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Value added biodiesel for corrosion inhibition

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Biodiesel is one of the prospective fuels, potentially capable of substituting petroleum fuel. Nevertheless, systems using this bioenergy resource are prone to corrosion than conventional fuels. Here, corrosion of copper metal in biodiesel with value added green coffee bean inhibitor has been evaluated by weight loss method, which produced 95.92% inhibition efficiency. Theoretically, corrosion is evaluated by artificial intelligence. The images of the surface obtained using CCD are augmented to 699 image samples. These augmented images are fed to back-propagation based neural network system for training, validation and classification for prediction of corrosion behaviour of copper in biodiesel with and without inhibitor. The neural network system has training, validation & testing prediction accuracies of 97.1%, 96.2% & 98.1%, respectively, and an overall accuracy of 97.1%. The proposed tool can be used to assess the corrosion behaviour dynamically in real time for futuristic prediction of corrosion behaviour of various metals including copper.

Keywords: Artificial intelligence, Biodiesel, Corrosion inhibition, Corrosion prediction, Deep learning

Introduction

Biodiesel is a source of bioenergy that are obtained from various biomasses such as vegetable oils, animal fats and seaweeds. Biodiesel is a sustainable alternative fuel for diesel, which is predicted to gain share in the transport market. Fossil fuel depletion and environmental degradation can be addressed with the substitution of renewable resources, particularly biodiesel for diesel[1,2]. However, metals in biodiesel do not have compatibility with each other that end in corrosion. Studies on the biodiesel initiated corrosion of different automotive materials such as metals/alloys and composites were evaluated by scientists throughout the globe[3-5].

Corrosion is one of the major problems in structural systems and leads to massive failure causing severe loss to lives and economy[6-8]. The requirement of corrosion risk assessment is increasing globally and limitations in current practices are not able to provide timely and accurate prediction outcomes. Notably, the biodiesel composition influences directly the corrosion resistance of metals present in the fuel circuit. Biodiesel becomes more corrosive when water and free fatty acids are present. The biodiesel corrosiveness also depends on the types of feedstock due to the difference in their chemical composition, especially regarding the unsaturation degree, which leads to the degradation process and formation of products with different degrees of corrosiveness [9].

However, biodiesel has been found to be more corrosive to automotive materials than diesel. The corrosion rates for copper, aluminium and steel immersed in sunflower oil based biodiesel for 3000 h are reported to be 0.008217 mmpy, 0.00412 mmpy and 0.004325 mmpy, respectively, at room temperature[10]. This is more likely due to the presence of oxygen moieties, auto-oxidation, increased polarity of biodiesel and its hygroscopic nature. Corrosion behaviour of different metals such as copper, brass, bronze, cast iron, carbon steel etc. in diesel and biodiesel was investigated by several researchers[11,12]. Copper alloys corrode more than ferrous alloys in biodiesel[13]. Sintered bronze nozzle was found to be affected by pitting corrosion after 10 h operation with biodiesel at 70°C[14].

To address this problem, value addition of biodiesel is done by blending them with corrosion inhibitors. Ethanol extract of rosemary leaves, Ricinus seed and *Vitex negundo* leaf extract were examined for its corrosion inhibition of metals, especially aluminium and copper in biodiesel and found to be efficient[15-17]. The antioxidant activities of the methanolic *Larrea tridentate* extract revealed its oxidative stability, considered as an alternative to the commercial synthetic antioxidant used in biodiesel[18].

Benzotriazole and adenine as corrosion inhibitors for copper and mild steel in palm biodiesel were evaluated for a duration of 60 days with static immersion tests between 25-27°C. Corrosion inhibitors such as benzotriazole and adenine at 100, 200, 300 ppm concentrations were added individually to palm biodiesel in which mild steel has degraded comparatively less than copper[19].

