and for 60 minute time of erosion test in the each angle.

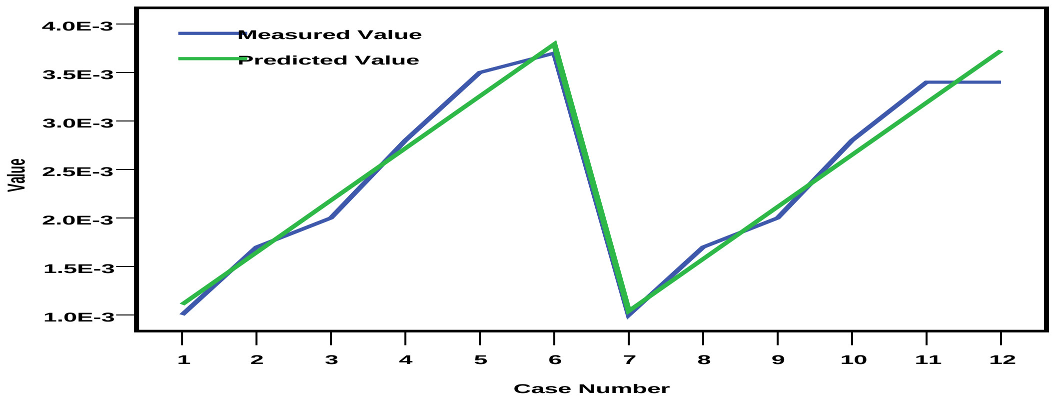

Figure 13: Comparison between measured and predicted values for the experimental data of erosion rate for Low carbon steel (caseA_o).

Figure 14: Comparison between measured and predicted values for the experimental data of erosion rate for Epoxy+2%Al spray pyrolysis (caseA_1)

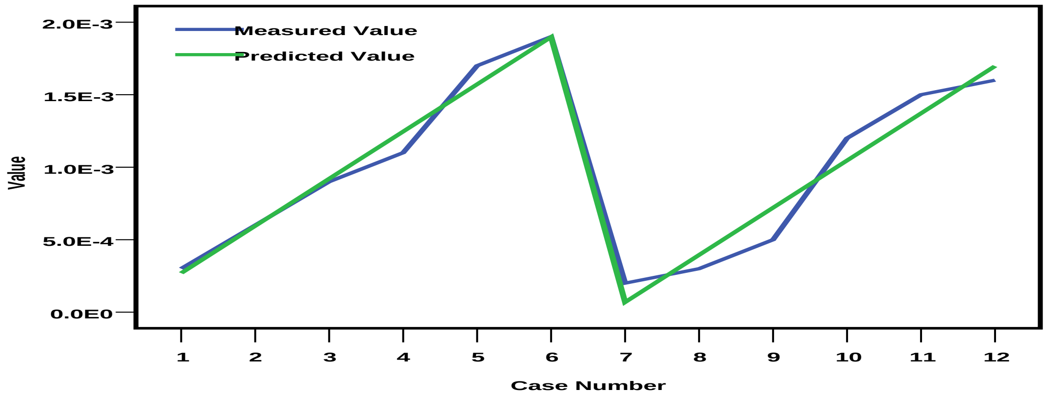

Figure 15: Comparison between measured and predicted values for the experimental data of erosion rate for Epoxy+2%TiO_2spray pyrolysis (caseA_2)

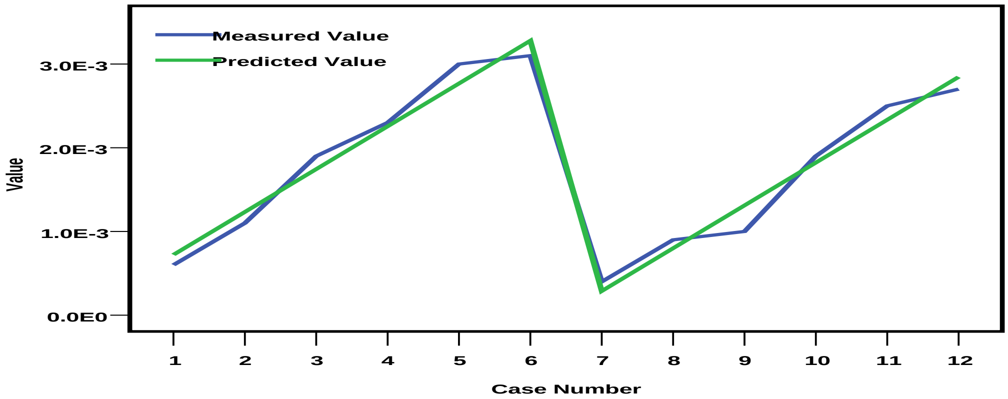

Figure 16: Comparison between measured and predicted values for the experimental data of erosion rate for Epoxy+2%Znspray pyrolysis (caseA_3)

