© 2018 IJSRST | Volume 4 | Issue 2 | Print ISSN: 2395-6011 | Online ISSN: 2395-602X
International Journal of Scientific Research in Science and Technology

Study of the corrosion inhibitors using the Field Emission Scanning Electron Microscope and Scanning Electron Microscope : A Mini-Review

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Abstract - The application of Field Emission Scanning Electron Microscope and Scanning Electron Microscope in the investigation of corrosion inhibitor for metal in acidic environment has reviewed. In this paper the detail procedures of FE-SEM and SEM method to study corrosion inhibition properties of corrosion inhibitors for metal in acidic environments are discussed, and the possible conclusion is made based on the results of several articles. It is summarized that FE-SEM and SEM is an important method to determine the corrosion inhibition prpoperties and the mechanism of corrosion protection of corrosion inhibitors.

Keywords: FE-SEM, SEM, Corrosion inhibitors, Metal.

Introduction: Corrosion of metals, alloys and other materials under the influence of environment is very grave issues worldwide. The cost of corroision related damage and maintenance is overwhelming. According to studies, losses due to corrosion cost trillion of dollars each year, accounting significant percentage of a country's Gross Domestic Product (GDP).[1] The use of corrosion inhibitors is one of the most commonly used method to protect metal from corrosion especially in corrosive environment like hydrochloric acid, sulpuric acid etc. In principle, corrosion inhibitor molecules get adsorbed on the metal surface either by physorption and, or chemisorption mechanism and form protective barrier.[2-4] These protective barrier protect metal surface from aggressive attack of corrosive environment. The inhibitor molecules having electron donar atoms, pie orbitals, high electron moiety serve as potential adsorption sites which share electrons with the metal substrate surface. The corrosion inhibition study using corrosion inhibitors has been undertaken by various researchers, scientists etc. There are various methods that have been used in recent time to study anticorrosive effect of corrosion inhibitors like weight loss method, electrochemical method (Polarisation study, AC impedance), quantum chemical calculation method, Surface morphological study: Field Emission Scanning Electron Microscope (FE-SEM), etc. In recent advances in studying corrosion inhibitors, the FE-SEM method has attracted the attention of researchers, scientists, academicians, etc. It has emerged as a powerful technique to study the ability of corrosion inhibitors to protect metal surfaces in the presence of a corrosive environment.[6-9]

The primary objective of this paper is to present a mini-review of the application of the FE-SEM and SEM techniques in the study of corrosion inhibition ability of the inhibitors.

2. Field emission scanning electron microscopy: Field emission scanning electron microscopy is an advanced technology or in more simple form, is an improved version of SEM (Scanning electron microscope). It *provides high-resolution images of the surface morphology of the materials.* FE-SEM is typically performed in a high vacuum because gas molecules tend to disturb the electron beam and the emitted secondary and backscattered electrons used for imaging.

Received : 03 Jan 2018 | Accepted : 15 Jan 2018 | January-February-2018 [(4) 2: 2333-2336]

3. Advantage of Field emission scanning electron microscopy (FESEM):

(a) It provides two-dimensional topography of materials which is used to study microstructure of the materials. It provides topographical and elemental information at magnifications of 10x to 300,000x, with virtually unlimited depth of field.

(b) FE-SEM Compared with conventional scanning electron microscopy (SEM), FE-SEM produces clearer, less electrostatically distorted images with spatial resolution down to 1 1/2 nanometers - three to six times better.

(c) FE-SEM can *examine minor area contamination spots.*

4. FE-SEM Vs. SEM: Benefits and Limitations

- FE-SEM is more advanced technology than SEM while both provides two dimenstional topography of the materials that is used to study surface morphology of the materials.
- FE-SEM provides more clearer, less electrostatically distorted high resolution images than SEM.
- In the SEM method, a resolution of 3-7 nm is reachable, while in the FE-SEM the resolution is 1.5 nm or better.
- The conductive layer coated on a sample to be subsequently imaged by FE-SEM must be very thin and uniform and have finer grain size compared to that is required for SEM imaging.
- Better contrast in low-density materials imaging through FE SEM technique is possible.
- In FESEM, the electron source requires higher vacuum environment (higher than 10^{-9} Torr) during operation to ensure electron stability and prevent contamination of the cathode.
- FE-SEM electron sources suffer low beam current stability

5. **Corrosion inhibitor :** A corrosion inhibitor is a substance when added in small concentration to the corrosive environment that reduces the rate of corrosion by reducing the aggressive attack of corrosive environment to the metal. The use of inhibitor is one of the most economical and commonly used method to protect the metal from corrosive environment.

6. Material and sample preparation for FE-SEM image to evaluate corrosion inhibition:

6.1 Preparation and cleansing of the metal surface : Metals which is under consideration for corrosion experiment like mild steel is cut into the small pieces of dimension like 3 cm × 2 cm × 0.3 cm or 3 cm × 3 cm × 3 cm etc. Prior to each experiment the metal surface was abraded with emery papers (grade 320 to 1200), washed with double distilled water and dried with cool air. [11, 12]

6.2 Preparation of Test solution: The analytical grade of corrosive solution like HCl, H_2SO_4 etc. in which the protection ability of corrosion inhibitor is to study is taken and it is diluted with double distilled water upto the the required strength in which experiment is to carried out.

