



Investigation of the Corrosion of Heating Treatment Medium Carbon Steel in Sulfur Aqueous Solution



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ABSTRACT

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To achieve the necessary mechanical properties and high corrosion resistance, heat treatment is based on a significant alteration in the microstructure of metals and alloys. The metal is heated above a critical temperature during heat treatment processes. Several methods, such as quenching and cooling in various media, followed by tempering and other heat treatment operations, can be used to achieve this. Steel can be made more malleable by annealing, which also increases ductility and improves corrosion resistance. In this study, thirty medium carbon steel specimens were used, which were split into different groups, various heat treatments were applied. First quenching, first tempering, second quenching, and second tempering were among the heat treatment procedures used, and cooling media made of distilled water were used at various temperatures. After that, the corrosion rate in these specimens was looked at, and the results were compared to the corrosion rate in the base specimen. The results show that repeated heat treatment of the metal generally lowers the rate of corrosion in the metal, particularly when distilled water is used for the cooling process after the two tempering stages at a temperature of almost absolute zero. The findings show that the second sample had the lowest corrosion rate of all the samples. When compared to the corrosion rate in the basic sample, the corrosion rate in this sample decreased by about 92.9%.

1. INTRODUCTION

A fundamental academic and industrial topic that has gotten a lot of attention is mild steel corrosion. However, the majority of industrial machinery typically corrodes because acid solutions are so aggressive. Acid pickling of iron and steel, chemical cleaning, ore production, and oil well acidification are a few of the main industrial applications of acid solutions [1]. Almost all facets of human endeavor need the usage of steel. In general, medium carbon steels are used for a variety of purposes, particularly in the onshore and offshore oil and gas, automotive, and food processing industries. As a result, there should be ways to influence and regulate the characteristics of medium carbon steel materials while they are being used with corrosive media with the aim of enhancing their performance. In the history of engineering, corrosion has been the cause of a great number of accidents. The problem that occurs with medium carbon steel is that the oxidation-produced oxide does not securely attach to the metal's surface and consequently peels off readily, leading to pitting. Heat treatment and alloying are the two main factors that give steel its exceptional adaptability [2, 3]. Heat treatment is the process of applying heating and cooling to a solid metal to give it the required condition and qualities [4]. According to studies, organic inhibitors work through adsorption process and as a

result they protect the metal by coating it. Organic compounds that are composed of different atoms and have a high electron density, such as phosphorus, sulfur, nitrogen, or oxygen, or those that have many bonds that are considered adsorption centers, are excellent in reducing the occurrence of corrosion [5-7]. The mechanism of interaction of industrial metals with the environment in which they operate cannot be overlooked, as they operate in different conditions and media [8, 9]. As a result, the metals that are exposed to harsh conditions in different mediums may change their properties, which may lead to the failure of that material very early in its operational life, for which it was designed to function, due to corrosion [10, 11]. Due to the severe harm and capital cost losses caused by corrosion impact, many studies have been looking into carbon steel corrosion resistance under different heat treatment conditions [12-15]. Perhaps as a result of their chemical similarity to emulsified water in oil, synthetic seawater and other brines containing hydrogen sulfide are frequently used as corrosive media [16, 17]. For instance, as is well known in the oxidation of metals, which occurs as a result of the loss of electrons, the reaction rate for activation polarization depends on the excess voltage in order to transfer the charge. In many instances, the controlling factor is the activation polarization during the occurrence of corrosion in medium and strong acids [18, 19]. One of the significant outcomes of this study is the

improvement in corrosion resistance of the models that underwent heat treatments as a result of the models' spherical annealing procedure [20]. The researchers conducted this experimental study on pipes to determine the effect of different heat treatments on the behavior of the corrosion property in them, in the presence of acetic acid and carbon dioxide. The results of this experimental study showed that the annealed samples were less corrosive compared to the other samples that were used in this study [21]. A variety of carefully chosen corrosion media were used to investigate the corrosion resistance of a recently developed steel known as (HSLA). The results showed that samples of steel with high martensite content and high temperatures of hardening exhibited the best corrosion resistance [22]. By applying various heat treatments, the researchers investigated the corrosion resistance of fresh steel. They found that steel with a high martensite microstructure had the best corrosion resistance [23]. The effect of heat treatments on the corrosion resistance of low-carbon steel alloys was studied. A cooling medium was used in a brine solution saturated with carbon dioxide. It was noted from the results obtained that corrosion rates decreased significantly in the models that were subjected to quenching and tempering operations, especially when High temperatures. Both the precipitation of the alloy's carbides and the drop in density are responsible for this [24]. This study demonstrated that martensitic steel is extremely robust following automatization and quenching, and that proper heat treatments are required to produce high hardness in the material [25]. In this study, varied concentrations of sodium chloride and hydrochloric acid, as well as the effects of heat treatment time, were utilized to examine the effects of temperature and heat treatment time on the corrosion resistance of steel. According to the study, hardened steel has roughly (27%) more corrosion resistance than regular carbon steel [26]. Researchers examine the corrosion patterns of carbon steel and stainless steel in this article when there is elemental sulfur present. The annular fin technique was used to examine the alloys' propensity for corrosion at two temperatures with and without elemental sulfur. Results indicated that temperature was one of the most

crucial factors in the corrosion of steel alloys when sulfur was present. Additionally, the experimental results from earlier studies showed that when steel came into direct contact with wet elemental sulfur under conditions free of salt, it was severely corroded [27, 28]. Numerous studies have supported the use of the gravity method to evaluate corrosion to predict primary sulfur corrosion in natural gas pipeline applications [29, 30]. This study resulted in the conclusion that the presence of martensite and ferrite influences corrosion behavior. The amount of martensite increases from 1023 to 1173 K as the austenite temperature rises. The growth of micro-galvanic corrosion cells, which serve as active corrosion centers, in the region between the ferrite and martensite phases is a reflection of this.