Figure 17: Comparison between measured and predicted values for the experimental data of erosion rate for 100%Epoxy spray pyrolysis (caseA_4)

Shore D Hardness:

The specimen coated with epoxy composite reinforcement with 2%wt (Al, TiO_2) have an increase in hardness than coated specimens with epoxy by (14.70%, 11.76%) respectively ,and the coated specimen with epoxy reinforcement with 2%wt Zn are decreased hardness than coated specimens with epoxy by(3.52%). The increasing of hardness, due to the presence of homogeneity in the form of particle shape which are more spherical and bonding with epoxy than other a addition the aggregation of particles led to decrease the hardness. show table 3 hardness before and after erosion.

Table 3: Shore D Hardness of spray pyrolysis before and after erosion wear

Materials	Shore D Hardness Spray pyrolysis	
	Before erosion wear	After erosion wear
1-Epoxy+2%Al	97.5	98.5
2Epoxy+2%TiO_2	95	97
3- Epoxy+2%Zn	82	88
4- Epoxy100%	85	86

Adhesive strength:

The adhesive strength of coated specimens with reinforcements of 2 %wt (Al, TiO_2, Zn) lower than the adhesive strength of coated specimen with epoxy by (25.42%, 52.76%, 40.1%) respectively. The result adhesive strength of the coated specimens with epoxy further more than all specimens coated with composites, due to the presence of oxygen containing polar group (OH) on the polymer surface and bonding with the surface of substrate .As shown fig. (18) below.

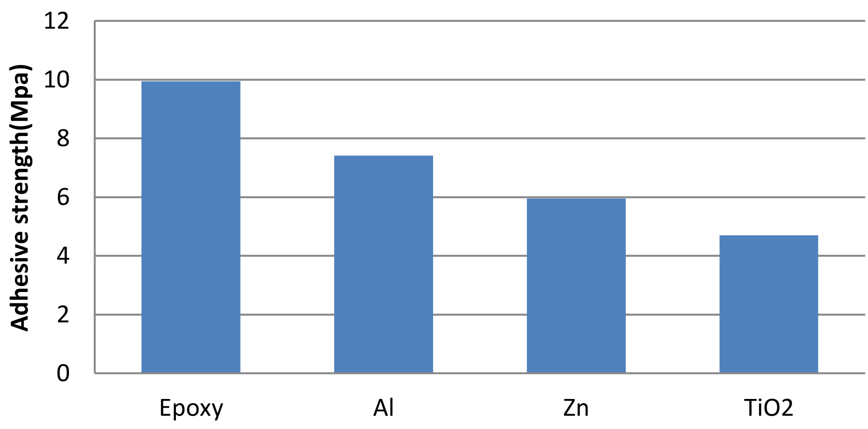

Figure 18: Adhesive strength (Mpa) of spray pyrolysis.

Atomic Force Microscopy

The roughness average of coated specimen with epoxy composites reinforced with 2%wt (Al,TiO_2,Zn) are lower than the roughness average of specimens coated with epoxy by (87.61%,94.11%,90.83%) respectively. The surface roughness tests result are shown in table (4) and figure(19)that indicate all coated specimens with composite material have significantly lower surface roughness than coated specimen with epoxy matrix . The adding particle filler which tends to occupy voids in thin film coating and serve as the bridges inter connected matrix

Table 4: Roughness average of spray pyrolysis coating

Spray Pyrolysis	
Coating type	**Roughness Average(nm)**
1-Epoxy+2%Al	0.223
2-Epoxy+2%TiO2	0.106
3-Epoxy+2%Zn	0.165
4-Epoxy 100%	1.8

