

Assessment of steel corrosion caused by pre-existing microorganism in produced water from an oil and gas facilities and its inhibition using glutaraldehyde

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Abstract

Microbiologically induced corrosion (MIC) is defined as corrosion caused by the activity of microorganisms which contribute to the high annual cost in oil and gas industries. Abundant microbial flora can be found in oil field formation waters which makes MIC inevitable. This brief preliminary study involves the growth of the microbes obtained from produced water sample from an oil and gas facility. Their corrosion activities on AISI 1018 carbon steel were also studied, including the utilization of glutaraldehyde as a corrosion inhibitor. The microbial growth was conducted on solid agar and liquid nutrient medium. The bio screening procedures were done by measuring the optical density using spectrophotometer and measurement of microbe dry weight. Laboratory scale corrosion test was conducted using weight loss method in simulated field condition. Glutaraldehyde was added in varying concentrations from 50 to 200 ppm to determine its effective dosage. The growth of colourless bacteria was observed in the agar medium, validated by the increased optical density in the liquid broth. The colourless bacteria were suspected as sulphur oxidizing bacteria (SOB) which is the main type of microbe that occurs in water. SOB induces corrosion in metal pipelines due to its oxidation reaction of sulphur compounds. The corrosion rate measured was 0.59 mm/year. The addition of 200 ppm of glutaraldehyde recorded the lowest corrosion rate which was 0.24 mm/year. It can be concluded that water sample from oil and gas field contained bacteria that causes MIC and can be controlled using glutaraldehyde, which has strong antibacterial and antifungal properties.

Article Info

<https://doi.org/10.24191/mjct.v4i1.10946>

Article history:

Received date: 8 August 2020

Accepted date: 23 April 2021

Published date: 30 April 2021

Keywords:

Biocide
Microbiologically-influenced corrosion
Pipeline
Sulphur-oxidizing bacteria
Glutaraldehyde

1.0 Introduction

Over the past years microbiologically induced corrosion (MIC) has become a major problem under varied service conditions, and on different materials. Also known as biocorrosion, MIC is related to the reduction of metal by corrosion processes, which involves microorganisms (Yahya et al., 2011; Nik Him and Ahmad Robert, 2018). The main source of microorganism can arise from the original reservoir or from injected water (Yun and Huap, 2010; Nik Him et al., 2018). Bacteria can multiply rapidly into colonies, which lead to the generation of pitting and galvanic corrosion, plugging of surfaces and downhole equipment, including the injection-well formations, and finally will promote corrosion of surface piping and downhole tubulars (Mahat et al., 2012; Abdullah et al., 2014). In general, MIC can be categorized into two groups based on their oxygen requirements: aerobic

bacteria, such as sulphur-oxidizing bacteria (SOB) and anaerobic, for example sulphate reducing bacteria (SRB). Both can communicate and run oxidation-reducing processes that will cause the dissolution of the protective and dense oxide layer on metal surfaces, replacing it with a less protective and porous sulphide layer (Valencia-Cantero and Peña-Cabriales, 2014). MIC occurs frequently in pipelines and can be divided into three stages. The first stage is the formation of film and biofilm with hydrogen permeation by redox reaction. This stage is dominated by general galvanic corrosion. Stabilization of film, biofilm and the metal, happened in stage two, while stage three is where the detachment of film and biofilm occur, which leads to localized galvanic corrosion (Popoola et al., 2013).

Most industries recommended the usage of biocide in chemical method for controlling biocorrosion in their systems. Biocide can be defined as an additive that eliminates microorganism. Glutaraldehyde is one

of the most commonly used biocides in the oil and gas industry due to its strong antibacterial and antifungal activity. Other advantages of using this biocide include low production cost, noncorrosive, effective at low concentrations, its solubility in water, and it is effective in large pH range (Simões et al., 2011; Lin et al., 2013; Xu et al., 2017; Wentworth et al., 2018). The biocidal activity of glutaraldehyde is by binding strongly to the outer cellular layers of the bacteria, which then alters the RNA, DNA and protein synthesis, and finally leads to the death of the bacteria (Maillard, 2002; McDonnell, 2007; Maillard, 2018).

Produced water must be regularly tested to mitigate the occurrence of MIC in pipelines. However, the task to constantly test and treat the produced water is tedious and costly. Therefore, corrosion mitigation can be planned properly by using biocide at the right dosage. Among the scope of this short study is to determine the existence of certain types of microorganism, specifically SOB, in produced water that can induce corrosion. The study then focussed on the corrosion that is induced by the microorganism on steel pipelines, and subsequently to determine the optimum dosage of glutaraldehyde to inhibit the corrosion caused by the microorganisms.

