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CORROSION INHIBITION STUDY OF CARBOXYMETHYL CELLULOSE-IONIC LIQUID VIA ELECTROCHEMICAL AND MACHINE LEARNING TECHNIQUE

(Kajian Perencatan Kakisan Menggunakan Karboksimetil Selulosa-Cecairan Ionik Melalui Teknik Elektrokimia dan Pembelajaran Mesin)

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Abstract

Corrosion is a natural phenomenon defined as the deterioration of a substance or its properties due to interactions between the substance and the environment. Prolonged exposure to corrosive environment had negative consequences, including increased repair and maintenance costs, decreased structural integrity, and fatalities. An approach to address the issue is to use a corrosion inhibitor. Numerous inhibitors have lately been developed or made accessible on the market. However, some could be dangerous or contain of volatile organic compounds (VOCs). Our study introduces carboxymethyl cellulose (CMC) mixed with 1-ethyl-3-methylimidazolium acetate ([EMIM][Ac]) ionic liquid, also known as CIL, as a corrosion inhibitor on mild steel in seawater. The functional group of combined CIL was examined using Fourier transform infrared spectroscopy (FTIR). The study employed mild steel specimens immersed in varying concentrations of CIL, subject to electrochemical impedance spectroscopy (EIS) measurements at different temperatures. The obtained result of EIS measurement were analyzed to calculate corrosion inhibition efficiency (IE) of CIL. 46 of electrochemical data were fed into a machine learning technique to forecast the effectiveness of the inhibition. Results indicate when inhibition concentration rises, so does inhibition efficiency (IE). The inhibitory efficiency of CIL decreased as the temperature of the test solution rose. At an ambient temperature of 950 ppm, an IE result of 83% was recorded. Levenberg-Marquardt (LM), Bayesian Regularization (BR), and Scale Conjugate Gradient (SCG) training algorithms were compared via Neural Network Fitting Tool (NNTool). LM was found to be the best backpropagation training algorithm, providing the highest regression value (R) of 0.907 and the lowest mean square error (MSE) of 0.006 when compared to BR and SCG. With R value closer to 1 and MSE close to 0, the use of Artificial Neural Networks (ANN) appears to offer a new insight in predicting methods with the goal of easing the hassle and time-consuming of experimental work.

Keywords: carboxymethyl cellulose, corrosion inhibitor, electrochemical, ionic liquid, machine learning

Abstrak

Kakisan adalah fenomena semula jadi yang ditakrifkan sebagai kemerosotan bahan atau sifatnya akibat interaksi antara bahan dan persekitaran. Pendedahan yang berpanjangan kepada persekitaran yang mengkakis memberi kesan yang negatif, termasuk peningkatan kos pembaikan dan penyelenggaraan, penurunan integriti keatas struktur dan mengakibatkan kemusnahan. Salah satu penyelesaian kepada isu ini ialah menggunakan perencat kakisan. Banyak perencat kakisan telah dibangunkan atau boleh didapati di pasaran. Namun, sebahagian daripadanya mungkin berbahaya atau mengandungi banyak sebatian organik meruap (VOC). Kajian ini memperkenalkan karboksimetil selulosa (CMC) dalam 1-etil-3-metilimidazolium asetat ([EMIM][Ac]) cecair ionik, juga dikenali sebagai CIL, sebagai perencat kakisan pada keluli lembut dalam air laut untuk menangani isu alam sekitar. Kumpulan berfungsi kimia hasil dr campuran CIL ini telah diperiksa menggunakan spektroskopi inframerah transformasi Fourier (FTIR). Kajian ini menggunakan spesimen keluli lembut yang direndam dalam kepekatan CIL yang berbeza, diuji menggunakan spektroskopi impedans elektrokimia (EIS) pada suhu yang berbeza. data pengukuran EIS yang diperolehi dianalisis untuk mengira kecekapan perencatan kakisan (IE) CIL. 46 data elektrokimia digunakan di dalam teknik pembelajaran mesin (ML) untuk meramalkan keberkesanan perencatan. Keputusan menunjukkan bahawa apabila kepekatan perencatan meningkat, kecekapan perencatan juga menunjukkan peningkatan. Kecekapan perencatan CIL menurun apabila suhu larutan ujian meningkat. Pada kepekatan 950 ppm pada suhu ambang, keputusan kecekapan perencatan dengan peratusan 83% telah direkodkan. Algoritma latihan Levenberg-Marquardt (LM), Bayesian Regulisasi (BR), dan Gradien Konjugasi Skala (SCG) telah dibandingkan melalui aplikasi Ketepatan Jaringan Saraf (NNtool). LM didapati sebagai algoritma latihan rambatan belakang terbaik, memberikan nilai regresi (R) tertinggi 0.9072580 dan ralat min kuasa dua (MSE) terendah sebanyak 0.0054787 jika dibandingkan dengan BR dan SCG. Dengan nilai R yang menghampiri 1 dan MSE menghampiri 0, penggunaan Rangkaian Saraf Tiruan (ANN) memberi pandangan baharu dalam kaedah peramalam dengan matlamat untuk mengurangkan kerumitan dan penghambatan kerja eksperimen yang memakan masa.