In this this study, thirty models of medium carbon steel will be made, divided into groups, and then heat treated. All groups will undergo the first quenching and first tempering, with distilled water at various temperatures serving as the cooling medium. After that, a sample group will be separated, and the remaining sample groups will undergo a second quenching and second tempering. Distilled water will be used as the cooling medium at various temperatures. The rate of corrosion in each model will then be determined by corrosion experiments, and the best corrosion resistance model will be selected by comparing all models to the base model.

2. MATERIALS AND EXPERIMENTAL PROCEDURE

2.1 Materials

In this study, medium carbon steel was used, which is used in many industrial, marine, military, construction and other fields, according to the German Standard (DIN). The chemical composition of this medium carbon steel was investigated using a spectrometer. Table 1 shows the chemical compositions of the metal used and the standard compositions for this type of medium carbon steel.

Table 1. Results of chemical analysis for the metal used

Wt. %	C	Si	Mn	P	S	Mo	Cu	Fe
Standard Value	0.42-0.5	0.15-0.35	0.5-0.8	≤0.035	≤0.035	-----	-----	Rem
Actual Value	0.476	0.331	0.738	0.027	0.031	0.068	0.011	98.318

2.2 Experimental procedure

2.2.1 Samples preparation and heat treatments

The term heat treatment of alloy steels refers to the process of heating and cooling these alloys, provided the metal is in its annealed state. The main purpose of heat treatments is to improve some of the properties of steel, and because heat-treated alloy steels will be used to work in different places and mediums, these hardened alloys are susceptible to corrosion. In this study, thirty-three samples of carbon steel were prepared to study the corrosion resistance behavior of heat-treated samples cooled in distilled water at different temperatures, whereas the shapes and measurements of those samples complied with the requirements of the test apparatus. The thirty-three models were divided into five groups for repeated heat treatments and Table 2 shows the classification of these models.

Figure 1 illustrates the various stages of the models' heat treatments, which were carried out in accordance with the subsequent steps:

1. The models underwent first quenching by being heated to an oven temperature of (870°C) Fahrenheit for 45 minutes.
2. As shown in Table 2, the samples were cooled with distilled water that was at various temperatures.
3. Putting the models in the oven for 25 minutes at (350°C) to do the first tempering of the models.
4. Keep the group samples (B), and carry out the second quenching of the rest of the samples for the groups (C, D, E, F), by heating these samples in the oven at a temperature (850°C) and for a period (45 min.).
5. Performing the second tempering of the models for groups C, D, E, and F by putting them in the oven at (450°C) for 25 minutes.

Table 2. The classification of the groups of all models

Group	Specimen	First Heat Treatment (Quenching + Tempering)	Temperature of Water °C	Second Heat Treatment (Quenching + Tempering)	Temperature of Water °C
A	S		As Received Specimen		
	S-1		0°C		
	S-6		5°C		
B	S-11	Quenching + Tempering	10°C	----	----
	S-16		15°C		
	S-21		20°C		
	S-26		25°C		
	S-2		0°C		
	S-7		5°C		
C	S-12	Quenching + Tempering	10°C	Quenching + Tempering	0°C
	S-17		15°C		
	S-22		20°C		
	S-27		25°C		
	S-3		0°C		
	S-8		5°C		
D	S-13	Quenching + Tempering	10°C	Quenching + Tempering	10°C
	S-18		15°C		
	S-23		20°C		
	S- 28		25°C		
	S-4		0°C		
	S-9		5°C		
E	S-14	Quenching + Tempering	10°C	Quenching + Tempering	20°C
	S-19		15°C		
	S-24		20°C		
	S-29		25°C		
	S-5		0°C		
	S-10		5°C		
F	S-15	Quenching + Tempering	10°C	Quenching + Tempering	30°C
	S-20		15°C		
	S-25		20°C		
	S-30		25°C		

Conducting a revision after each tempering has the goal of removing internal tensions that develop in the hardened metal as a result of the change from the ferrite phase to the martensitic phase. A further advantage of the revision process is the elimination of thermal stresses brought on by the disparity in cooling rates between the metal's surface and core.

internal and external vessels. The electrochemical cell typically has three electrodes, which are as follows:

1. Using carbon steel as the working electrode to calculate the voltage from it in accordance with the reference electrode.
2. The auxiliary electrode is made of platinum and has a surface area of (1 cm²).
3. Calomel that is fully saturated (Hg/Hg₂Cl₂ sat. KCl), serving as a reference electrode.

