GLOBAL JOURNAL OF ENGINEERING SCIENCE AND RESEARCHES

DEVELOPMENT OF ANALYTIC MODELS FOR PREDICTION OF CORROSION RATE OF 13% CHROMIUM STEEL EXPOSED TO DIFFERENT ENVIRONMENTAL CONDITIONS

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ABSTRACT

This paper, by experimentally investigating the influence of different corrosion product layers on the corrosion resistance of 13% chromium steel in HCl solution, describes the level of the corrosion rates induced by deposited iron sulfide and elemental sulfur layers on the steel surface. In order to facilitate the experiment numbers, three analytic prediction methods, which are the optimal solution, curve fitting and artificial neural network, were applied to predict the corrosion rates of 13% chromium steel. Results showed that the fitness between measured and predicted corrosion rates by curve fitting indicates a good correlation between experiments and developed model, however, the minimum deviation from the measured data was obtained with artificial neural network model which is insignificant compared to the deviation of the two other models

Keywords: *13% chromium steel, Neural Network, Iron sulfide, Elemental sulfur, Corrosion modelling.*

I. INTRODUCTION

Corrosion, either sweet corrosion generated by CO2 or sour corrosion generated by H2S [1], is realized as a main issue that the oil and gas industry are faced with. During the production stage, pipelines and other equipment will be corroded due to the reactions between CO2, H2S and Fe [2]. Pipelines designed to withstand 50 years of operation, however, under a "worst case" general corrosion rate may fail after a few months of operation due to localized corrosion. Loss of containment from a pipeline failure is a costly event as it would cause an emergency shutdown in the production of oil and gas, an emergency repair of the pipeline, and probably an environmental clean-up at the leak site [3]. In an effort to minimize pipeline failures and loss of containment, companies around the world in the oil and gas industry sponsor research programs focused on better prediction methods and better mitigation methods of localized corrosion. Normally, prediction of these two kind of corrosion, in order to estimate the equipment lifetime, are not a simple step because the general understanding of the corrosion mechanism, especially in a certain environmental conditions, is usually below the required level to predict the accurate corrosion rate[4]. Depending on the environmental conditions, such as temperature and pH, the rate of these reactions will vary which determine the rate of corrosion process. In the literature, a number of research studies the prediction of the corrosion rate in sweet oilfields that can be classified in three general groups: mechanistic, semi-empirical and empirical models [2], [4], [5]. Among these models, by utilizing a large number of experimental CO2 corrosion data, it was shown that the empirical model, especially one based on the Artificial Neural Network (ANN), has the highest accuracy of corrosion rate estimation, while the lowest accuracy belongs to the mechanistic model [4].

In the case of sour corrosion, it is well known that the corrosion products such as iron sulfide, formed on the steel surface immediately after a small concentration of H2S is introduced into the system which determine the corrosion pattern with regard to the environmental conditions [6], [7]. Generally it was found that formation of corrosion products on the steel surface significantly affect the estimation of upcoming corrosion process and consequently the prediction of finial corrosion rate [8].To estimate the corrosion induced by sour corrosion products, despite many studies and proposed mechanistic models that have appeared in the literature, there is still lack of a reliable and accurate predictive model which can predict the corrosion rate in the presence of H2S corrosion products on the steel surface [9]–[11], [6]. The most recent research in this area showed that the developed corrosion models by ANN have indicated the highest accuracy among all other predicted models [1], [12], [13].

The corrosion of 13 % chromium steel, one of the most common martensitic steel in oil and gas applications, in either sweet or sour condition, have been investigated from various aspects [14]–[16]. According to those investigations, one of the most effective factors in the corrosion process in oil and gas pipelines and especially in the sour ones, is the presence of deposited elemental sulfur produced by the H2S corrosion process [17]. It is known from prior research that the presence of dry elemental sulfur in contact with carbon steel is not considered as a corrosion threat to steel; however, by adding water to the system, the corrosion process may be dramatically accelerated [18]. A literature review has shown that the nature of H2S corrosion product layers controls the kinetics of this corrosion process from both phase type and morphology perspectives [6].