2.0 Methodology

The first part of the study focused on bio screening test and to ensure bacteria growth in compatible environments. This consisted of culturing process, incubation, and aseptic techniques.

The second part of the study focused on corrosion inhibition tests to investigate the optimum biocide concentration that will result in low corrosion rates of carbon steel.

2.1 Material

Bacteria sample was isolated from a produced water storage tank of an oil and gas production facility in Malaysia. Two media types were used for the isolation process which were solid nutrients agar, NA and liquid nutrients broth, NB (Merck). Steel coupon AISI 1018 carbon steel plate (127 mm x 25.4 mm x 2.54 mm) was used for the corrosion tests.

2.2 Sample Sampling

Untreated water sample from a produced water storage tank of an oil and gas production facility was collected in a 100 mL sterile bottle to ensure no

contamination. The sample bottles were stored at low temperature in a dark storage container until testing.

2.3 Media Preparation

Two types of media were prepared for the samples: solid media nutrients agar and nutrients broth. The nutrient agar solution was prepared by mixing 10 grams of nutrient agar powder (Merck) with 500 mL distilled water. The nutrient broth solution was prepared by mixing 1.2 g broth powder (Merck) with 150 mL distilled water. Both solutions were heated and stirred at 600 rpm to ensure complete dilution of the agar nutrients powder in the solution. Both agar and the broth solution were then poured into sterile bottles. The pH value was adjusted to be the same average pH value of the bacteria sample which was 6.72.

An autoclave chamber was utilized to sterilize the nutrient agar and nutrient broth solutions from bacteria, spores, and germs. The two prepared solutions, as well as other equipment used in the experiment were subjected to high-pressure saturated steam at 121°C for 15 to 20 minutes and were then left to cool in the autoclave for about 3 hours.

2.4 Culturing and Incubation Process

The autoclaved nutrient agar solution was poured into the eight petri dishes equally and left to harden for about 15 minutes. Sterile loop was immersed into the bacteria sample and a loopful of culture was swap over the surface of the cooled and hardened agar plate (Bhagobaty, 2014).

The autoclaved nutrient broth solution was transferred into sterile bottles. Each bottle contained 20 mL of the broth with 1 mL of bacteria sample pipetted into each bottle. The bottles were shaken to ensure the solution broth and the bacteria properly mixed.

The incubation process was done to promote the growth of microbes in the petri dishes and in the broth solution (Little et al., 2001). All petri dishes and the solution broth were kept in the controlled chamber at temperature between 30 to 40°C for about 3 to 5 days. The changes in the petri dishes and in the broth solutions were recorded daily.

2.5 Bacteria Growth Curve

The sterile loop was used to transfer bacterial in the agar plate into 10 mL nutrient broth and shaken properly. The solution was kept in the controlled chamber for incubation process for 24 hours.

After 24 hours of incubation process, the solution was taken out and mixed into 90 mL of nutrient broth solution. Zero hour started once this step was applied. Immediately, 1 mL of the solution was transferred into a cuvette while another 1.5 mL was transferred into an Eppendorf tube. The dry weight of an Eppendorf tube was measured and recorded as initial weight. Next, the tube was centrifuged at 4°C and 10000 rpm for about 20 minutes until there were sediments at the bottom. Then, it was taken to the oven for drying process for 24 hours. Once the sediments were dried enough, the weight were measured and recorded. The steps were repeated for every 24 hours, till the dry weight of each sediments increasing and stopped when it was slightly constant as recommended by (Tanner, 1989). The graph of dry weight versus time was plotted.

Optical density, measured in a spectrophotometer, was used to measure of the concentration of bacteria in a suspension. As visible light passes through a cell suspension the light was scattered. Greater scatter indicates that more bacteria or other material was present (Widdel, 2010; Javed et al., 2017). The same procedure was done for 1 mL cuvette but using the spectrophotometer to measure the turbidity value for each cuvette. The initial reading for optical density of absorbance for each cuvette was recorded. The reading was continuously taken and recorded for every 24 hours until the reading for a cuvette increasing and stopped once it was slightly constant. All measurement was set at 560 nm wavelength and using the broth as blank solution. The graph optical density (OD) versus time was plotted.