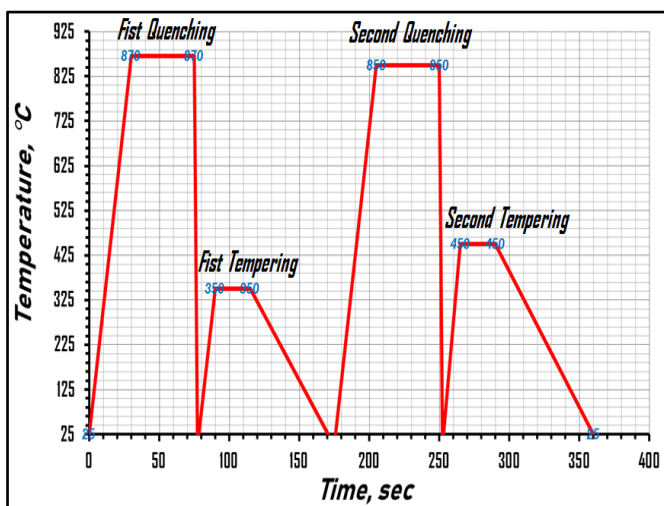


Figure 1. The different stages of the models' heat treatments



Figure 2. The system of corrosion/EIS measurement

2.2.2 Corrosion tests / EIS measurement

As shown in Figure 2, the corrosion measurement/EIS set consists of a computer, thermostat, magnetic stirrer, potentiostat/galvanostat, and EIS (Vertex One, Ivium Technologies, Netherlands). Pyrex cell volume (100 mL) with

To establish steady state open circuit potential (Eocp), the working electrode was submerged in the test solution for 15

minutes. At K, a cooling-heating circulating water bath was used for all tests.

According to the OCP value, the electrochemical measurements for corrosion were carried out in a potential range of (± 250) mV.

For EIS measurement, in impedance 10 mV of amplitude was used, and the range of frequency was between 0.1 Hz and 100 KHz.

Description of corrosion/EIS system

The corrosion/EIS cell. Electrochemical cell made of Pyrex with (100 ml) capacity Consists of two vessels, Internal and external. Figure 3 illustrates the three electrodes and the corrosion/EIS cell. In an electrochemical cell are three electrodes and a thermocouple.



Figure 3. Set up the corrosion cell and three electrodes

The three electrodes can be explained as follows

A reference electrode was used to calculate the potential of the working electrode using the potential of a reference electrode. Reference electrode potential is a well-known and precise value. It consists of two tubes; as shown in Figure 4, the inner tube contains Hg/Hg₂Cl₂ that has been saturated with KCl solution, with the reference electrode positioned 2 mm away from the working electrode.

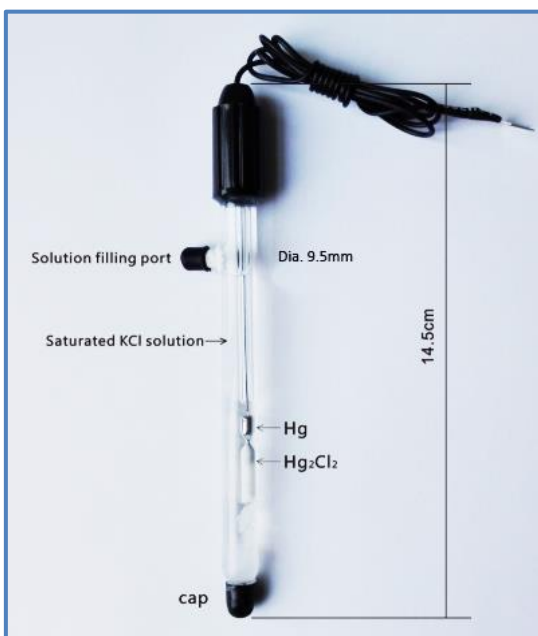


Figure 4. Saturated calomel electrode

The auxiliary electrode was made of high purity platinum metal with area (1 cm²) as explained in Figure 5.

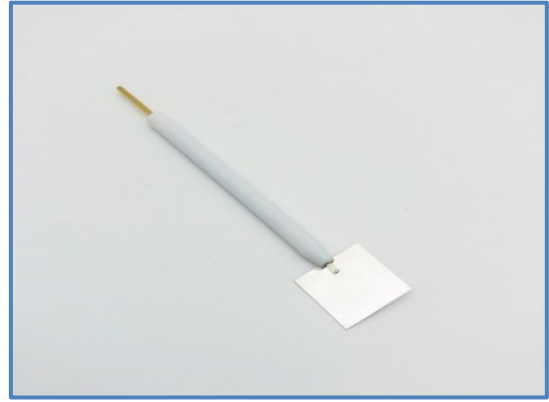


Figure 5. Platinum electrode

The working electrode is the area under investigation, where its potential ought to be assessed. This electrode was created from a metallic wire of 20 cm in length and attached to the mounted specimen in Figure 6.