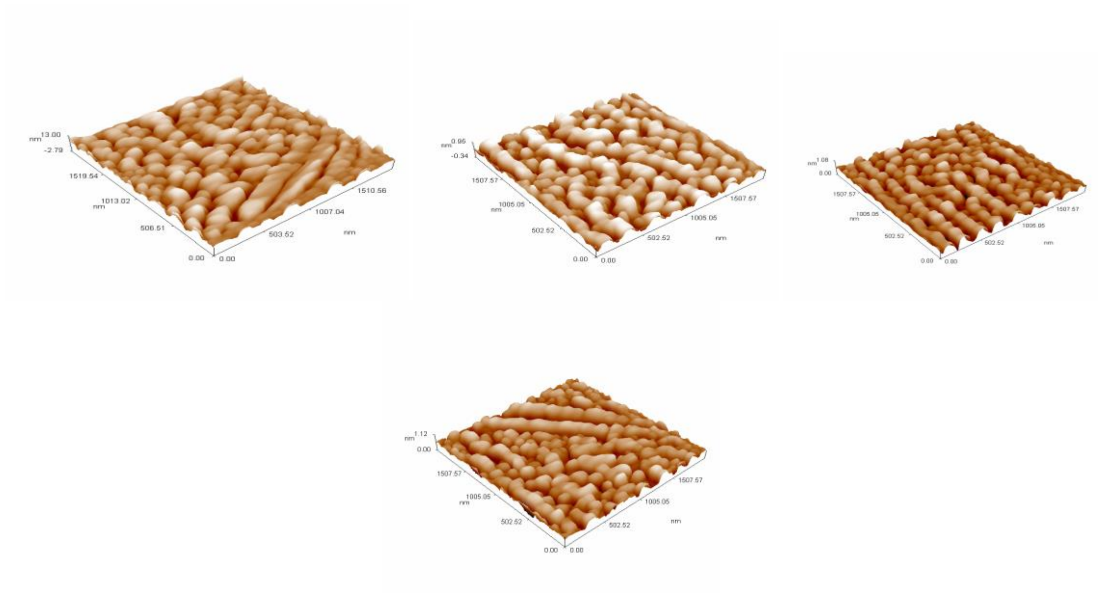

Figure 19: Atomic force microscopy of epoxy, aluminum, titanium dioxide and zinc respectively.

Contact Angle Measurement

The contact angle of specimen coated with epoxy are (63º) and coated specimens with epoxy composites reinforcement (Al, TiO_2, Zn) by (64º, 67.69º, 66º) respectively. The contact angle decreased due to increasing the roughness of coating materials on the substrate and increasing of the adhesion strength. Show figure20 to 21.

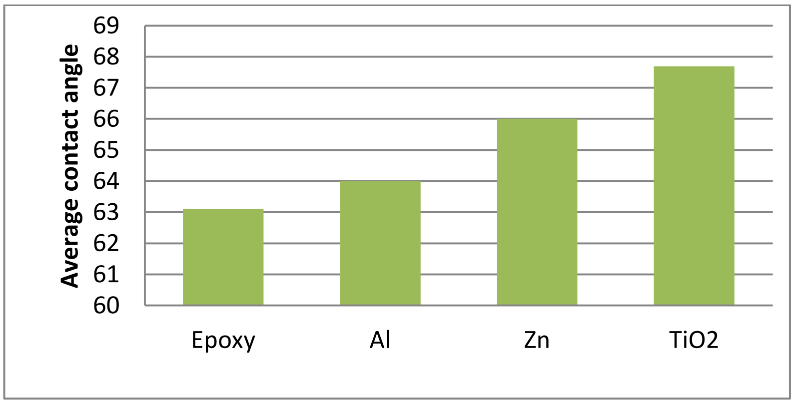

Figure 20: Average Contact Angle of spray pyrolysis

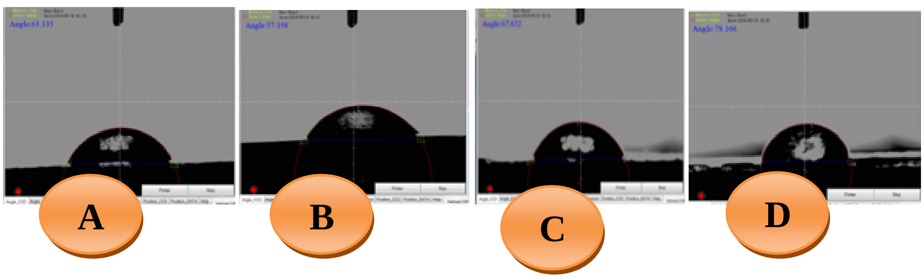

Figure 13: photograph Average contact angle A:Epoxy spray, B:Alspray, C:Znspray, D:Tio_2 spray.