6.3 Sample Preparation for FE-SEM image : The fixed volume of experimental corrosive solutions are taken in the different beakers then corrosion inhibitor is added to these beakers containg experimental corrosive solution with varying concentrations except one beaker which is to keep blank experimental solution without inhibitor. Now at the given temperature usually room temperature (or experimental requirment) the metal samples for FE- SEM analysis prepared as above then immersed in each of the beaker having experimental solution in the presence and absence of the inhibitos for the given period of time and then taken out from the test solution, cleaned with doubly-distilled water followed by acetone and dried with cool air. Finally the FE-SEM images are captured by FE-SEM Supra 55 (Carl Zeiss, Germany) etc. at different resolution and magnification levels.

6.4. Analysis of FE-SEM image : FE-SEM is a powerful tool to study qualitative interaction between the inhibitors and the metal surface. FE-SEM provides the two dimenstional topography of the metal specimens and then surface morphology of theses metal specimens are studied. It gives important information about the inhibition ability of the studied inhibitors. In the absence of inhibitor, the surface morphology of the metal surface becomes very rough with numerous pits or crack owing to rigrous attack by corrosive medium. while in the presence of the inhibito with different concentrations, the surface remain almost smooth or crack free as compared to blank solution. If this type of results obtained then it indicates that the studied inhibitor has protecting abilty of the metal from corrosive environment. In some cases the smoothnes of the metal surface increase with increasing inhibitor concentrations. It indicates that the protection ability of the inhibitor increases as increase in inhibitor concentration. It is belived that corrosion inhibitor molecules adsorb on to the metal surface through the donar atom or atom having high electron density of the inhibitor molecules either by physisorption or chemisorption mechanism and provides a protective layer on the metal surace that reduces the aggressive attack of corrosive solution. In this way the inhibitor protects the metal surface.

7. Experimental studies of corrosion inhibitor performance by FE-SEM: Literatur survey revealed that FE-SEM method along with other methods like gravimetric method, Potentiostatic polarization measurements has been become an important tool to explore corrosion inhibition perforfance of the corrosion inhibitors as well as mechanism of corrosion inhibition.

M.J. Bahrami et al. studied the Experimental and theoretical investigation of organic compounds as inhibitors for mild steel corrosion in the sulfuric acid medium by potentiostatic polarization, FTIR spectroscopy, and SEM techniques.[13] In the absence inhibitor i.e in a blank acid solution metal surface morphology is strongly damaged (uniform and pitting corrosion) while in the presence of an inhibitor, the rate of corrosion is significantly decreased. It is found that in the presence of an inhibitor the adsorbed layer is formed that protects the material surface. The SEM results are in good agreement with electrochemical measurements.

Resit Yıldız studied inhibiting effect of 4,6-diamino-2-pyrimidinethiol (4D2P) on mild steel corrosion (MS) in 0.1 M HCl solution by linear polarization resistance, electrochemical impedance spectroscopy and potentiodynamic measurements in the presence of different concentrations of 4D2P ranging from 10.0 mM to 0.5 mM. The effect of 4D2P on the MS corrosion was also studied by scanning electron microscopy. Electrochemical meassurments revealed revealed that the corrosion inhibition efficiency of 4D2P inhibitor is 95.8 % at 10 mM inhibitor concentration. The SEM image of metal surface in the absence and the presence of the inhibitor showed that surface morphology of the metal surface in the presence of the inhibitor 4D2P remain smooth with very less no. of pitting as compared to that in the absence of the inhibitor.SEM images also explore that 4D2P is good corrosion inhibitor.[14]

Bin Xu et al[15] investigated two pyridinecarboxaldehyde thiosemicarbazone compounds as corrosion inhibitors for mild steel in hydrochloric acid solution by electrochemical techniques, SEM and theoretical calculation methods. The experimental results show that the inhibition efficiency of 2-pyridinecarboxaldehyde thiosemicarbazone (2-PCT) is higher than that of 4-pyridinecarboxaldehyde thiosemicarbazone (4-PCT), and both the two compounds are good inhibitors for mild steel in 1.0 M HCl. The SEM images of mild steel specimens revealed that the surface morphology was severely corroded in the absence of inhibitors. However in the presence of inhibitors, the damage of mild steel surfaces was reduced and even the polishing lines can be observed. This observations clearly prove that 2-PCT and 4-PCT can exhibit good inhibiting ability on mild steel surface. The inhibitors may form protective films adsorbed on metal surface, resulting in a decrease in the contact between iron and the aggressive solution.

Gökmen Sı gırcık et al [16] investigated the inhibition efficiency of benzonitriles with functional amine groups in different positions, for mild steel corrosion in 0.5 M HCl solution. For this purpose, electro-chemical impedance spectroscopy and potentiodynamic measurements were realized. The surface analysis was also carried out by scanning electron microscopy technique. The SEM results were found in good agrrement with that of electrochemical results.

V.V. Torres et al[17] investigated the behaviour of 1,3-dibenzylthiourea (DBTU) and 1-benzyl-3-diisopropylthiourea (DPTU) as carbon steel corrosion inhibitors in HCl solution by weight loss measurements, potentiodynamic polarization curves, electrochemical impedance and scanning electron microscopy analysis.

8. Conclusions: FE-SEM and SEM is an important and reliable tool to explore the corrosion inhibition properties and the mechanism of corrosion inhibition of corrosion inhibitors.

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