Kata kunci: karboksimetil selulosa, penghambat pengaratan, elektrokimia, cairan ionik, pembelajaran mesin

Introduction

Mild steels are the most extensively used structural materials in a variety of industries due to their good material qualities such as mechanical durability, ease of processing, and low manufacturing cost. This material is frequently utilized in industries as a primary building material for the creation of structural shapes, automobile parts, buildings, plants, pipelines, bridges, and tin cans [1]. Mild steel, on the other hand, is prone to corrosion when exposed to conditions with high moisture levels and corrosive substances [2, 3]. Corrosion is defined as the degradation of a substance or its qualities because of interactions between the substance and its surroundings. It would be more feasible to avoid corrosion rather than strive to entirely eradicate it [4]. Hence, a corrosion inhibitor is utilized by adding it to a closed system to slow down, prevent, and lower the rate of corrosion [5]. Natural products are frequently being researched for corrosion inhibitors as an alternative to conventional corrosion inhibitors due to recent environmental concerns [6, 7, 8].

Carboxymethyl cellulose (CMC) is a cellulose derivative that has been identified as the most abundant and widely used cellulosed derivatives due to the presence of polar functional groups such as carboxyl and hydroxyl groups, which contribute significantly to CMC's strong adsorption ability to metal structures [9, 10]. Despite the fact that CMC is frequently used in the food and pharmaceutical industries [11], some research has indicated that this cellulose derivative is also effective as a corrosion inhibitor [9, 12]. In this project, 1-ethyl-3-methylimidazolium acetate ([EMIM][Ac]) ionic liquid was chosen as the CMC solvent due to its loosely bounded anions, which can strongly interact with the polar functional (-OH, $-NH_2$) groups of polysaccharides, causing them to solubilize and increasing the performance of CMC inhibitor [13]. The objective of this study is to investigate the stability and polarization resistance of carboxymethyl cellulose ionic liquids (CIL) as corrosion inhibitor for mild steel in seawater incorporate ANN for prediction study. Most researchers commonly utilize acidic and artificial NaCl electrolytes in their electrochemical studies involving CMC and CMC with additive [15-24]. In contrast, this research opts for natural seawater. The selection to employ seawater as the electrolyte offers the advantage of investigating the material's behavior in marine environments and offshore applications. This approach aids in gaining insights into the natural corrosion effects in the environments and contributes to a better understanding, ultimately facilitating the development of strategies to mitigate corrosion in such settings. To reduce the time and effort required for laboratory work, a computational method based on machine learning was used to make predictions.

Artificial Neural Network (ANN) approaches were a viable choice for describing the corrosion inhibition behavior of carboxymethyl cellulose (CMC) in 1-ethyl-3-methylimidazolium acetate ionic liquid (CIL). The Levenberg-Marquardt (LM), Bayesian Regularization (BR), and Scaled Conjugate Gradient (SCG) training algorithms were used, and their performance indicators were analyzed. Furthermore, no machine-learning technique has been used to predict the corrosion inhibition of carboxymethyl cellulose in the ionic liquid ([EMIM][Ac]). By computing the corrosion inhibition efficiency prediction by using machine learning serves as an alternative to extrapolate the experimental data. The establishment of this model would be another new finding where it would beneficial to the research community. This model can be used to determine the corrosion inhibition behavior without the need for replicating the same experiment. This precise and robust model brought a new insight for the researchers parallel with the progression in Industrial Revolution 4.0 (IR 4.0).