Porosity Measurement:

The results show the pore size of coating specimens with epoxy composites reinforcements

(Al, TiO_2, Zn) are lower than coating specimens with epoxy by(56.80%,77.18%,90.52%) respectively show table5 below.

Table5: pore size of spray pyrolysis.

Spray pyrolysis	
Materials	**Pore size**
1- Epoxy+2%Zn	186.6 nm
2- Epoxy+2%TiO_2	449.5 nm
3-Epoxy+2%Al	850.9nm
4-Epoxy 100%	1970nm

Conclusions

1- Corrosion rate of coated specimens with epoxy composites reinforcement (Al, TiO2, Zn) are lower than coated specimens with epoxy by (73.42%,91.75%,97.9%) respectively. while that coating low carbon steel with epoxy reduced corrosion rate by (89.4%) respectively as compared to low carbon steel .

2- The weight loss of coating low carbon steel with epoxy at 90º and 30º by (51.06%, 43.2%) respectively and the weight loss at90ºand 300of coated specimens with epoxy composites reinforcement (Al, TiO2) lower than coated specimens with epoxy by (34.78%,17.39%) and (47.61%,23.80%) respectively.

3- The weight loss at90º, 30º of coated specimens with epoxy composites reinforced (Zn) are higher than coated specimens with epoxy (34.78 %, 28.57%) respectively.

4- The hardness of (shore D) of coated specimens with epoxy composites reinforcement (Al, TiO_2) are higher than

5- coated specimen with epoxy by (14.70%, 11.76%)respectively, While the hardness of coated specimens with epoxy composites reinforcement (Zn) are lower than coated specimen with epoxy by (3.52%) respectively.

6- The adhesive strength of coating specimens with epoxy composites reinforcements (Al, TiO_2, Zn) are lower than coating specimens with epoxy by(25.42%,52.76%,40.1%) respectively.

7- The pore size of coating specimens with epoxy composites reinforcements (Al, TiO_2, Zn) are lower than coating specimens with epoxy by (56.80%,77.18%,90.52%) respectively.

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الرش بالانحلال الحراري للفولاذ المنخفض الكربون بواسطة مادة مركبة ذات اساس طلاء بوليمر

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الخلاصه:

في هذا البحث تم دراسة تأثير طريقة الطلاء بالرش الانحلال الحراري على الفولاذ منخفض الكربون مع الايبوكسي تقل معدل التاكل اقل بنسب(89.4%)، بينما العينات المطلية بالايبوكسي مع 2 % من(Al, TiO2,and Zn) كانت اقل من العينات المطلية بالايبوكسي بنسبة (73.42%,91.75%,97.9%) على التوالي.اظهرت النتائج الفقدان بالوزن لاختبار التعرية للعينات المطليه بالايبوكسي فكان مقدار الانخفاض بالوزن بزاوية 90و 30 درجة للطلاء بالرش الانحلال الحراري كانت اقل بنسبة (51.06%,43.2%) مقارنة بالعينات الغير مطلية من الفولاذ المنخفض الكاربون، بينما الفقدان بالوزن بزاوية 90 و 30 درجة للعينات المطليه بمواد مركبه والمدعمه(Al, TiO_2) كانت اقل مقارنة بالعينات المطلية بالايبوكسي بنسبة (34.78%,17.39%) و (47.61%,23.80%)على التوالي.الفقدان بالوزن لخاصية التعرية بزاوية 90و 30 درجة للعينات المطليه بمواد مركبه مدعمه 2% من (Zn) اعلى من العينات المطلية بالايبوكسي بنسبة (34.78 %, 28.57%) على توالي. تم تقدير الترطيب لسطوح العينات المطلية فكانت اقل من 70 درجه اي السطح يمتلك درجة ترطيب جيده وطاقة سطح عاليه. اظهرت النتائج ان كافة العينات المطليه بالمواد المركبه والمحضره بالطلاء بالرش الانحلال الحراري قوة التلاصق بين طبقة الطلاء وسطح الفولاذ منخفض الكربون اقل مما عليه للعينات المطليه بالايبوكسي .اظهرت قياسات سطوح العينات المطلية بالايبوكسي والمواد المركبة، بواسطة مجهر مطياف القوة الذرية (AFM) تمتلك زيادة في الخشونة. وباستخدام المجهر SEM كان حجم المسامات لسطوح العينات المطليه بالمواد المركبه اقل مما عليه للعينات المطليه بالايبوكسي.