Guava leaf extract was used as an antioxidant additive to soybean biodiesel in order to verify its anti-corrosive properties. The study of the copper exposed to the said biodiesel with and without the said antioxidant was carried out at room temperature for 2520 h and at 60°C for 1440 h. However, the corrosion IE for the same is not impressive with only 55.37% and 21.11% at room temperature and 60°C, respectively[20].

Copper corrosion inhibition studies were done at different acid values and temperatures in neem oil biodiesel with *Ricinus communis* seed extract as corrosion inhibitor. Maximum of 95% inhibition efficiency was observed for copper in neem oil biodiesel. Aluminium corrosion in acidic conditions at different temperatures was controlled significantly with the extract of the green coffee bean[21]. Analogically, the potential of green coffee bean as corrosion inhibitor in neem based biodiesel is examined in this work.

Currently evaluation of corrosion is done by manual methods such as visual inspection, chemical, electrochemical, and mechanical analysis[22]. These techniques were able to quantify the amount of corrosion but they are tedious, time consuming and cannot evaluate real time corrosion monitoring. Deep Learning is the process of using computational models to analyze a data set to identify or extract the relationship between them through layer by layer learning. Deep learning is gaining popularity in a wide range of applications including drug discovery, genomics, weather prediction etc. Use of deep learning algorithm is gaining popularity in the prediction of behaviour of metals and changes they undergo due to stress, or under different environmental conditions.

Study and prediction of performance or impact of corrosion inhibitors on metal surfaces are also being evaluated by deep learning networks. Deep learning techniques are highly robust and able to identify, extract and predict outcomes in prediction studies[23]. Although study and development of large number of novel inhibitors are gaining pace it is equally important for the development of reliable, fast and accurate prediction tools for performance simulation of corrosion inhibitors.

Deep learning (also known as deep structured learning) is a part of broader family of machine learning methods based on Artificial Neural Networks (ANN) with representation learning. A corrosion rate prediction model with machine learning-based algorithm showed good accuracy. Limited studies are available in the literature related to mathematical and ANN investigations[24]. The current study enhanced the corrosion prediction model and proved the feasibility of Deep Learning in corrosion evaluation. The ANN has the ability to learn without any prior knowledge on the relationship between the environmental factor and it provides more robust prediction outcomes than conventional linear regression models. The ANN has a high capacity of classification ability and larger tolerance level compared to conventional modelling.

A machine learning based algorithm was developed for performance prediction of benzimidazole derivatives based corrosion inhibitors, where six different benzimidazole based inhibitors were designed and simulated for their efficiency of operation successfully[25]. A number of artificial intelligence based prediction algorithms were developed for corrosion studies. Theoretical and experimental results were compared to identify the best performing algorithm. Genetic Algorithm based artificial intelligence system is identified as the top performer[26]. Quantitative models were developed, which linked chemical structures and properties using deep learning algorithms. Linear and non-linear quantitative structure and property relationship models were established. These models were able to predict efficiency of corrosion inhibition for more than 41 pyridines, for which eight variables namely exponential of the calculated adsorption energy, LUMO, van der Waals surface area, van der Waals volume, polarizability, electron affinity, electrophilicity and electron donor capacity were taken for analysis[27]. Parameters such as Hammett constants, dipole moment, HOMO energy, LUMO energy and energy gap as data input for neural networks were considered to correlate and predict the efficiency of corrosion inhibition[28].

Artificial Intelligence based classification of corrosion behaviour of metals in response to corrosion inhibitors in biodiesel is evaluated in this work. Deep learning based training of artificial intelligence modules enables for faster, dynamic and accurate corrosion and inhibition prediction. We have used the Baysian Classification Algorithm in this study, which is more effective in terms of Root Mean Square when compared to other algorithms such as Support Vector Machine based classifier, Stepwise linear regression classifier; Tree based classifier, and linear regression classifier algorithm. Bayesian network are best known for prediction of incidents and outcomes by understanding their probalistic relationships. The principle of Bayesian network is that they represent a range of variables and their respective conditional dependencies in the form of a directed acyclic graph.