Materials and Methods

Sample preparation

the experiment employed a square form of marine grade mild steel-chemical composition-carbon 0.051%, manganese 0.734%, silicon 0.126%, sulphur 0.004%, phosphorus 0.016% and iron remaining [14] with dimensions of 25mm x 25mm x 3mm. Prior to the EIS experiment, the coupons were polished with multiple grits (120, 600, 800, and 1200 grit) of emery paper to remove defects and attain the desired surface roughness. The coupon was then washed with acetone, rinsed with distilled water, quickly dried in air, and stored in desiccators before use. Carboxymethyl cellulose is constantly mixed with 1-ethyl-3-methylimidazolium acetate ([EMIM][Ac]) at 60°C for 30 minutes. Following that, the prepared inhibitor is subjected to Fourier transform infrared (FTIR) spectroscopy utilizing an INVENIO FT-IR Spectrometer with a wavelength range of 400 to 4000 cm^{-1} and a spectra resolution of 4 cm^{-1} to identify and analyze the functional group of a sample based on its interaction with infrared light. Immersion of mild steel in seawater is conducted for three hours to allow the adsorption of CIL onto the mild steel surface. The seawater was collected from Institute of Oceanography and Environment (INOS) Universiti Malaysia Terengganu.

Electrochemical impedance spectroscopy (EIS)

The impedance analysis of the mild steel interface in the presence and absence of CIL in seawater were characterized by using electrochemical impedance spectroscopy (EIS) Gamry 1010E. Three electrodes corrosion cell setup was used where the mild steel was employed as working electrode, Ag/AgCl as reference electrode, and platinum rod as counter electrode. Those electrodes were placed in a 200 mL beaker of seawater as electrolyte solution. For each experiment, volume of the test solutions was maintained to ensure the consistency of the parameter to all experiment. The testing parameters for the EIS are tabulated in Table 1. The ambient temperature was a foundational reference in standard laboratory conditions, establishing a baseline for electrochemical studies [25]. The elevated temperature of 50 °C was chosen to examine the moderate temperature effects on the corrosion inhibitor's behavior. At high temperature, 70 °C was utilized for higher temperature investigations. The decline in inhibition efficiency might happen as temperature rises was a characteristic observed in inhibitors physically adsorbed onto the metal surface resulted in desorption of some adsorbed corrosion inhibitor molecules from the metal surface[22, 26]. Electrochemical impedance spectroscopy (EIS) experiments were performed with a small amplitude perturbation 10 mV (peak-to-peak) for an AC signal where the surface area of the mild steel coupon was 3.125cm^2.

Table 1. Testing parameter of CIL for EIS experiment

Temperature (°C)	**Concentration (ppm)**	**Frequency (Hz)**
	0	
Room Temperature	350	
50	550	0.1 – 1000000
70	750	
	950	

The corresponding data then analyzed by using Gamry 1010E EChem Analyst. Determination of the corrosion respond parameters in the Nyquist plot was obtained via construction of equivalent circuit compatible with the Nyquist diagram. To fit the model using simplex method, theoretical transfer function Z(ω) was constructed parallel with the combination of a resistance R_p and a constant phase element (CPE), then, both in series with another solution resistance R_s as shown in Figure 1. To validate the suited equivalent circuit, the experimental data are fitted to the equivalent circuit and the circuit elements are obtained [27].

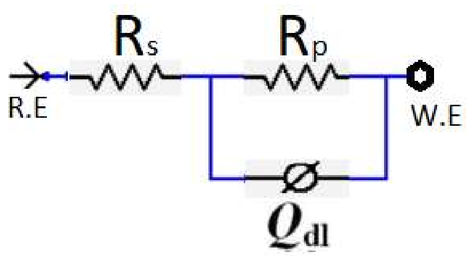

Figure 1. Equivalent circuit for the EIS Analysis

The inhibition efficiency (IE) was determined from Polarization Resistance, R_p according to Equation 1:

$$IE(\%) = \frac{R_{p(inhibited)} - R_{p(uninhibited)}}{R_{p(inhibited)}} \times 100\% \qquad (1)$$

Artificial neural network

The Multilayer Feed Forward Neural Network (MFNN) was utilized to estimate the effectiveness of CIL's inhibition using MATLAB® software (R2021b, MathWorks®, Natick, MA, USA). The experimental data obtained from EIS characterization served as the source of the 46 datasets for the modelling investigation. Min-max technique of data normalization was adopted in this study which in the end, the values are between 0 and 1 for an accurate predictive model. The technique is based on the following Equation 2.

$$x = \frac{x_{input} - x_{min}}{x_{max} - x_{min}} \qquad (2)$$

where x = (x1, . . ., xn) and x is the normalized data.

A satisfactory normalization is one of the most important aspects of the training process, which represents a direct influence on the model and offers benefits such as suitable results and a considerable decrease in calculation time [28].