Figure 6. Specimen holder

3. RESULTS AND DISCUSSION

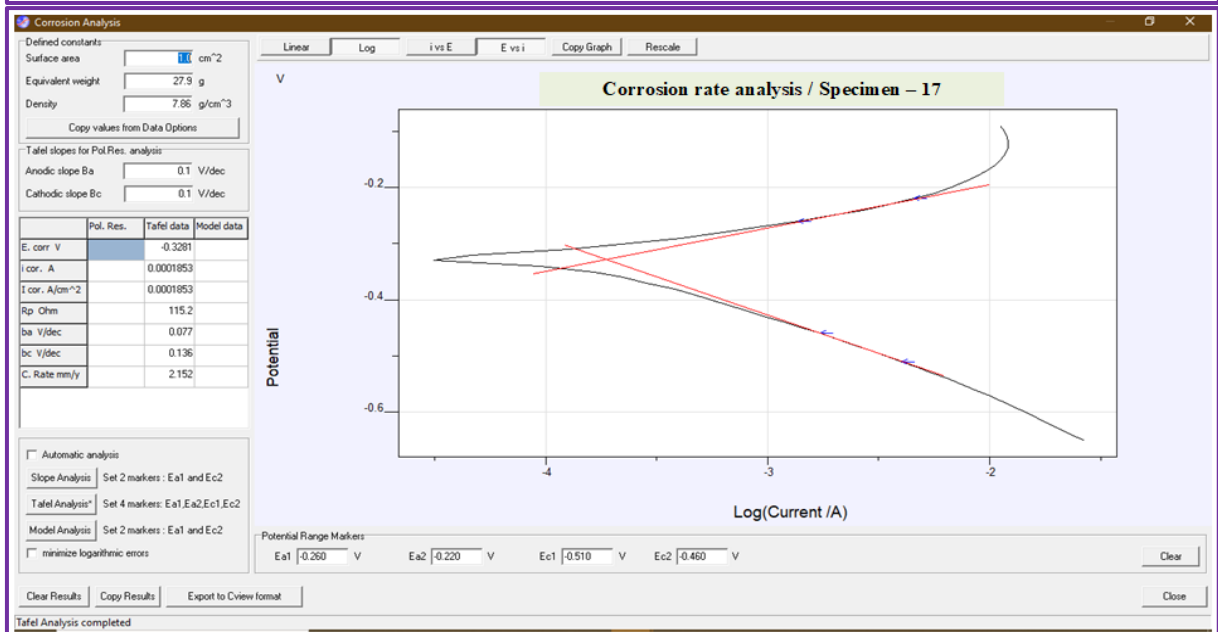
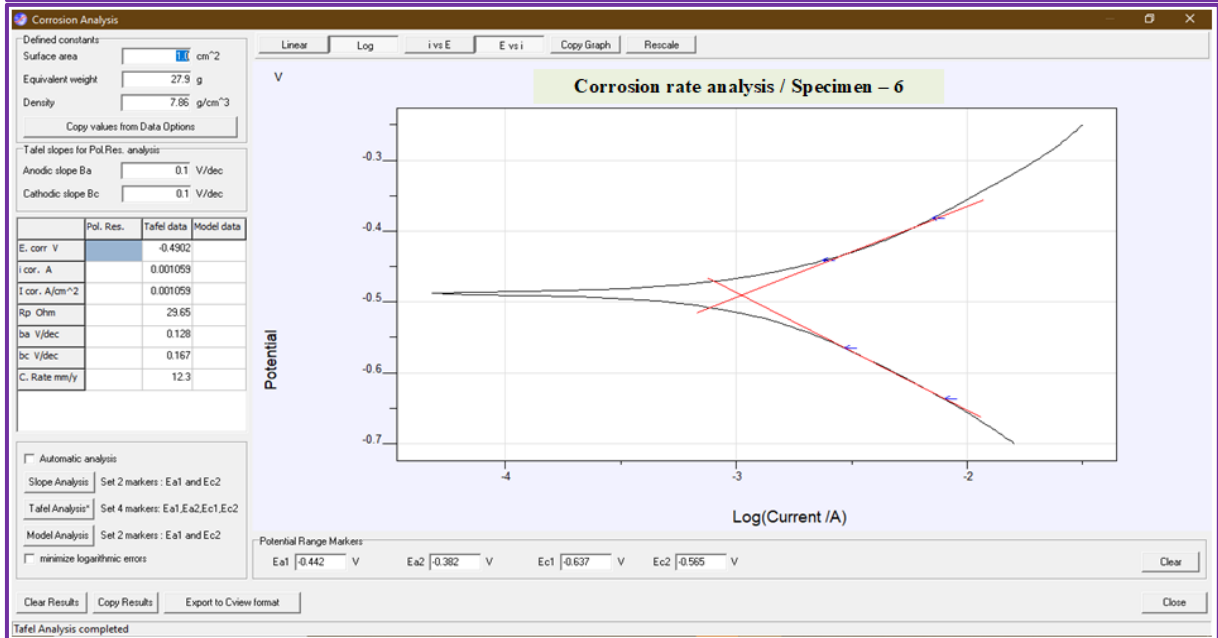
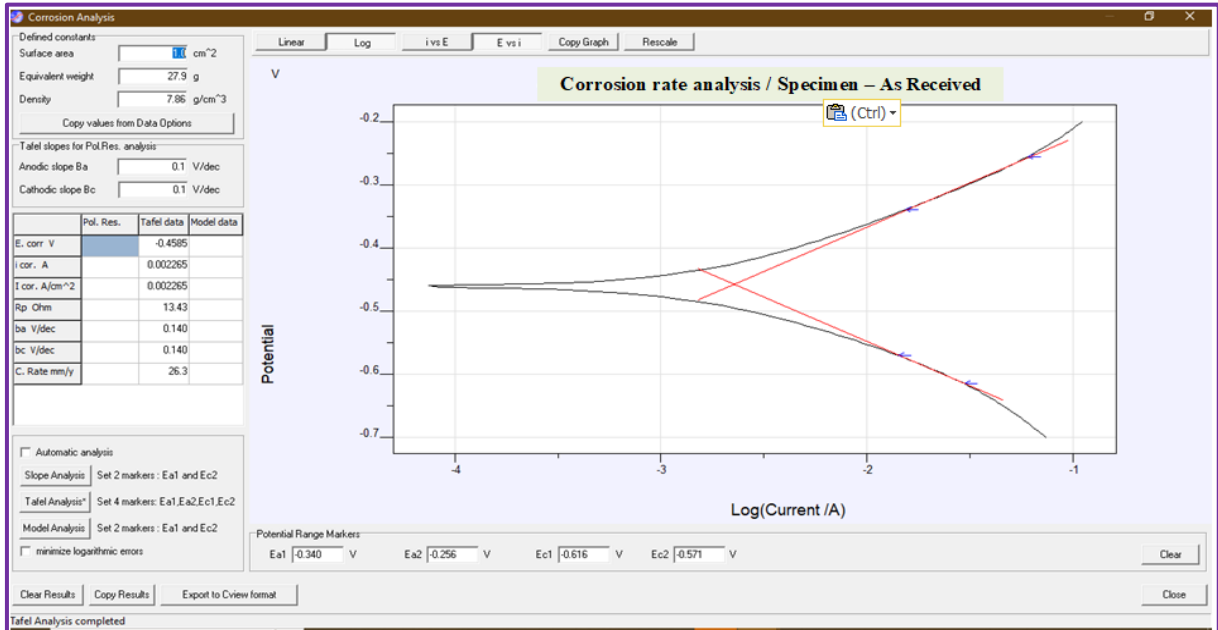
In this study, an electrochemical analyzer was used to read the corrosion current (i_{corr}) and the voltage, which are important to obtain the necessary information, in order to determine the corrosion rate of the steel used in this study. The formula provided by ASTM Standards G59 and G102 [30] and derived from Faraday's Law is used to estimate corrosion rates (R_m) [31-33]:

$$R_m = K_1 \left[\frac{EW \cdot i_{corr}}{\rho} \right] \tag{1}$$

where:

- R_m = Rate of corrosion,
- i_{corr} = Corrosion current,
- K_1 = Constant = 3.27,
- ρ = Density = 0.00786 gm/mm³,
- EW = Equivalent weight = 27.9 gm.

Figure 7 shows how the variables obtained from the experiments conducted on all models were used to calculate the corrosion rate for a portion of the selected models (one model was chosen from each group).



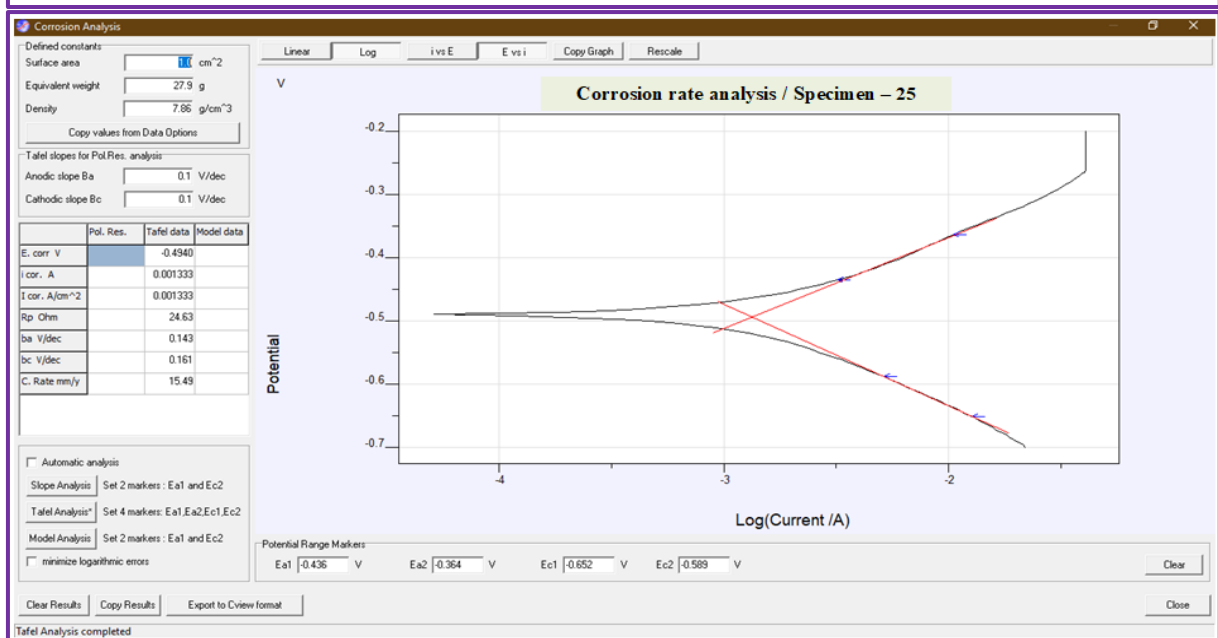
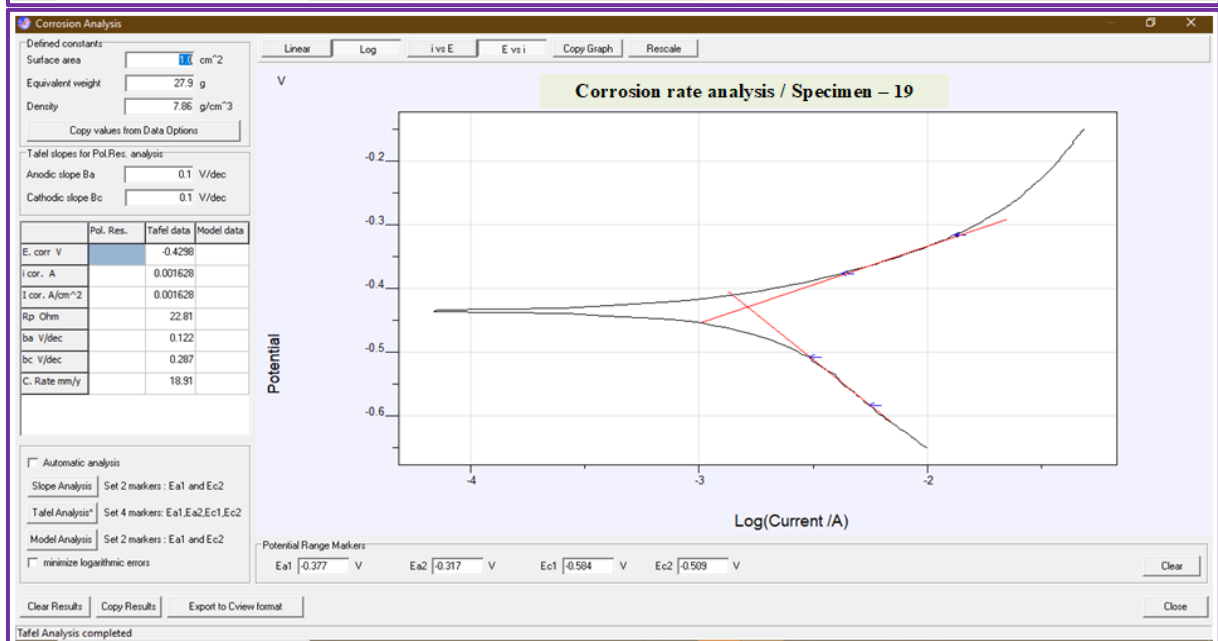