2.6 Sample preparation for corrosion test

The first step was the preparation of the nutrient broth where four grams of the nutrient broth powder was weighted to be diluted with 500 mL of distilled water. It was then heated to 70°C at 700 rpm until the solution become clear, before being autoclave at 121°C for 15 to 20 minutes and left to cool for three hours. After the autoclave process, 100 mL of the produced water sample was poured into the broth solution, shaken well and incubated in the controlled chamber for 24 hours. Sulphur was prepared to complete the corrosion mechanism in the broth medium. Sulphur was first prepared by mixing 10 g of sulphur powder with a few drops of distilled water in a test tube. The mixture was then closed tight to ensure no other chemical reaction could happened and sterilized in a water bath at 95°C to avoid melting point of the sulphur

(Jacob, 2013). After the sterilization process, the sulphur was taken out for use in the corrosion tests.

2.7 Corrosion test

The procedure was designed to investigate the corrosion inhibition efficiency of glutaraldehyde on SOB with the most reliable data based on the ASTM G1-03 and NACE Standard RP0775-2005 (Anon, 2005; ASTM G1-03, 2017). The chosen coupon was AISI 1018 carbon steel plate (127 mm x 25.4 mm x 2.54 mm). Surface polishing and cleaning of specimens before weighing and exposure were critical to remove any contaminants and corrosion products that could affect the test results. Carbon steel coupons were first polished with silicon carbide sandpaper, (grit number 400 and 600). After polishing, specimens were rinsed with isopropyl alcohol and then was dried in a dry air stream. The clean, dry specimens were measured, weighed to within ± 0.1 mg and recorded. The coupon was installed using a nylon fishing line to eliminate the possibility of galvanic effects resulting from metal-to-metal contact. The coupon was inserted in the vertical position of the test cell.

The corrosion rates of the flat carbon steel coupons were determined by the mass loss method. The coupon was immersed in the solution of overnight grown bacteria culture in nutrient broth. The unit layout was modified to fulfil the objective of the experiment. It consists of a 500 mL glass cell with a cap to cover the top of the cell, a thermometer to monitor the temperature, and a magnetic stirrer that ensures circulation of the model fluid inside the cell. The steel coupons were mounted using fishing line at the centre of the beaker. The cell was placed on a heater to heat up the solution to 40°C. The methods followed were based on ASTM methods (ASTM G1-03, 2017).

Once the temperature was stabilized at 40°C, 50 ppm of biocide was poured into the system and the magnetic stirrer was activated at 300 rpm, where the fluid circulation started. The test time was measured from the moment the cell was filled with the simulation fluid. The coupon was taken out after five days and proceeded with cleaning procedure before the weight loss calculation. The coupon was cleaned according to standard procedure which is to immerse it for 1 hour in a mixture of 200 g sodium hydroxide (NaOH), 20 g granulated zinc and 1000 mL distilled water, at 90°C. Care were required so that uncorroded metal was not removed with the corrosion products. Finally, the coupons were rinsed with isopropyl alcohol or acetone

before dried in a dry air stream and weighed to within ± 0.1 mg.

The data was recorded. The same steps were repeated for another five tests using the same procedure as recommended by the industry laboratory level, including the first three set up with no biocide usage. Then, the average weight loss per year was calculated and recorded. Both procedures were repeated at varying biocide concentrations; 75 ppm, 100 ppm, and 200 ppm of biocide.

The weight loss (mg) was determined, and the corrosion rates were determined using Eq. (1):

$$CR = \left(\frac{Wk}{DA t} \right) \tag{1}$$

where k is a constant factor 87.6 for corrosion rate unit of millimetres/year, W is the weight loss in milligrams, D is the metal density in g/cm^3 , A is the exposure area of the sample in cm^2 , and t is the time of exposure of the metal sample in hours.

The inhibitor efficiency (IE) was calculated using Eq. (2) (Rosli et al., 2019):

$$IE(\%) = \frac{100(CR_{uninhibited} - CR_{inhibited})}{CR_{inhibited}} \tag{2}$$

In general, the efficiency of an inhibitor increases with an increase in inhibitor concentration.

3.0 Results and discussion

3.1 Growth of bacteria from water sample

The appearance of bacteria growth in solid medium (agar nutrient) was observed on Day 6 of incubation. A clear petri dish appeared clear on Day 1 and the presence of bacteria appeared on Day 6 as illustrated by Fig. 1. Bacteria growth was already observable near the corner of the petri dish after 24 hours of incubation, which then grew into a larger distribution. The bacteria is colourless which showed a good indication of the sulphur oxidizing bacteria presence in the water sample (Bharathi, 1989; Okabe et al., 2007; Fenchel et al., 2012; Pokorna and Zabranska, 2015).