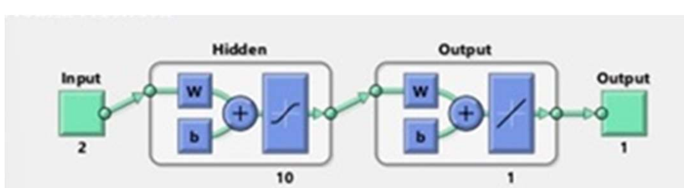

Figure 2. Artificial Neural Network Architecture

To forecast the inhibition efficiency (IE) behavior model of CIL, the Neural Network Toolbox in MATLAB was employed. The default settings of the NNTool were utilized, with 70% (32) of the data allocated for training, 15% (7) for validation, and 15% (7) for testing purposes, chosen randomly from a dataset of 46. The distribution of data between training, validation, and testing in a model of small data range to ensures the chosen subset accurately represents the entire dataset, reducing bias and minimizing the risk of overfitting [29]. As shown in Figure 2, architecture models are composed of three layers with a 2:10:1 structure. Temperature (°C) and Concentration (ppm) were the two parameters that made up the first layer, also known as the input layer. The model is built using 10 nodes in the hidden layer, and the output layer predicts inhibition as IE. As training algorithms, Levenberg-Marquardt (LM), Bayesian Regularization (BR), and Scaled Conjugate Gradient (SCG) were employed. Since there is no established standard procedure, a trial-and-error approach using 10 nodes with one concealed layer is used [30]. Performance Indicator (PI) used to measure the effectiveness and efficiency ANN developed. Here, 2 PI are used. Regression accuracy R is used to measure accuracy and mean square error (MSE) is used to measure error. The accuracy measures evaluate values from 0 to 1, whereby the best model is considered when the evaluated values are close to 1, while the best model for error measures are selected if the evaluated value is close to 0.

Results and Discussion

Fourier transform infrared (FTIR) spectroscopy

Figure 3 depicts the FTIR spectra of the cellulose ionic liquid (CIL). It displays a notable peak in the range of 3600-3100 cm^{-1}, corresponding to the stretching vibration of the O-H (hydroxyl) group [31]. This FTIR range revealed changes in the O-H stretching peak, signifying modifications in the hydrogen bonding pattern. The peak's increased broadening and intensity indicate the development of new formations of hydrogen bonds due to the disruption of the compact structure of the intramolecular hydrogen bonding in the glucose units of CMC [32]. The peak at the range of 1500-1620 cm^{-1} corresponds to the stretching vibration of the C=C group that can be found in the imidazole ring stretch [33]. Based on [34], it is suggested that around 1700 cm^{-1} corresponds to the C=O group and a peak at around 1200 cm^{-1} corresponds to C–N stretching. The peak at 1016 cm^{-1} in the FTIR range is typically associated with the symmetric stretching vibration of C–OC– in CMC [35]. The alkanes functional group exhibited spectral IR bands at around 750 cm^{-1} due to C-H rocking vibrations and at 600 cm^{-1} due to C-H bending vibrations [34]. The FTIR analysis revealed the presence of characteristic functional groups in the CMC mixed with the ([*EMIM*][Ac]) mixture, which was consistent with its expected molecular structure.

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Electrochemical impedance spectroscopy (EIS)

The corrosion inhibition efficiency of mild steel using CIL at Open Circuit Potential (OCP) was investigated by the area-normalized EIS tested in different concentration and solution temperature. With the different concentration used in the experiment, the result of polarization resistance, R_p shows an increasing trend of value as seen in Figure 4 and Table 2. The R_p for mild steel in the absence of CIL at ambient temperature was recorded with the value of 163.4 $\Omega.cm^2$. The R_p value of inhibited sample at ambient temperature for 350, 550, 750 and 950 ppm were recorded at 274.2, 482.6, 665.4, and 980.0 $\Omega.cm^2$ respectively. The calculated inhibition efficiency, IE were 40.30%, 65.98%, 75.12% and 83.09% for 350, 550, 750 and 950 ppm respectively. The increase of inhibition efficiency with increased concentration of the inhibitor may be attributed to increasing of the number of –O–,–OH, and –COOH groups, which increased the surface coverage of CMC molecules on the metal surface [9]. With the concentration variation, the electrochemical change is indicative of inhibition of corrosion process at increased concentration of the inhibitors by adsorbing on the surface of the metal thereby reducing the active surface area exposed to the aggressive corrosive electrolyte medium [1].