As aforementioned issues above, in this study we are motivated to propose several analytic approaches to predict the corrosion rate of 13% chromium steel in a simulated sour environment by considering the presence of various sour corrosion product layers on the surface of steel, which can be named as optimal solution, curve fitting and ANN. In this way, the optimal solution assumes the measured corrosion rate as an output of an initial function, therefore, solver strives to find appropriate coefficients for the initial function to minimize its error, and then this initial function accuracy is improved and can be used for estimation purposes [19]. In the next proposed model, which is based on curve fitting, the measured corrosion rates in 2-D space plotted and curve fitting utilized to capture a polynomial. Thus, the obtained polynomial in proportion to input can estimate the corrosion rates. The finial applied approach in this study is ANN. An ANN is an information processing pattern that is inspired by the brain's processing information system. [20]. The utilized pattern in ANN is composed of various layers which can be classified in the following layers: input, hidden and output layers. Hidden layers are always formed from a number of hidden neurons whose output is connected to the inputs of other neuron and is therefore not visible as a network output. Typically ANN comprise some form of learning rules that mutate the weights of the connections between the layers. In the following section, the capability of each model in estimation of the corrosion rates based on environmental conditions will be discussed.

II. METHOD & MATERIAL

A. Material and sample preparation

According to industrial partner's request, the corrosion samples were made from conventional 13% Cr steel. Table 1 indicates composition of grade 420 chromium stainless steel. The working electrode was machined from the parent material into cylinders having dimensions of approximately 9 mm length and 9 mm diameter. It should be noted, prior to perform the experiments all specimens were polished with Coated Abrasive Manufacturers Institute (CAMI) grit designations 320, 600, 1000 corresponding to average particle diameters 36.0, 16.0, and 10.3 microns and finally 6 micron grit silicon carbide paper, and then cleansed with deionized water until a homogenous surface was observed. Thereafter, to avoid oxidation, the specimens were quickly dried by using cold air.

Table 1. The chemical composition of conventional 13% Cr stainless steel grade 420.

C	Cr	Mn	Si	P	S	V	Fe
0.027	12	0.22	0.3	0.014	0.0035	0.041	Bal.

B. Corrosion measurements

To investigate the effect of different corrosion product layers on electrochemical behavior of conventional 13% Cr steel, three series of experiments with consideration of different environmental conditions were conducted. In the first series of experiments the corrosion behavior of each sample was analyzed while its top surface was exposed into the electrolyte solution, 0.01 M hydrochloric acid, without any initial cover on it (i.e. no initial corrosion product layer), with different environmental conditions. Table 2 listed the experimental conditions in first series of the experiments.

Table 2. The experimental conditions in first series of the experiments

Sample No	1	2	3	4	5	6	7	8	9	10	11	12
T (◦C)	25	25	25	75	75	75	25	25	25	75	75	75
NaCl (g/L)	0	10	20	0	10	20	0	10	20	0	10	20
pH	2	2	2	2	2	2	4	4	4	4	4	4

In the second series of the experiments, behavior of corrosion was analyzed while the working electrode were initially covered by a thin iron sulfide corrosion layer (i.e. FeS layer) synthesized by an acidic chemical bath [21]. The mechanism of FeS formation in this acidic bath is composed of first the slow release of iron and sulfur ions within solution and then the deposition of these ions on the alloy surface. The iron and sulfur ions are provided from iron trichloride and thioacetamide, respectively. The formation of FeS film from this acidic bath is dependent on whether the deposition rate of the ionic product of iron and sulfur is higher than solubility of FeS or not. Adding urea to the solution adjusted the balance between hydrolysis and deposition. The proposed reactions for this mechanism is described as follows [22]:

$FeCl_2 \rightarrow Fe^{2+} + 2\,Cl^{-}$

$CH_3CSNH_2 + H_2O \leftrightarrow S^{2-} + CH_3CONH_2 + 2H^{+}$

$CO\,(NH_2)_2 + H_2O \leftrightarrow 2NH_3 + CO_2$

$NH_3 + H_2O \leftrightarrow NH_4^{+} + OH^{-}$

$Fe^{2+} + S^{2-} \leftrightarrow FeS$

Finally, the overall reaction can be written as:

$Fe^{2+} + CH_3CSNH_2 + CO\,(NH_2)_2 + 2H_2O \rightarrow FeS + CH_3CONH_2 + 2NH_4 + CO_2$

This second series enabled the estimation of effect of deposited FeS layer on electrochemical and corrosion behavior of 13% Cr stainless steel in presence of various chloride concentration. Table 3 presents the experimental conditions in the second series of the experiments.

At the last series of experiments, the electrochemical behavior was analyzed while the electrode was initially covered by sublimed elemental sulfur 99.9999% (ACROS) [23]. This third series enabled the estimation of effect of deposited elemental sulfur on electrochemical and corrosion behavior of 13% Cr stainless steel in presence of various chloride concentration. Table 4 indicates the experimental conditions in third series of the experiments.