Experimental Section

Dried Green Coffee Bean powder of about 50 g were soaked in analytical grade ethyl acetate and subsequently warmed at 50°C for 1 h. After that, it was let to cool, then filtered and dried in vacuum oven for 24 h. The extract henceforth obtained in a paste form was used as a corrosion inhibitor.

Commercial neem oil was subjected to trans-esterification with methanol at 70°C in the presence of KOH catalyst for the preparation of neem biodiesel[29]. Standard solutions of biodiesel weighing 100 mL each were prepared separately in the absence and presence of green coffee bean extract inhibitor at different concentrations such as 0.5 ppm, 1 ppm, 1.5 ppm, 2 ppm and 2.5 ppm.

Copper specimens subjected to the weight loss experiments as per ASTM D130 of dimensions 7.5 cm x 1.25 cm x 0.15 cm were then polished using 400-1200 abrasive papers, rinsed with deionized water and wiped off with acetone[30].The experiments with copper specimens immersed in neem biodiesel for 24 h were carried out at 303K, 323K, 343K and 363K for different inhibitor concentrations as said above. These specimens were weighed before and after the experiments using SCHIMADZU AUX220 balance. After the surface treatment and polishing of the copper metal plates, they were suspended in biodiesel without and with inhibitor inside a beaker with the help of thread (Fig. 1).

ANN evaluations

Copper plates were pre-processed for ANN evaluations similar to weight loss corrosion evaluations. The surface of copper plates was digitally photographed with high precision state of the art machine vision system with fixed frames and references through a CCD image acquisition system. A number of sample copper plates were immersed in biodiesel without and with green coffee bean extract corrosion inhibitor for 24 h under standard atmospheric room conditions maintained throughout experimentation. The copper plate samples were taken out for surface image acquisition. All the surface image recordings were executed at constant light conditions.

The acquired digital images were pre-processed by noise removal and filtering. Noise removal was carried out using averaging filter. Filtering was done using Band pass filter algorithm. All the surface images of copper samples were labelled. The labelled images were segmented and features extraction were done. The number of image samples obtained were 20 which were augmented to 699 image samples using MATLAB through image augmentation for the purpose of data feed to deep learning.

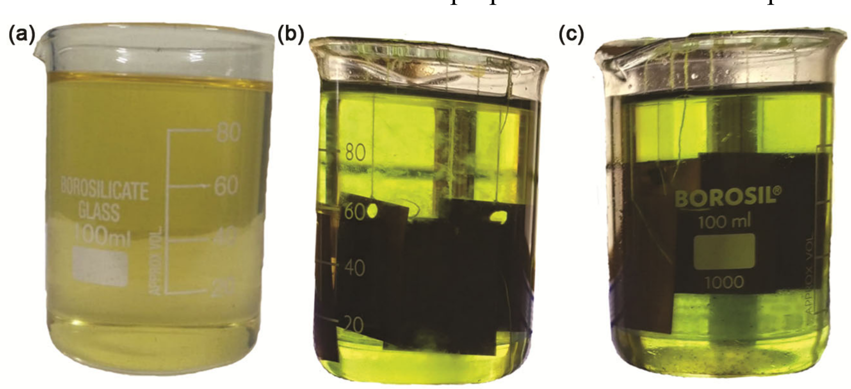

Fig. 1 — Corrosion weight loss evaluations with and without inhibitor in biodiesel

Key features and properties extracted from the pre-processed images as input for the neural network based trainer are corrosion area, peaks, solid edges, solid edge colours and solid edge faces. Neural Network Toolbox in Matlab^{TM} R2021 Software installed in Microsoft Windows 10 Operating System was used to run the deep learning network. The processor used is a Intel i7 Core 7th Gen Clock speed 3.60 Ghz, RAM 16 GB with Nvdia^{TM} GTX 1050 Graphics card with 3GB Frame Buffer RAM , Memory Speed 7 Gbps, Memory Bandwidth 84 GB/sec, Boost Clock 1518 MHz and Base Clock 1392 MHz capacity.