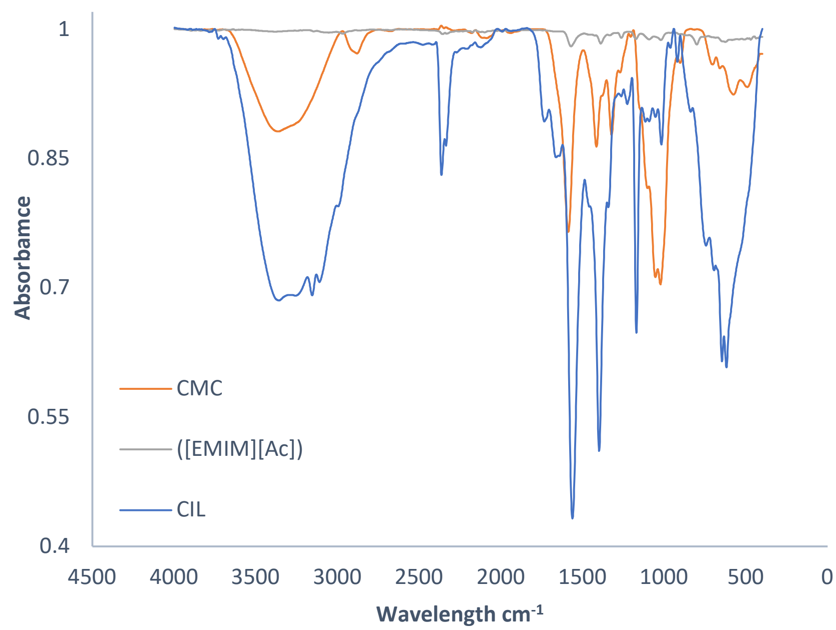


Figure 3. FTIR spectrum of CIL

Table 2. Impedance data for Mild steel in different concentration and temperature effect on CIL in seawater

Concentration (ppm)	Temperature (°C)	R_p (Ω.cm^2)	R_s (Ω.cm^2)	Y_0 (F)	IE %
blank	ambient temperature	163.400	3.129	0.014	0.00
350		274.200	4.739	0.004	40.30
550		482.600	6.856	0.005	65.98
750		665.400	3.914	0.020	75.12
950		980.300	4.231	0.014	83.09
blank	50	120.700	3.063	0.032	0.00
350		157.300	5.233	0.003	23.85
550		217.900	4.677	0.004	44.40
750		391.200	7.068	0.010	68.92
950		340.100	6.993	0.005	64.34
blank	70	85.270	3.111	0.031	0.00
350		155.400	5.465	0.008	45.06
550		201.600	3.239	0.004	56.85
750		295.600	4.876	0.002	70.59
950		249.200	4.876	0.002	65.21