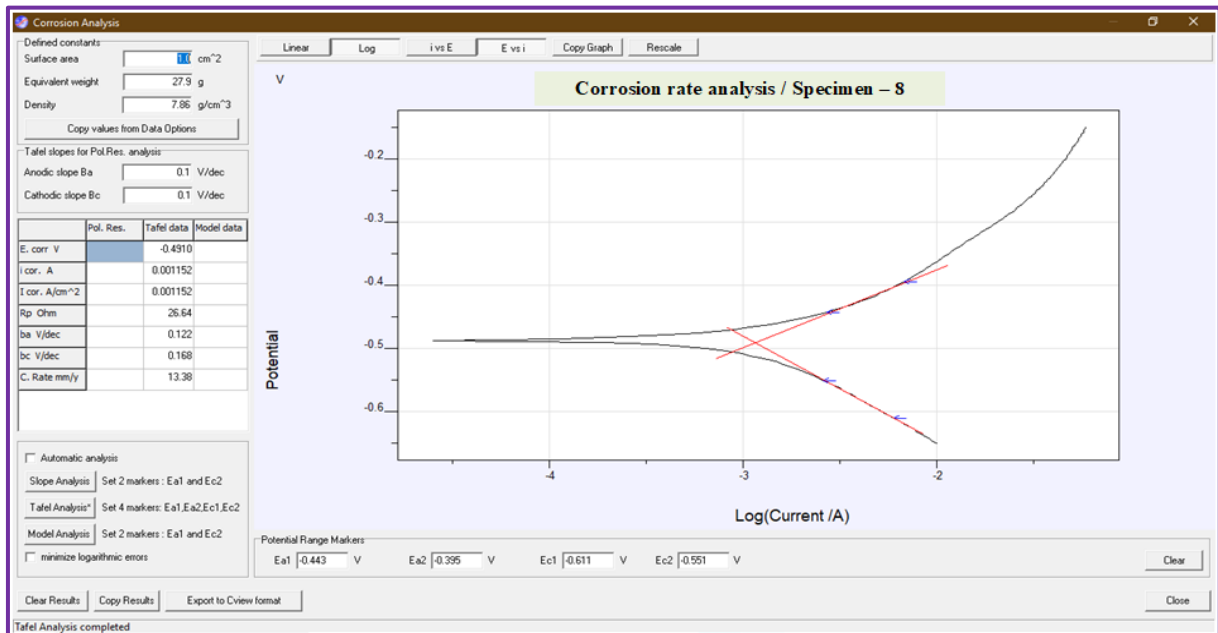


Figure 7. The method of calculating the corrosion rate

The results of the analysis showed that the corrosion rate is influenced by the type of treatment used and the temperature of the distilled water used for cooling. Table 3 shows the values obtained from the corrosion rate, after using Eq. (1),

through the variables obtained from the corrosion tests for models that underwent hardening processes with distilled water and with different degrees of cooling of the water used to cool the models after hardening.