The simplified block diagram of the neural network architecture used in the current study is shown in Fig. 2, 699 input samples were given to the neural network as feed-in data which has 10 image features extracted from each of the input images. These features are represented in the hidden layer. These features will be used by the Neural Network to correlate the relationships among them based on the weights (W) and biases (b) computed for each and every processing state to predict the outcome. The output layer will be two outputs which will be classified by the neural network system based on the weights and biases along with the computed relationships in the hidden layer.

Results and Discussion

The coffee tree belonging to the Rubiaceae family, genus Coffee was reported to have heterogenous compounds such alcohols, esters, hydrocarbons, chlorogenic acid and aldehydes in addition to 100 different volatile compounds[31].These heterogenous compounds can perform as potential corrosion inhibitors for copper metal.

The phsico-chemical properties of the neem oil based biodiesel synthesized in the laboratory has an acid value of 1.97 mg KOH/g, viscosity of 8.70 mm^2/S, density of 0.839 g/cm, cloud point of 10.2°C, Pour point of 6.6°C, flash point of 93°C, fire point of 102°C as evaluated by standard methods adopted [15]. Appearance is light yellow colour.

The percentage of copper in test specimens employed for the present study as evaluated by BOWMAN - XRF Spectrometer, MODEL: GOLD SCOPE SDD, SERIAL NO- XI18120024SDD was 100% as given in Fig. 3.

Weight loss studies

Literature indicates that copper metal is incompatible with biodiesel, which will decrease the stability of biodiesel and increase its corrosiveness[32]. From Fig. 4, it is evident that the copper metal corrosion is very rampant in biodiesel alone, whereas