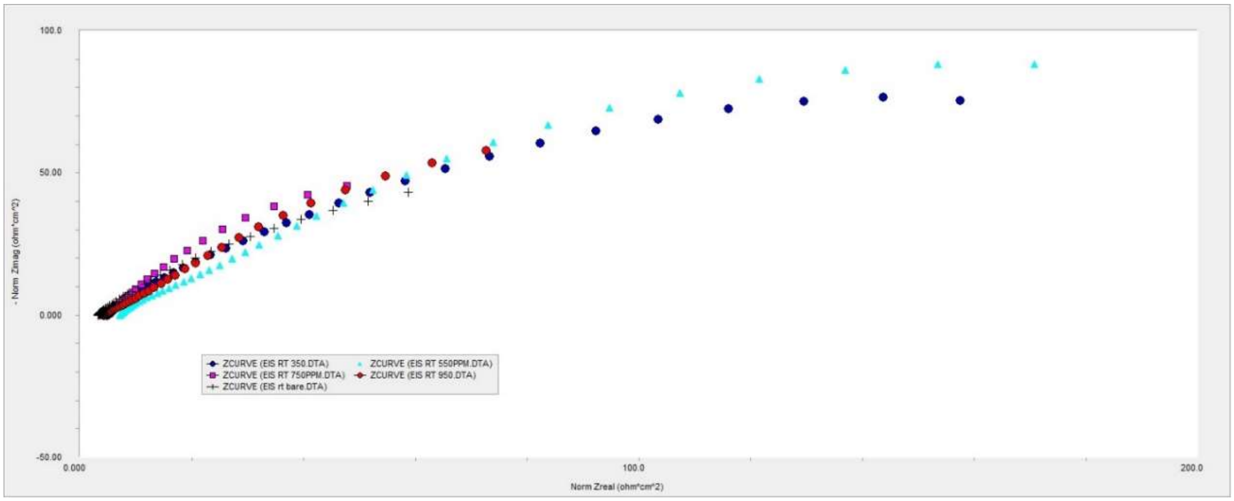


Figure 4. Nyquist Plot at ambient temperature

When testing the sample with a range of different temperatures, as shown in Table 2, the R_p for mild steel in both the presence and absence of CIL was recorded, with lower values obtained compared to the sample tested at ambient temperature. At 50 °C, the R_p recorded for mild steel in the absence of CIL was 120.7 Ω.cm^2. The R_p values of the inhibited sample at 50 °C for 350, 550, 750, and 950 ppm were recorded at 157.30 Ω.cm^2, 217.90 Ω.cm^2, 391.20 Ω.cm^2, and 340.10 Ω.cm^2, respectively. The inhibition efficiency was calculated as 23.85%, 44.40%, 68.92%, and 64.34% for 350, 550, 750, and 950 ppm at 50 °C, respectively, as shown in Table 2.

The Nyquist plot of inhibited and uninhibited samples tested at 70°C is shown in Figure 6. The plot shows a nearly perfect semicircle indicates that the electrochemical process is mainly contributed by the charge transfer process. Charge transfer is the difference in real impedance axis at low and high frequency regions. At 70 °C, the recorded R_p for mild steel in the absence of CIL was 83.27 Ω.cm^2. The inhibited samples record the R_p value at 155.40 Ω.cm^2, 201.00 Ω.cm^2, 295.60 Ω.cm^2, and 249.20 Ω.cm^2 for 350, 550, 750, and 950 ppm respectively. The calculated inhibition efficiency was 45.06%, 56.85%, 70.59% and 65.21% for 350ppm, 550ppm, 750ppm, and 950ppm at 70 °C respectively.

Temperature has a significant impact on corrosion rates and inhibiting efficiency. With the increase in the testing solution temperature from ambient to 70 °C, the inhibition efficiency decreased. At higher temperatures, the adsorption process may be hindered or reversed, leading to reduced inhibitor coverage and decreased corrosion protection due to reduced adsorption, as well as faster corrosion reactions because of the higher kinetic energy [36]. However, at temperatures of 50 °C and 70 °C, the inhibition efficiency decreased for both concentrations of 950 ppm. This might suggest a decrease in the effectiveness of the inhibition as the sample experienced the limitation of inhibitor adsorption. This occurs when inhibitor molecules do not have enough opportunities to adsorb onto a metal surface. According to [37], the decreased inhibition efficiency as the temperature increases is due to the physisorption mechanism, which might be attributed to an increase in the solubility of the precipitated inhibitor molecules on the mild steel surface. With the decrease in the number of molecules absorbed on the mild steel surface as the temperature rose, this resulted in a deficiency in the inhibitive performance.