Table 3. The experimental conditions in second series of the experiments

Sample No	1	2	3	4	5	6	7	8	9	10	11	12
T (◦C)	25	25	25	75	75	75	25	25	25	75	75	75
NaCl (g/L)	0	10	20	0	10	20	0	10	20	0	10	20
pH	2	2	2	2	2	2	4	4	4	4	4	4

Table 4. The experimental conditions in third series of the experiments

Sample No	1	2	3	4	5	6	7	8	9	10	11	12
T (◦C)	25	25	25	75	75	75	25	25	25	75	75	75
NaCl (g/L)	0	10	20	0	10	20	0	10	20	0	10	20
pH	2	2	2	2	2	2	4	4	4	4	4	4

During this study, corrosion experiments were conducted in a multi-port glass cell with a three electrodes setup at atmospheric pressure based on the ASTM G5-94 standard for potentiodynamic anodic polarization measurements [21]. Linear polarization resistance (LPR) technique was used to record the general corrosion rates after each experiments. The applied sweep rate for this measurements was 0.5mV/s. An Ivium Compactstat Potentiostat monitoring system was used to perform electrochemical corrosion measurements and record the final corrosion rates. The immersion time was 24 hours for each experiment, however, prior to start of each test the sample was immersed in the solution for 55 minutes accordance with ASTM G5-82 [21]. The pH was adjusted 2 and 4 by adding deoxygenated hydrochloric acid. A graphite rod was used as the counter electrode (CE) and saturated silver/silver chloride (Ag/AgCl) was used as the reference electrode (RE) and as mentioned in material preparation section, the conventional 13% Cr steel samples was used as working electrodes (WE). Figure 1. illustrates our utilized experimental set up for corrosion measurements.

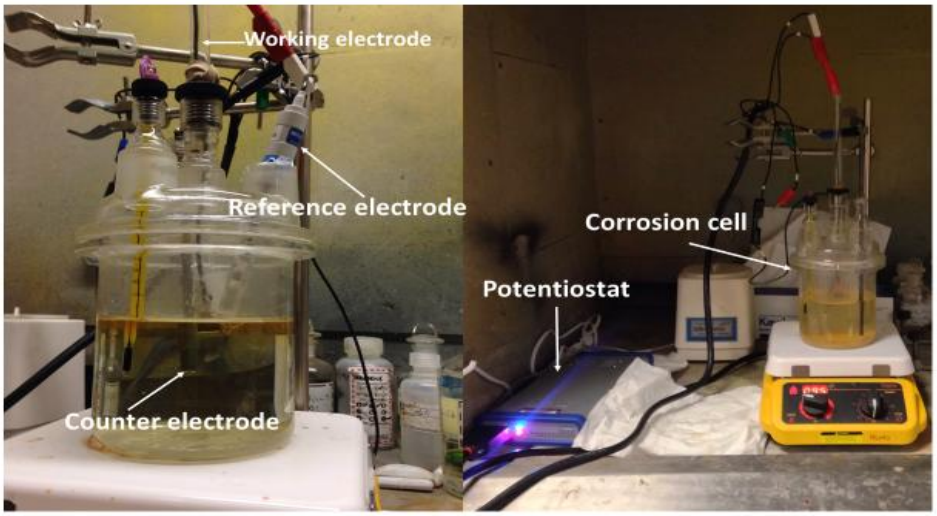

Figure 1. The utilized experimental set up for corrosion rate measurements

C. Optimal solution

The optimization was carried out using Microsoft Excel Solver software. Solver is part of a suite of commands with what-if analysis tools. Solver works with a group of cells that are related, either directly or indirectly, to the objective function equation in the target cell. Solver adjusts the values in the changing cells, called the adjustable cells to produce the result. Constraints are applied to restrict the range of values of the variables used in the objective function [19]. Microsoft Excel Solver tool uses the Generalized Reduced Gradient (GRG) non-linear optimization code to develop the optimal function [24].

D. Curve fitting

The polynomial curve fitting is a common task for data analysts in many fields of material science [25], [26]. However widespread application is not common largely because the use of statistics requires specialist knowledge, and no reference standards exist. The standard method to fit a curve to data is to use the least squares method [27]. In this study, due to nonlinearity of measured corrosion rates, the coefficients of a polynomial function were founded out by curve fitting method. In this regard, the measured corrosion rates were plotted and obtained data from the polynomial function were fitted to the measured results. The typical form of the utilized polynomial function can be identified as:

$$y=m_0 + m_1 * x + m_2 * x_2 + m_3 * x_3 +...+ m_9 * x_9.$$

Where m is the coefficients of a polynomial function and x is the independent variable.