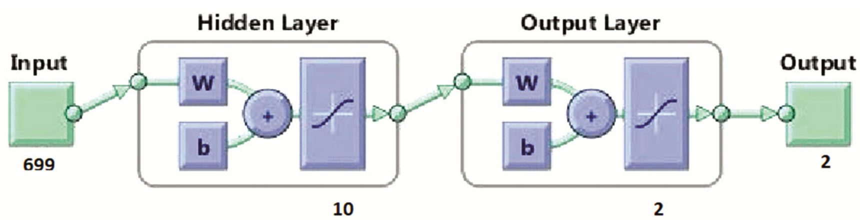

Fig. 2 — Neural network architecture

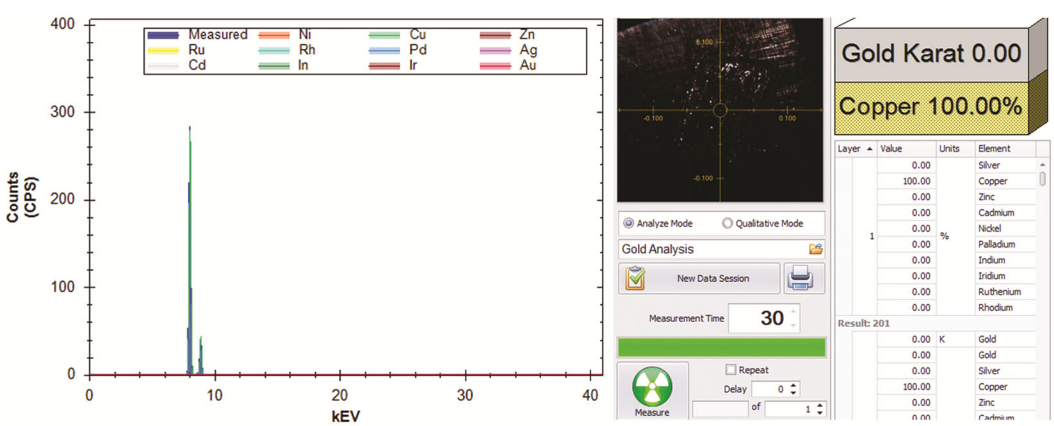

Fig. 3 — XRF image of copper metal employed for corrosion studies

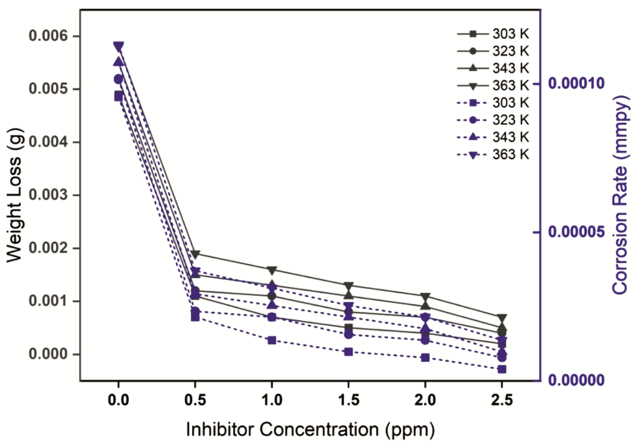

Fig. 4 — Different Inhibitor concentrations in neem biodiesel vs weight loss and corrosion rate at different temperatures

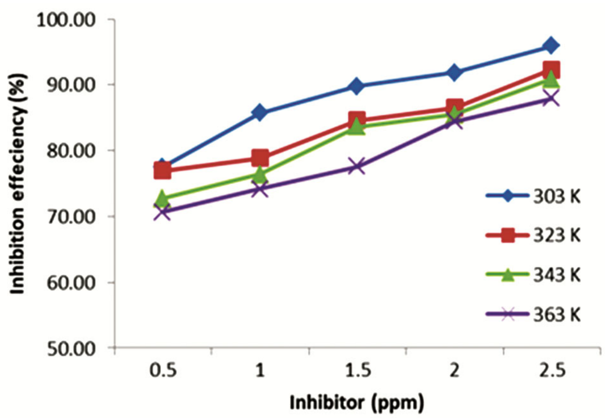

Fig. 5 — Different Inhibitor concentrations in neem biodiesel vs inhibitor efficiency at different temperatures

the corrosion in terms of weight loss is considerably less in the presence of inhibitors in biodiesel. The study also reveals that as the inhibitor dosage increases, the corrosion rate of copper in biodiesel decreases. This trend is observed across all temperatures investigated in the study as demonstrated in Fig. 4. The increase in corrosion inhibition with increase in inhibitor dosage, a maximum of 95.92% as shown in Fig. 5 is a further proof of the proposed corrosion inhibition.

Surface characterization of copper metal surfaces

Copper metal specimens exposed to biodiesel without and with 2.5 ppm inhibitor concentration were photographed at 1000X magnification using digital microscope to observe the surface morphology changes and the images are shown in Fig. 6a-c. Specimen (Fig. 6b) immersed in biodiesel alone is more damaged with tiny pits and fissures and became more rough along with colour change when

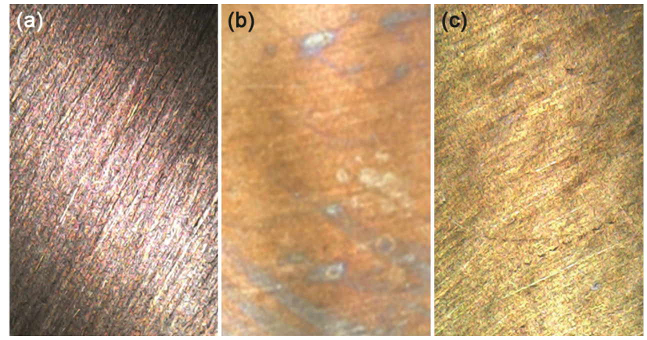

Fig. 6 — Copper specimens at 1000X Magnification: (a) Copper metal, (b) Copper in biodiesel without Inhibitor and (c) Copper in biodiesel with Inhibitor

viewed and compared with images of unexposed copper specimen (Fig. 6a) and copper specimen immersed in biodiesel with inhibitor (Fig. 6c). These diagrams unequivocally reveal the efficiency of the inhibitor in protecting the copper metal surface from biodiesel lured corrosion.