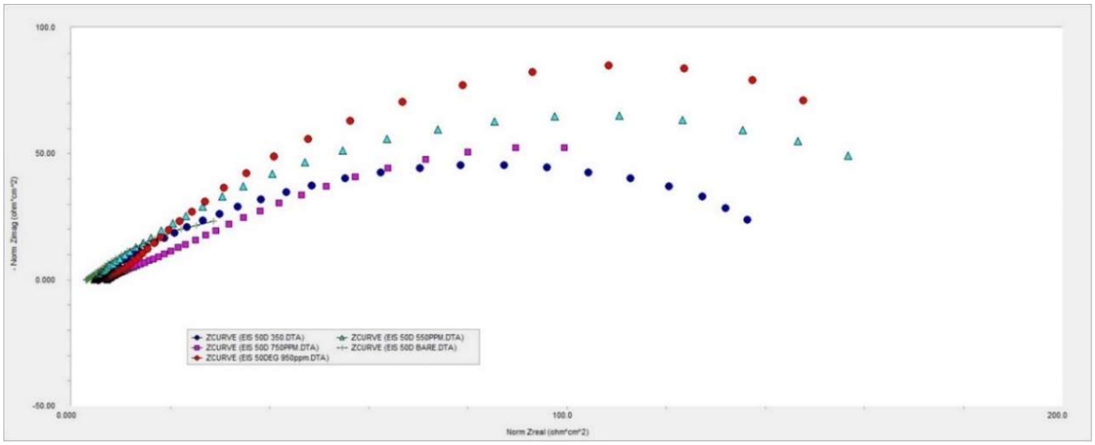


Figure 5. Nyquist Plot of CIL at 50°C testing solution temperature

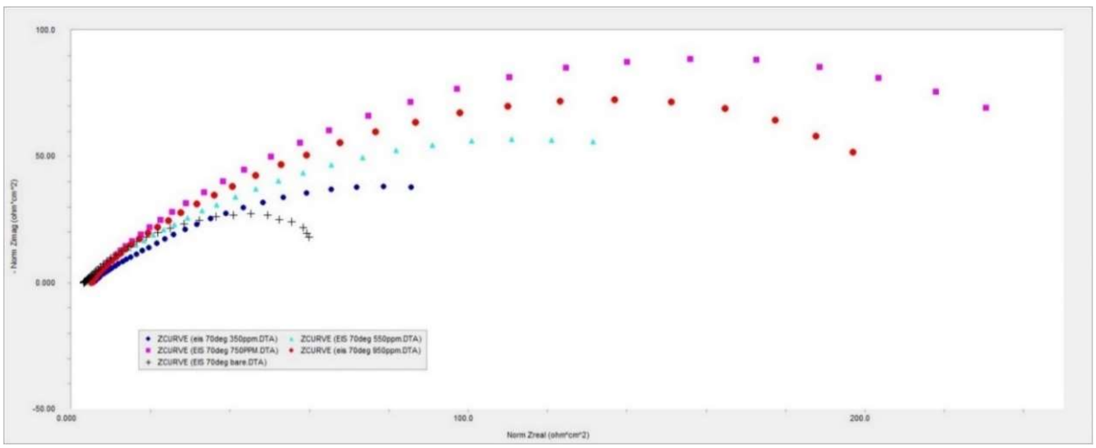


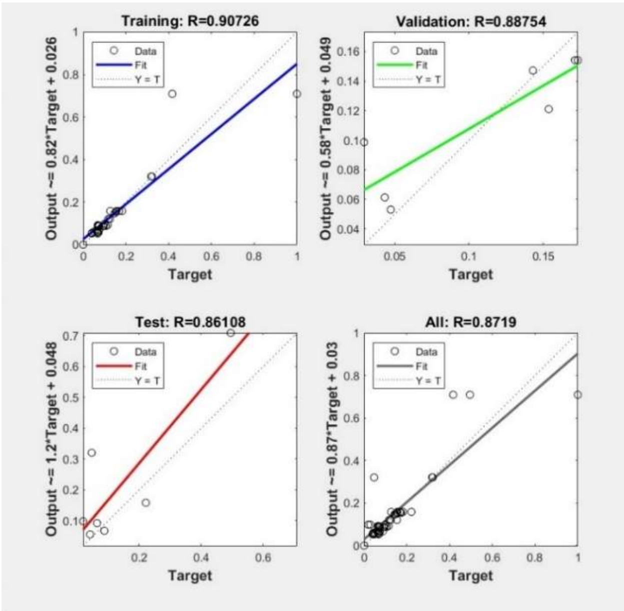
Figure 6. Nyquist Plot of CIL at 70°C testing solution temperature

Artificial neural network

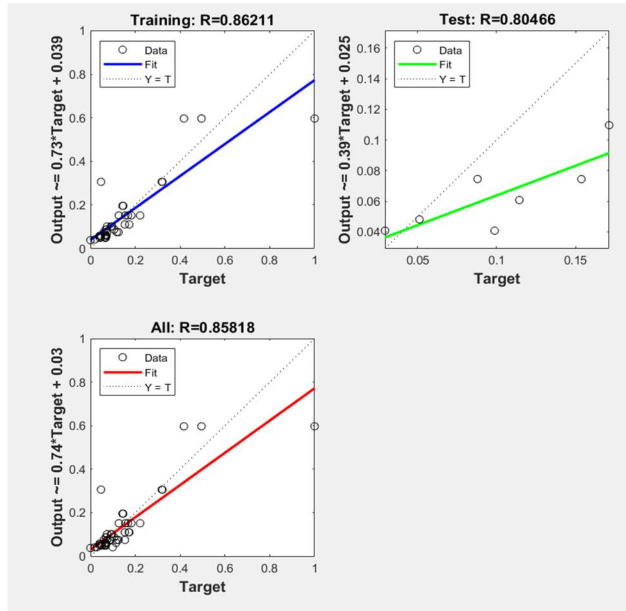
Table 3 shows the values of the performance indicator (PI) obtained from the modelling study via an artificial neural network. The correlation coefficients (R) for the Levenberg Marquardt (LM) training algorithm in the training, validation, and testing phases were 0.907258, 0.887539, and 0.861082, respectively. Based on the fitted line shown in Figure 7a, the fitted slope line falls within the expected range, showing the good relationship between the predicted and observed values obtained for the training, validation, and testing samples. The results from the Bayesian Regularisation (BR) training algorithm showed slightly lower precision as the R values for the training and testing phases were 0.862106 and 0.804661, in comparison to those obtained from LM, as shown in Figure 7b. The Scale Conjugated Gradient (SCG) training algorithm produced R values of 0.359149, 0.828020, and 0.333080 for training, validation, and testing, respectively. The SCG had the lowest precision of all the training algorithms.