Table 3. The values obtained from the corrosion rate

Group	Sample	S					
A	Corrosion rate (mm/yr)	26.30					
B	Sample	S-1	S-6	S-11	S-16	S-21	S-26
B	Corrosion rate (mm/yr)	0.1102	0.123	0.1289	0.1334	0.1381	0.1433
C	Sample	S-2	S-7	S-12	S-17	S-22	S-27
C	Corrosion rate (mm/yr)	0.0188	0.0193	0.0202	0.0215	0.0253	0.0287
D	Sample	S-3	S-8	S-13	S-18	S-23	S-28
D	Corrosion rate (mm/yr)	0.1283	0.1338	0.1364	0.1392	0.1465	0.1533
E	Sample	S-4	S-9	S-14	S-19	S-24	S-27
E	Corrosion rate (mm/yr)	0.1777	0.1812	0.1864	0.1891	0.1943	0.1999
F	Sample	S-5	S-10	S-15	S-20	S-25	S-30
F	Corrosion rate (mm/yr)	0.1433	0.1461	0.1489	0.1521	0.1549	0.1593

A comparison between the base sample and the samples from group-B is shown in Figure 8. The figure makes it clear that the first sample and its value (0.1102 mm/yr) had the lowest rate of corrosion in this group, whereas the twenty-sixth sample and its value (0.1433 mm/yr) had the highest rate of corrosion.

The base sample and the samples from group C are compared in Figure 9. The second sample and its value (0.0188 mm/yr) had the lowest rate of wear in this group, according to the figure, while the twenty-seventh sample and its value (2.87 mm/yr) had the highest rate of corrosion.

The base sample and the samples from group-D are contrasted in Figure 10. The figure shows that the third sample and its value (0.1283 mm/yr) had the group's lowest rate of corrosion, whereas the twenty-eighth sample and its value (0.1493 mm/yr) had the group's highest rate of corrosion.

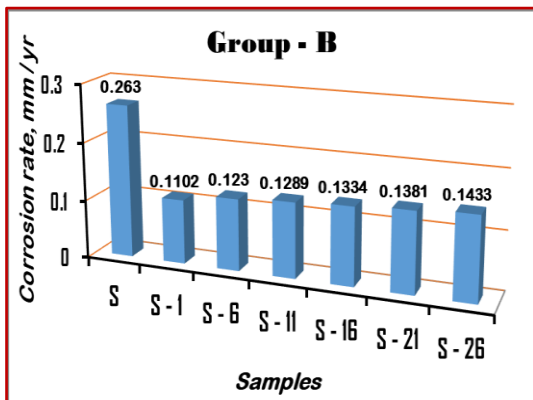


Figure 8. Comparison of the rate of corrosion between the base sample and the sample group-B

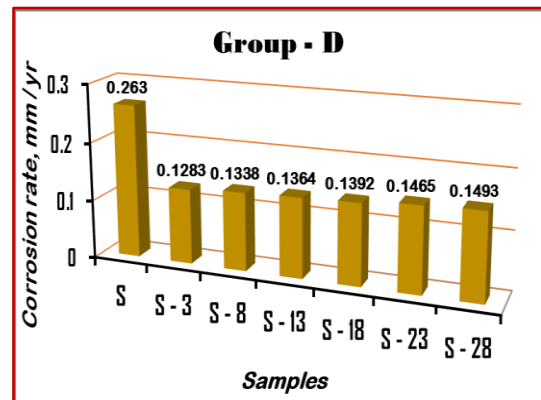


Figure 10. Comparison of the rate of corrosion between the base sample and the sample group-D

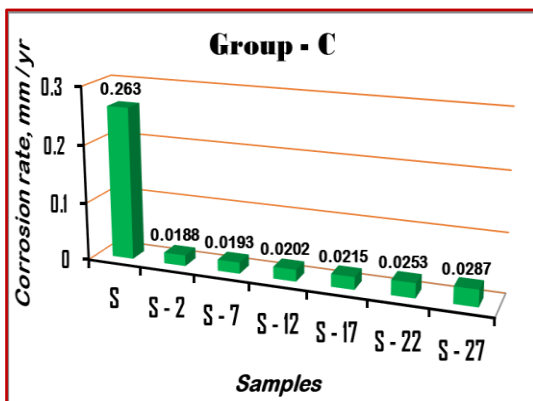


Figure 9. Comparison of the rate of corrosion between the base sample and the sample group-C