E. Neural networks modelling

The most important stage in the creation of a network which enables the transfer of the input data into the output data, is the learning stage [28]. At this stage the networks' parameters such as the network type, training algorithm and the number of neurons in the hidden layer of the network are modified to fit the experimental data. The number

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of input variables from experimental step will determine the number of input layer or in another words the number of input neurons and the number of output layers also come from the number of output variables in experimental step as well [29]. Designation of number of hidden layers between input and output layers is usually one of the most challenging part prior to create the networks because the increased number of hidden layers would not necessarily increase the networks efficiency and even may unfortunately decrease the speed of computing and make the networks much more complex. Therefore, the final efficiency of the networks would be directly affected by the interaction between neuron transfer functions and typical training patterns [4]. Further information regarding the ANNcan be found in [20], [30]. Figure 2. outlines our utilized configuration to predict the single output, corrosion rate, based on the four input layers: corrosion product layer, pH, temperature and salt concentration, through the five hidden layers.

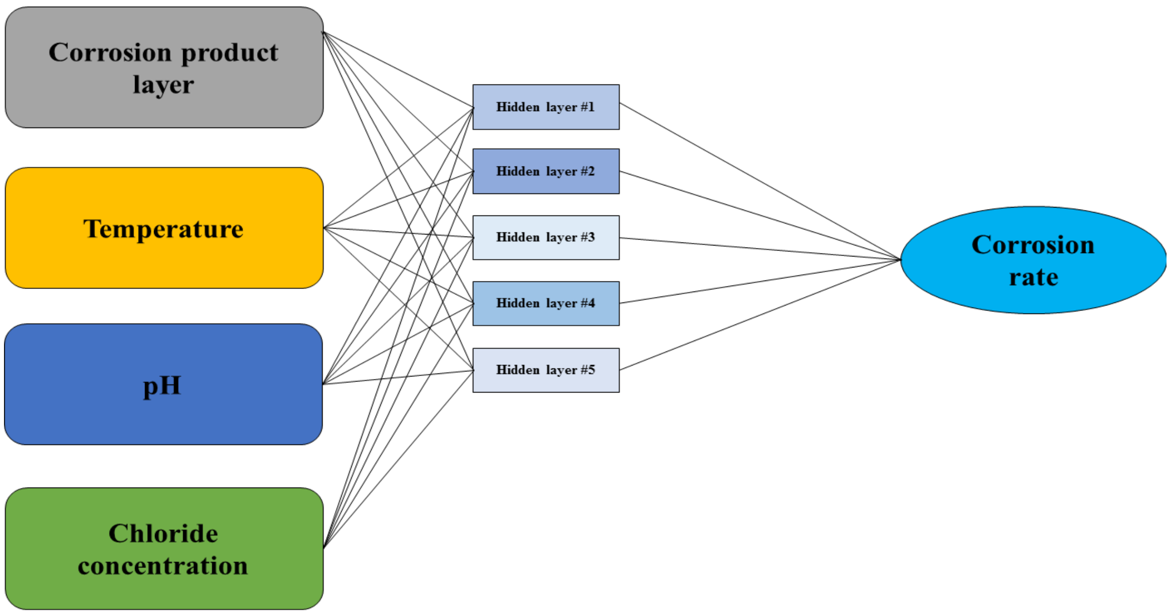

Figure 2. The architecture of ANNused for predicting corrosion rate

The designed network was trained by Levenberg-Marquardt algorithm, which is highly fast in computation, however, to reach the maximum performance it does require more memory in compared to the other available training algorithms [31] Table 5 presents the specification of utilized ANNparameters for corrosion rate prediction purposes.