Table 3. Performance indicator of training algorithm

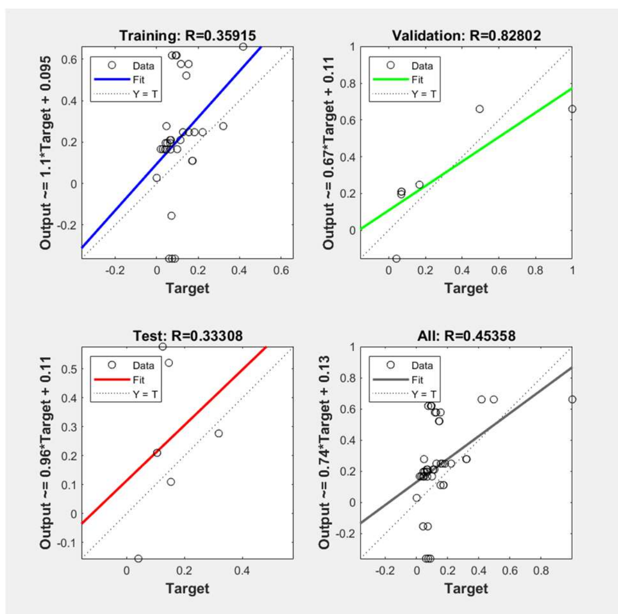
Training Algorithm	LM		BR		SCG	
PI	MSE	R	MSE	R	MSE	R
Training	0.005	0.907	0.008	0.862	0.073	0.359
Validation	0.001	0.888	-	-	0.035	0.828
Testing	0.019	0.861	0.002	0.805	0.058	0.333



(a)



(b)



(c)

Figure 7. Correlation of training, validation, testing and overall, between the actual and predicted corrosion rate for (a) Levenberg–Marquardt (LM), (b) Bayesian Regularization (BR) and (c) Scale Conjugate Gradient (SCG training algorithm for CIL inhibition efficiency prediction

Overall, the LM was found to be the best training algorithm. Other study also shows the use of LM gave better outcome [30]. This could be due to the LM backpropagation training algorithm is a modified version of Newton's method. The Levenberg-Marquardt algorithm also designed to approach second-order training speed without having to compute the Hessian matrix [38]. LM was selected as Backpropagation

Learning algorithm as it is known for its efficiency in minimizing the sum of squared errors and its ability to converge faster.

Conclusion

Corrosion inhibition performance of cellulose-ionic liquid (CIL) tested on marine grade mild steel with variation of temperature (°C) and concentration (ppm) using electrochemical analysis and further analyzed using Artificial Neural Network (ANN) has shown the promising result and observation. It was found that FTIR analysis shows the presence of functional groups in CMC mixed with ([EMIM][Ac]) mixture by formation of new hydrogen bonds as result of disrupting and breaking compact structure of intramolecular hydrogen bond of CMC which provides an adsorption site between the inhibitor and the metal. With increasing of CIL concentration, the result gives the higher inhibition efficiency and reducing corrosion rate. With the increase of testing solution temperature from ambient to 70°C, the result of inhibition efficiency is decreasing. LM was found to be the better training algorithm as the regression R is highest and MSE is lowest compared to BR and SCG. With the implication of the research result, CIL has the potential to be used as a natural and environmentally friendly corrosion inhibitor. To drive research forward with innovation and new techniques, the future prospects on enhancing the effectiveness of CIL as corrosion inhibitors should be implemented. This involves exploring various parameters including the immersion period, different CIL concentration or use of different inhibitor formulation. Assessing the corrosion condition under long period of time to simulate realistic conditions, testing CIL performance using different materials, comparing with different combination of ionic liquid, and conducting a thorough environmental impact assessment are possible to be implemented as well. Additionally, an approached of research on CIL interacts and influences marine biofilms on metal surfaces will give significance for the development of new technologies and implementation in marine industries. This feature acknowledges the intricate relationships that exist between corrosion inhibitors and the dynamic of marine environment, providing insights that lead to innovative solutions for effective corrosion protection in real-world marine applications

Acknowledgement

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