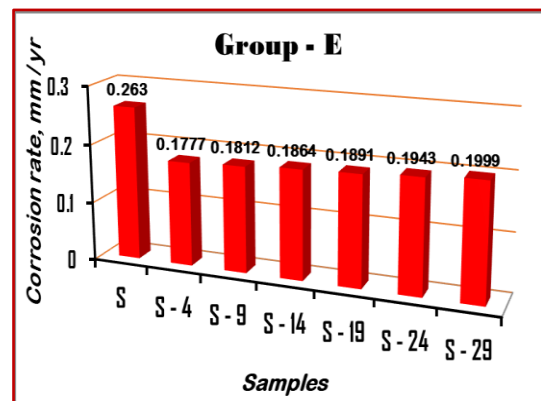


Figure 11. Comparison of the rate of corrosion between the base sample and the sample group-E

Figure 12, compares the base sample with the samples from group-F. The fifth sample and its value (0.1433 mm/yr) in this group-F, had the lowest rate of corrosion, whereas the thirty sample and its value (0.1593 mm/yr) had the highest rate of corrosion.

Figure 13 shows a comparison between samples from each group and the basic sample. The figure shows that the highest corrosion values were in the fourth group and decreased slightly from the original model, while the lowest corrosion values were in the second group and very low.

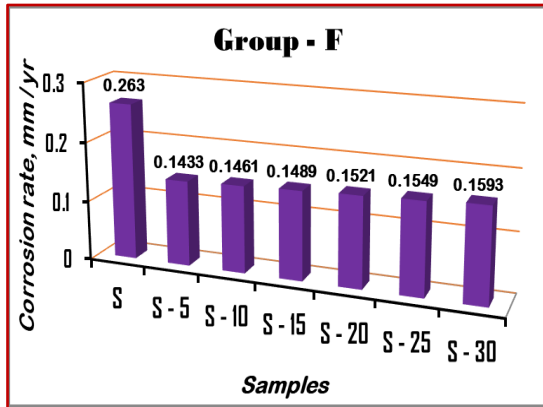


Figure 12. Comparison of the rate of corrosion between the base sample and the sample group-F

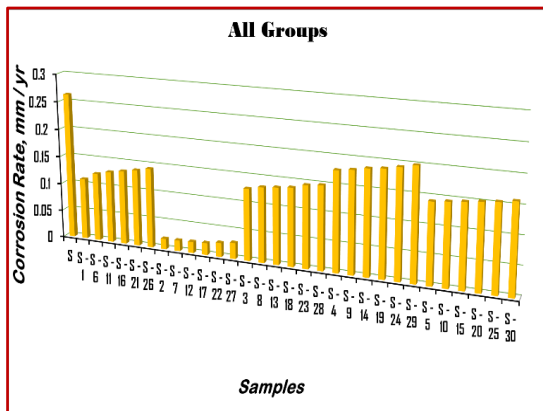


Figure 13. Comparison of the rate of corrosion between the base sample and the all samples

4. CONCLUSIONS

To determine the rate of corrosion of medium carbon steel, repeated heat treatments and quenching in distilled water at various temperatures were applied, followed by electrochemical tests. The study's findings led to the following conclusions:

1. According to the results, repeated heat treatment of the metal generally lowers the rate of corrosion in the study's metal, particularly when using distilled water at almost absolute zero degrees Celsius for the cooling process after the two stages of tempering. The second sample, which was hardened and reviewed twice, had the lowest rate of corrosion; it was cooled by tempering in distilled water at a temperature close to absolute zero degrees Celsius; the corrosion rate in this sample was approximately (92.9%) lower than that of the base sample.

2. The results indicate that group-C, on which repeated heat treatment was carried out, is the best group in which the rate of corrosion decreased significantly. In the first stage, the samples were quenched and cooled in distilled water at a variety of temperatures (0°C, 5°C, 10°C, 15°C, 20°C, 25°C), and in the second stage, they were second quenched and cooled in distilled water at a temperature of almost absolute zero degrees Celsius, with revisions tempering after each quenching process for the two stages.
3. The medium carbon steel used in this study, which has the largest particle size, had the highest corrosion rate, while the samples that were subjected to repeated heat treatments and in distilled water cooling media had lower corrosion rates, suggesting that the rates of corrosion of the sample depend on the size of the metal grains. It was the most resistant to corrosion and, depending on the temperature, had a smaller grain size.
4. It has been determined that the quenching and tempering processes increase corrosion resistance because they produce more homogeneous phases and increase the union of iron with carbon, which reduces the union of iron with oxygen, which causes corrosion.
5. The corrosion resistance of steels in various applications will be further improved, so it can be predicted that steels with a higher percentage of bainite/martensitic will have more advantages in manufacturing applications. By conducting heat treatment processes in different quenching processes, additional alloying elements can be avoided, leading to a significant cost reduction.

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NOMENCLATURE

E Corrosion, *V*
I Corrosion, μA
I Corrosion per surface area, A/cm^2

p Polarization Resistance, Ω
R β a Anodic Tafel constant, *V/decade*
 β c Cathodic Tafel constant, *V/decade*
CR Corrosion rate, *mm/year*
IE % Inhibition efficiency