Table 5. The specification of utilized ANNparameters

Network Parameters	Specification
Hidden layer size	a) 95, b) 85, c) 80, d) 40, e) 30
Network type	Feed- Forward
Transfer function used at network layers	Tangential- sigmoidal
Performance function	Least mean of squared errors
Training algorithm	Levenberg-Marquardt

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III. RESULT & DISCUSSION

a) Corrosion measurements

The general Corrosion Rates (CR) from Linear Polarization Resistance (LPR) measurements are given in Table 6. According to this table, for the first group of samples, with absence of initial corrosion layer on top, it is clear that increase of temperature and chloride concentration increased amount of the corrosion rates, however, with increasing pH from 2 to 4 the corrosion rate slightly decreased which, might be related to the kinetics of precipitation and facilitate corrosion product layers formation, therefore, decreasing the corrosion rates expected [2]. For the second group of samples that were covered with a thin FeS layer, the corrosion rates are generally higher than the first group of samples. It is worth mentioning that similar to the first group of samples (i.e. with no initial corrosion layer) increase of temperature and chloride concentration increased the corrosion rates while increase of pH decreased the corrosion rates. In addition, it can be highlighted that the highest corrosion rate, 4.46 mm/ year, was observed in the presence of elemental sulfur at 75◦C after addition of 20 g/L NaCl to the solution at pH 4.

Table 6. The measured Corrosion Rates (CR) under different environmental conditions.

Series #	T (◦C)	NaCl (g/L)	pH	CR (mm/y)	Series #	T (◦C)	NaCl (g/L)	pH	CR (mm/y)	Series #	T (◦C)	NaCl (g/L)	pH	CR (mm/y)
1	25	0	2	0.057	2	25	0	2	0.472	3	25	0	2	0.301
1	25	10	2	0.169	2	25	10	2	0.705	3	25	10	2	0.246
1	25	20	2	0.744	2	25	20	2	0.797	3	25	20	2	0.524
1	75	0	2	0.263	2	75	0	2	0.989	3	75	0	2	0.908
1	75	10	2	0.709	2	75	10	2	0.901	3	75	10	2	2.713
1	75	20	2	2.078	2	75	20	2	2.879	3	75	20	2	4.987
1	25	0	4	0.009	2	25	0	4	0.327	3	25	0	4	0.077
1	25	10	4	0.028	2	25	10	4	0.403	3	25	10	4	0.019
1	25	20	4	0.476	2	25	20	4	0.576	3	25	20	4	0.113
1	75	0	4	0.056	2	75	0	4	0.849	3	75	0	4	0.554
1	75	10	4	0.498	2	75	10	4	0.756	3	75	10	4	1.108
1	75	20	4	1.828	2	75	20	4	2.406	3	75	20	4	4.46

b) Optimal solution

From Table 6, one can see that the final corrosion rate is sensitive to all experimental parameters, i.e. the corrosion product layer, temperature, pH and chloride concentration. The complex and obscure mechanism of each parameter affects the results in microscopic and macroscopic levels, makes algebraic expressions incapable of predicting the rate of corrosion in this study. Thus, the corrosion rate is assumed to be a transcendental function of all the experimental parameters that account for the sensitivity of the corrosion rate to the all input parameters and their interactions. The initial utilized function is given by:

$$C_R = L\,(F + a)^m (T + b)^n (P + c)^q\,(C + d)^r$$

Where F donates the film parameter, T is temperature parameter, P is the pH parameter and C is the chloride concentration parameter. L, a, m, b, n, c, q, d and r are the user defined coefficients, whose values obtained from the optimization procedure.

The goal of optimization procedure is to minimize the sum of squares of residuals. By choosing the initial value of 1 for L, m, n, q and r and 0 for a, b, c and d the model prediction for corrosion rate at each experiment is calculated and the difference between the model result and the experimental result at each experiment is recorded. The sum of squares of residuals is:

$$SS_{res} = \sum_i (y_i - f_i)^2$$

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Where y_i is the experimental corrosion rate and f_i is the predicted corrosion rate. The solver toolbox then changes the values of L, a, m, b, n, c, q, d and r to minimise the magnitude of SS_{res}. This procedure is carried out repeatedly to find the optimized values for all the coefficients simultaneously.

I. Curve fitting

In order to utilize curve fitting method for prediction purposes, we need to plot inputs and outputs in two dimension. Since the number of inputs (i.e. corrosion product layer, temperature, pH and chloride concentration) is not identical to output (i.e. Corrosion Rate), therefore, the initial input function in terms of the 4 input parameters defined. This initial function, which is labeled as X_n, is expressed by:

$X_n (\alpha, \beta, \gamma, \lambda) = \alpha + \beta/10 + \gamma^2 + (\gamma\lambda + 1)$

where index of n is the number of experiment, α is normalized corrosion product layer parameter, which identified with three discrete values of 1, 10 and 20 that stand for the first, second and third series of experiments, respectively. And β is the normalized temperature parameter in Celsius, which varied between 25 to 75 ◦C, γ is the normalized pH parameter, which is varied between 2 to 4 and λ is normalized chloride concentration parameters, which is varied between 0 to 20 g/L. Thus, thanks to defined polynomial above, input value proportion to corrosion rate calculated and the following equation, which is defined by curve fitting method, utilized for corrosion rates prediction:

$CR= -0.006* X_n^2 + 0.136* X_n - 0.025$

Different degrees of polynomial from 2 to 8 were tested to find the best fitted model. With regard to the values of R-square which indicates thecloseness of the data to the fitted regression line, the best fitness was obtained with polynomial of degree two. In this case the measured R-squared is 0.78 which is relatively high and so it can be assumed that the fitness of the model with the experiments is relatively reasonable. Figure 3. shows the curve of p (x) in MATLAB®.