The optical density (OD) measurement from the spectrophotometer absorbance values represented the biomass concentration in the liquid media. The media was turbid at the end of the experiment. The turbidity correlates with the cell density or the bacteria concentration as can be clearly seen in Fig. 2. The change in the phases of bacteria growth is noticeable.

The graph shows the different phases in the life of the microorganism, starting with a lag phase, followed by a log phase, shown as the rapid increase in the cell density between Day 2 and Day 4. Stationary phase is the plateau between Day 4 to Day 10, and finally a sharp decrease indicates the death phase of the microorganism.

The nonsignificant growth observed on the first two days was due to the microorganism adjusting and coping with the new environment that it was introduced into. The microorganism came from a nutritionally poor medium and therefore needed to take some time to get adapted with the new environment, which was full of nutrient (Widdel, 2010). Based on the observation, short period was required by the bacteria to suit with the new medium of nutrient.

After Day 2, the sample then showed increase in OD measurement, indicating the increasing rate of bacteria growth, which is termed as exponential phase or log phase. The organism were in the state of rapidly

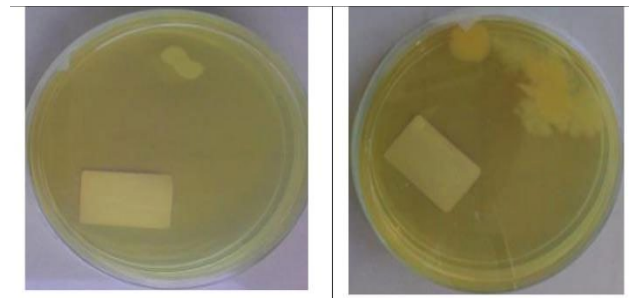


Fig. 1: Bacteria growth on; (a) Day 1 and (b) Day 6.

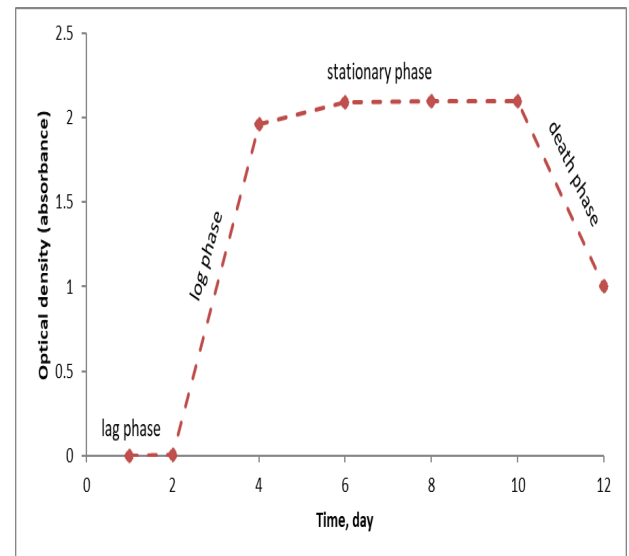


Fig. 2: Optical density measurement in liquid broth in liquid broth media.

growing and dividing, where there were increment of the metabolic activity of the organism, including the starting point of DNA replication process repeated by binary fission at constant rate (Wallden et al., 2016). Furthermore, the nutrients need by the organism were used completely to help the number of bacteria to increase exponentially where the single cell divided into two, replicate into four, eight, sixteen and so on.

The rapid growth halted at around Day 4 when the growth rate started to decrease to an almost constant reading until Day 10. This phase is the stationary phase, indicating the depletion of all the nutrients in the medium had been used by the bacteria during the previous rapid multiplication process. The process caused the accumulation of waste materials, inhibitory compound and the toxic metabolites in the medium. This condition leads to the change of medium environment such as the pH, which introduced the unfavourable environment for the bacteria growth. It caused the reproduction rate to slow down where the new cell divided is equal to the cell that began to die which resulted in constant measurement of the absorbance and finally the bacteria stop to divide (Tang et al., 2009).

The reduction of the nutrients and the presence of accumulation of all the waste product and other toxic in the medium cause the bacteria to head into death phase which explained the decreasing absorbance value from day 10 to 12. The bacteria have no ability to reproduce due to the worst environment and the death was rapid. The culture cannot grow forever,

because of limited amount of nutrient and culture volume.