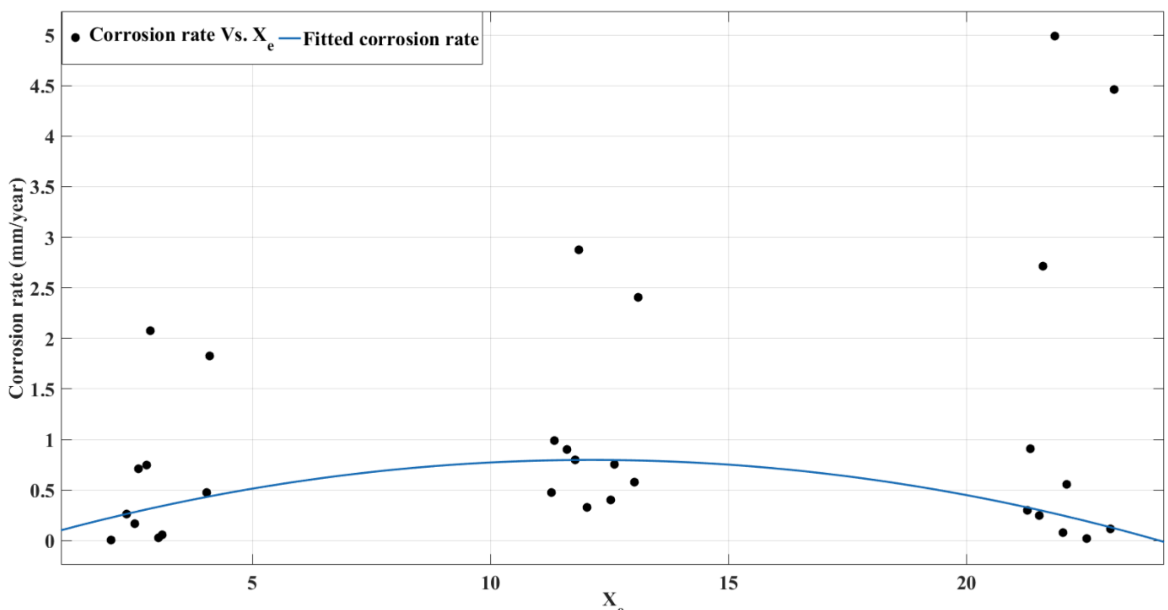

Figure 3. The developed model by curve fitting in MATLAB®

As displayed in Figure 3. the model was not able to fit with measured data in some areas, for instance, where the corrosion rates are higher than 1 mm/y. Thus, the model cannot be considered as an accurate model and still has weakness in prediction of the data.

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c) Artificial Neural Network

After determining the optimum ANNstructure, and prior to the process of training, the whole dataset of 36 input-output pairs was randomly divided into the 30 training data set, white cell in Table 6 and the six validation data set, gray cells in Table. 6. Figure 4 and 5. demonstrate the measured values of corrosion rates and the values predicted by technique for the training and validation data set, respectively. It can be seen that in both two data sets a good prediction was achieved.

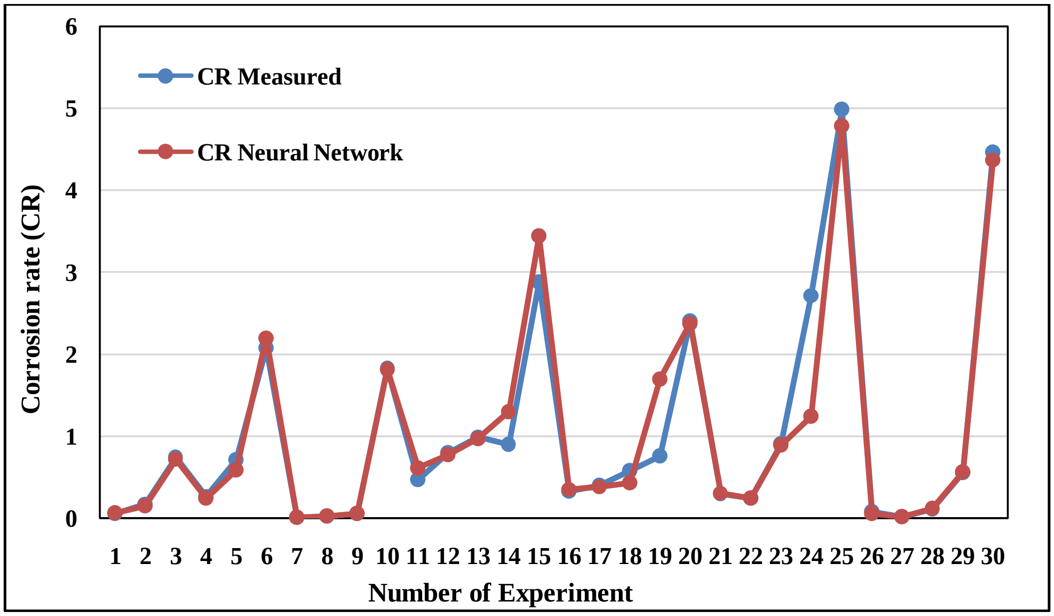

Figure 4. The corrosion rate (CR) amount based on measured and predicted by the ANNfor the training data set

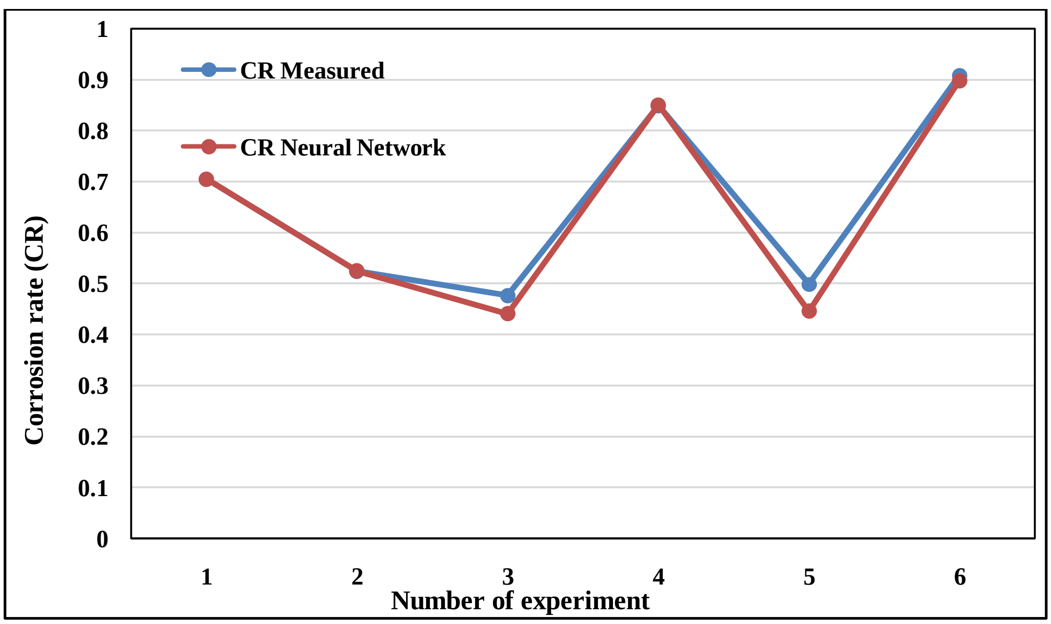

Figure 5. The corrosion rate (CR) amount based on measured and predicted by the ANNfor the validation data set

Detailed error analysis for the training and validation data sets is presented in Table 7, for corrosion rates
.

Table 7. Error analysis for the training and validation data sets

Error analysis of	Training data set	Validation data set
Maximum error	55.5%	11.8%
Minimum error	0.2%	0.06%
Average	15.5%	3.5%
Standard deviation	1.2%	0.2%

It can be concluded from Table 7 that the developed model has a low percentage of error for both set of training and validation data sets, which indicates the significant fitness of the model with the measured data. The small values of standard deviation also confirmed the accuracy of the predictive model.

d) Comparison of three models

Three models for prediction of corrosion rate were developed. Models are designed to predict the values of corrosion rate in different environmental conditions including various corrosion product layers, temperature, pH value and chloride ions concentration. In order to investigate performance of each proposed model, we selected 6 data sets (i.e. utilized validation data set for the ANNcomputation) and their predicted corrosion rate by the optimal solution, curve fitting and ANNtechniques are reported in Table 8. Furthermore, amount of the predicted corrosion rate error in reference to measured once demonstrated.