The OD results was compared with the dry weight of the dehydrated sample. The graph of optical density versus dry weight is shown in Fig. 3. It correlates the generation of bacteria growth, specifically on the reading of the optical density. It could be used to estimate the value of the bacteria dry weight using the same sample with the same medium and environment provided.

The dry weight could give how many grams do we have in per litre of sample by plotting the graph and an acceptable correlation, the dry weight could be determined through the OD. Optical density correlates directly with biomass so that cell concentration could be monitored without having to conduct tedious procedures for measuring the cell dry weight or concentration of cells by hemocytometry or plating for colony forming units (CFU/mL) (Myers et al., 2013). Based on the graph, the relationship between the optical density and the dry weight is expressed in the mathematical equation by obtaining the value of R^2 which was 0.9544.

3.2 Corrosion rates and biocide inhibition tests

The average corrosion rates at the end of the five-day experiment are shown as the column chart in Fig. 4. The corrosion rate without any biocide addition was recorded as 0.586 mm/y. The effectiveness of glutaraldehyde as the corrosion inhibitor for MIC can

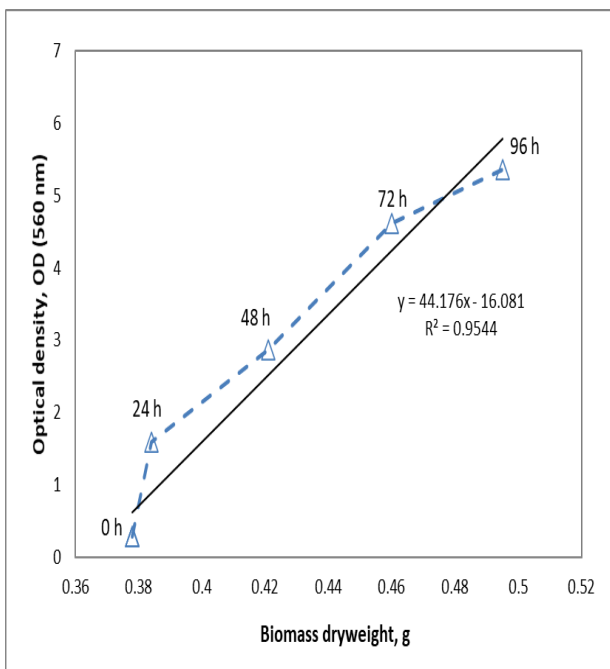


Fig. 3: Correlation between OD and dry weight.

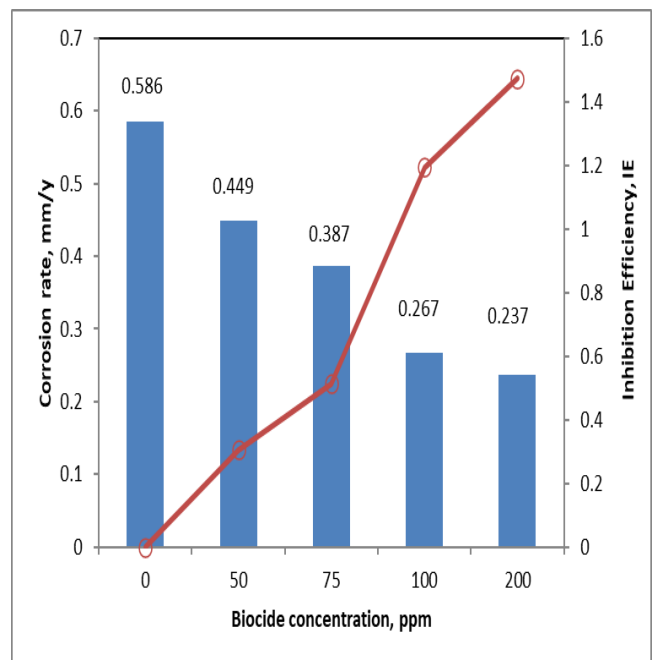


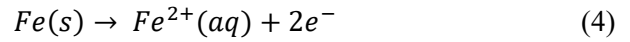
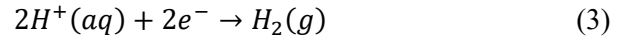
Fig. 4: Coupon corrosion rates and inhibitor efficiency.

be observed by the decrease in the corrosion rates at the various concentrations in ppm. The lowest corrosion rate was recorded at the highest biocide concentration which was 200 ppm.