Table 8. Performance of each proposed model for validation data set

Case	**CR measured**	**CR Optimal solution**	**CR curve fitting**	**CR neural network**	**%Error** Optimal solution	**%Error** Curve fitting	**%Error** neural network
1	0.705	0.2697	0.795	0.7046	61.74	12.76	0.05
2	0.524	0.8659	0.2748	0.5243	65.25	47.5	0.05
3	0.476	0.2678	0.4313	0.4402	43.74	8.82	8.13
4	0.849	0.3977	0.7978	0.8502	0.3977	6.03	0.14
5	0.498	0.6769	0.3911	0.4453	35.9	21.4	11.83
6	0.908	0.702	0.320	0.8978	2.7	64.7	1.13
Average	0.66	0.53	0.50	0.64	34.95	26.86	3.55

As it is indicated in table above, the average of the error for the model developed by ANNis significantly low compared to the other two models.

Deviations from measured results are given in Figure 6. Maximal deviation of results predicted with optimal solution and curve fitting models compared to measured results is 0.45 and 0.58 mm/y in absolute terms, obtained in 4th and 6th instance respectively, while maximal deviation of results predicted with ANNequals 0.05 mm/y, obtained in 5th instance.

Comparison of the deviation of each model from the measured data indicates that the model developed by the ANNhas the minimum general deviation and is able to predict the corrosion rate with the highest accuracy among the other models.

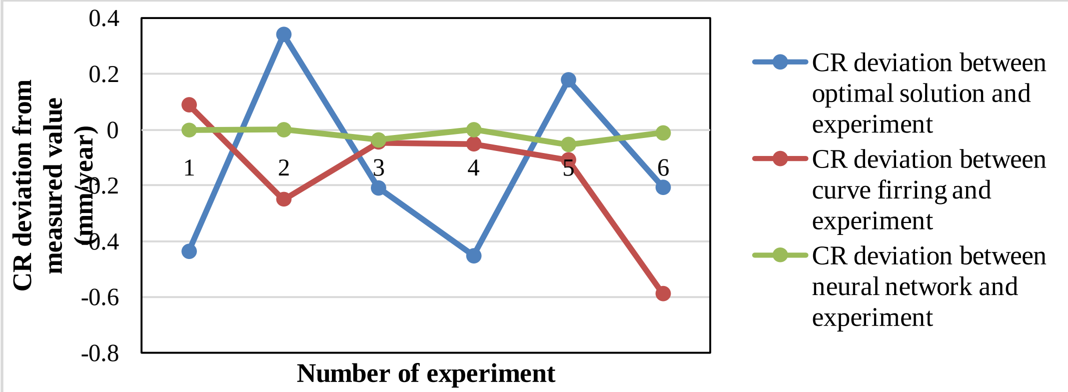

Figure 6. Analysis of corrosion rate (CR) difference between values obtained with models and measured results.

IV. CONCLUSION

From the conducted electrochemical measurements on 13% Cr steel and developed models, the following conclusions can be made:

- From the linear polarization resistance measurements, the corrosion rates for 13% chromium steels increases with the increase in chloride concentration and temperature however, it decreases with increase of pH values. This trend was observed in all series of experiments.
- Moreover from the linear polarization resistance measurements, the highest corrosion rates for 13% chromium steels were measured at the third series of experiments, in the presence of elemental sulfur at 75 ◦C, while the minimum ones were measured at the first series of experiments, without any initial corrosion product layer on the sample surface at 25 ◦C.
- The fitness between measured and predicted corrosion rates results by curve fitting indicates a good correlation between experiments and developed model.
- The minimum deviation between predicted and measured data was obtained with ANNmodel which is insignificant compared to the deviation of the two other models from the measured data.

The ANN model can be improved by optimizing the network parameters such as number of hidden layers, type of hidden layers and their transfer functions, learning algorithms and changing the ratio of training and validation data while in order to increase the accuracy of the curve fitting and optimal solution models is conducting more experiments and increasing the number of input data for the model.

V. ACKNOWLEDGEMENTS

The research in this paper is supported by the Suncor Reservoir Souring Initiative at Memorial University of Newfoundland. We acknowledge Seyedfakhreddin Nabavi for his comments on the model predicted by curve fitting and ANN.

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