The decrease in corrosion rates with the increased biocide concentration relates to the efficiency of glutaraldehyde as a corrosion inhibitor. From visual observation, the colour of the solution completely changed from light brown to dark blackish brown, where sulphur oxidizing bacteria produced the same effect with iron bacteria where they convert sulphide into sulphate, producing a dark slime that could cause clog pipelines (Jørgensen, 1982; Hao et al., 1996). The blackening of water may indicate SOB problem. There was a strong "rotten egg" or "sulphur water" odour from the sample which support the theory of hydrogen sulphide production (Liu et al., 2015).

The solution pH dropped from 7 to 3.3 in the uninhibited corrosion test. This may be explained by the oxidation of sulphur that may have occurred through the metabolic activities of microbes, causing the production of sulphuric acid. The acid formed is said to form the sulphate and released the hydrogen ions into the aqueous solution, thus increased the hydrogen ions concentration which account for the pH reduction. The hydrogen ions then caused a cathodic reaction by receiving the electron released by the metal. The accepted electron by the hydrogen ions from the cathodic reaction was triggered further of releasing electrons from the iron, which increased the rate of dissolution of iron into the electrolyte (water).

MIC involving sulphur oxidising bacteria includes a progression of different species according to the precise pH conditions (Berndt, 2011). The mechanism is present in the following Eq. (3), (4), and (5).



The final pH of the tests with the addition of biocide was also measured, as shown in **Fig. 5**. The increase in pH indicates the reduction in the acidity of the solution due to the reduced corrosion and microorganism activities with the addition of biocide.

This result implies that the application of biocide reduced the metabolic activities of sulphur-oxidising bacteria (SOB), which indicate the less oxidation of sulphur to sulphuric acid. Glutaraldehyde act to inhibit the inhibition process of the microbes by delaying the cell growth and reduce the absorbance of nutrients by the cell, rather than killing them, due to the glutaraldehyde ability to form crosslink proteins (Russell, 1994; Wen et al., 2009).

4.0 Conclusions

The water sample from the produced water storage tank of an oil and gas production facility contained a few types of bacteria including major sulphur oxidising bacteria (SOB) as shown by the appearance of colourless colony on the nutrient agar, supported by the microbial growth curve. The presence of live and active microbes in the industrial sample is a possible source of corrosion to steel structures.

The corrosion test confirmed that the SOB cause high corrosion rate of the carbon steel coupon. The addition of glutaraldehyde showed positive results in the inhibition of corrosion by microorganism by eliminating the growth of bacteria.

Further studies can be conducted to analyse the type of corrosion products that had formed on the steel, which can confirm the corrosion mechanism and factors affecting it such as the presence of oxygen, carbon dioxide, and sulphur compounds.

Acknowledgement

The authors would like to thank the Department of Oil and Gas, School of Chemical Engineering, UiTM Shah Alam for the funds and facilities provided to conduct this study.

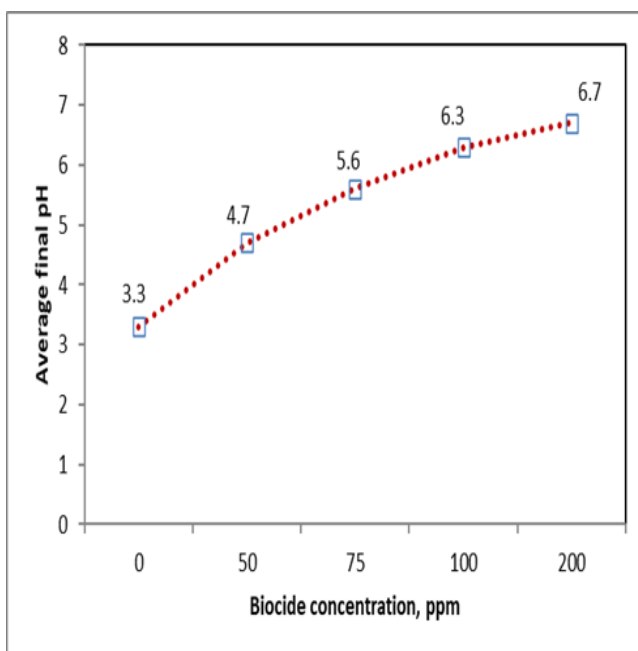


Fig. 5: Final pH measured after Day 5 of corrosion test.

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