ANN analysis of copper corrosion in biodiesel

The main features of the corroded images analysed were corrosion area, intensity peaks, solid edges, colour of solid edges and solid edge faces. These features were extracted from the input corroded images and given to ANN for analysis. From the Fig. 7, it can be interpreted that the cross entropy level varies from a peak point up to 0.054197 at the epoch point 9 out of a maximum number of 15 epochs. Training above 9 epochs does not converge the system performance to further improvement. From Fig. 7, it is understood that the best validation performance is 0.054197 at the epoch 9.

Fig. 8 shows the distribution of errors during the prediction of corrosion behaviour. The number of error points disseminated nearer to the zero is higher which implies that, the neural network has a low error histogram and high performance in this corrosion prediction and classification model.

Fig. 9 (a) shows the variation of gradient coefficient vs Number of Epochs. The outcome of the training state executed at 15 numbers of epochs stood at 0.0051712, which is almost near to zero. Therefore, it is evident that a low value of gradient coefficient is attributed to a better performance of the training and testing states; hence the proposed prediction of corrosion inhibition study through neural network based prediction can be a viable tool. From the Fig. 9(a), it can be realized that the gradient coefficient keeps on decreasing on increasing number

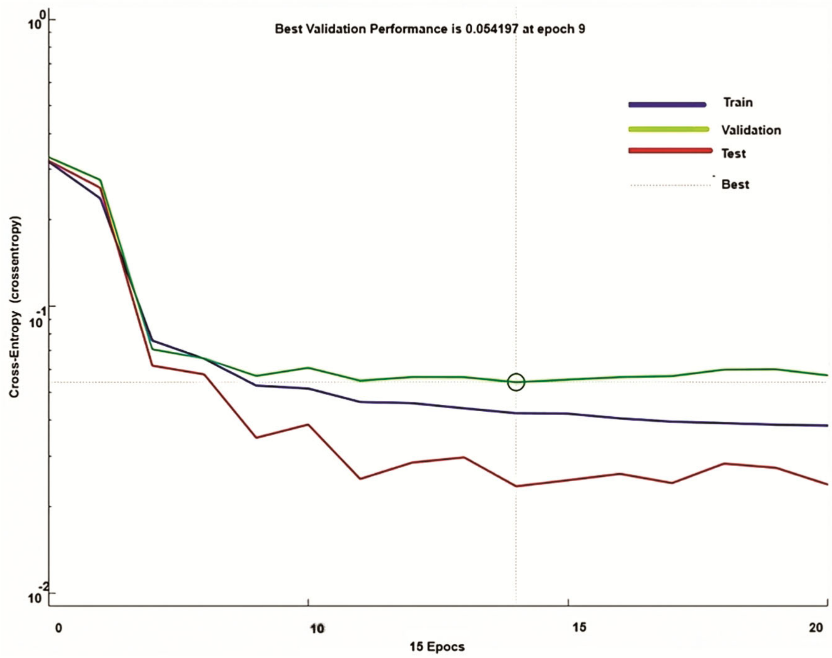

Fig. 7 — Training error and validation performance curve of corrosion prediction

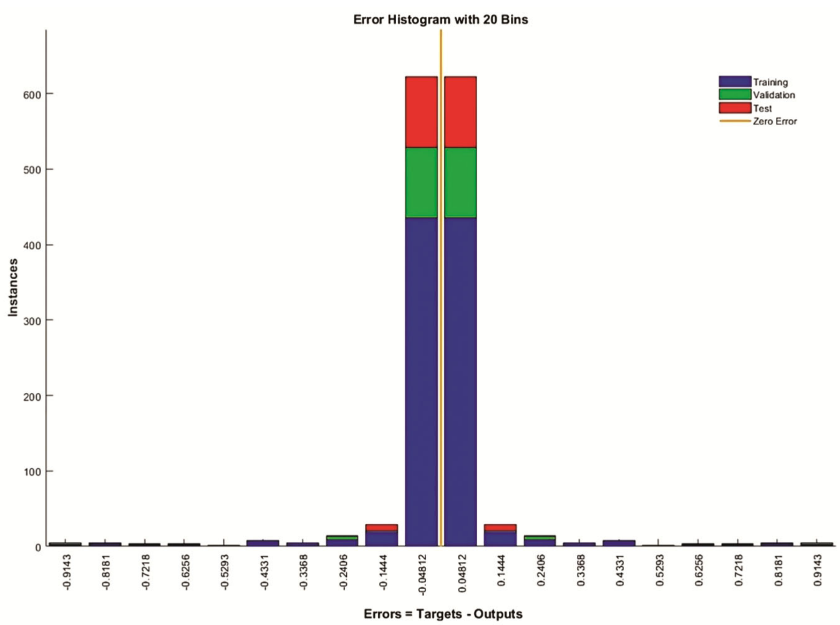

Fig. 8 — Distribution of error during the classification in histogram

of epochs, which further validates the currently proposed tool to be a viable technique for corrosion evaluation. Fig. 9(b) represents the validation failures in validation state against the number of repetitions denoted as epochs. It is observed that, the validation checks are failed after the 6th epoch. The 6th epoch is taken as the best epoch of validation; beyond this, further validations are not required. Hence the weights and parameters corresponding to the 6th epoch is treated as final model weight for the test state. In a similar work done by Ude et al. [31], performance of engine and emission for African pear seed oil was evaluated through Artificial Neural Network Analysis, and the best validation was found at epoch 27.

From Fig. 10, it is evidenced that all the three states namely training, validation, testing and the combined

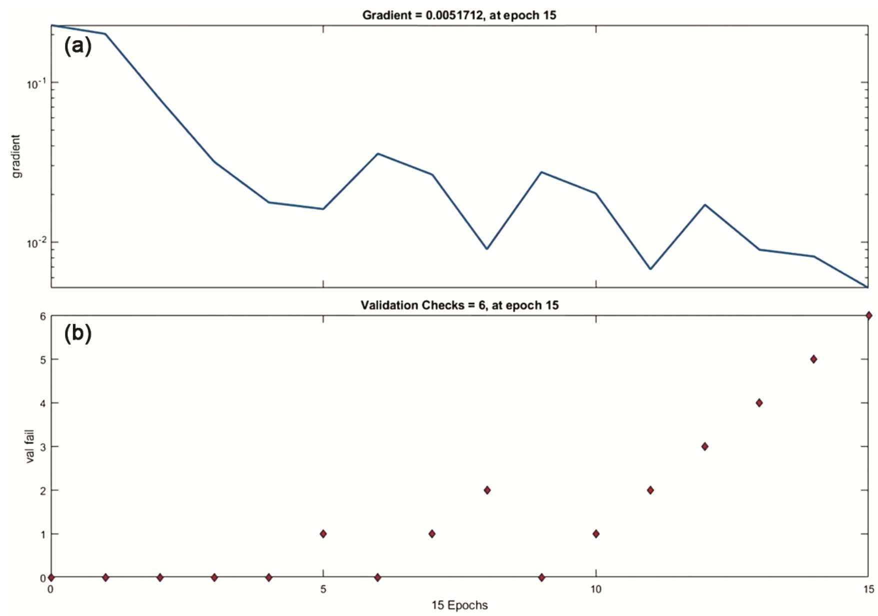

Fig. 9 — (a) Variation in Gradient coefficient vs Number of Epoch and (b) Training State Validation State vs epoch

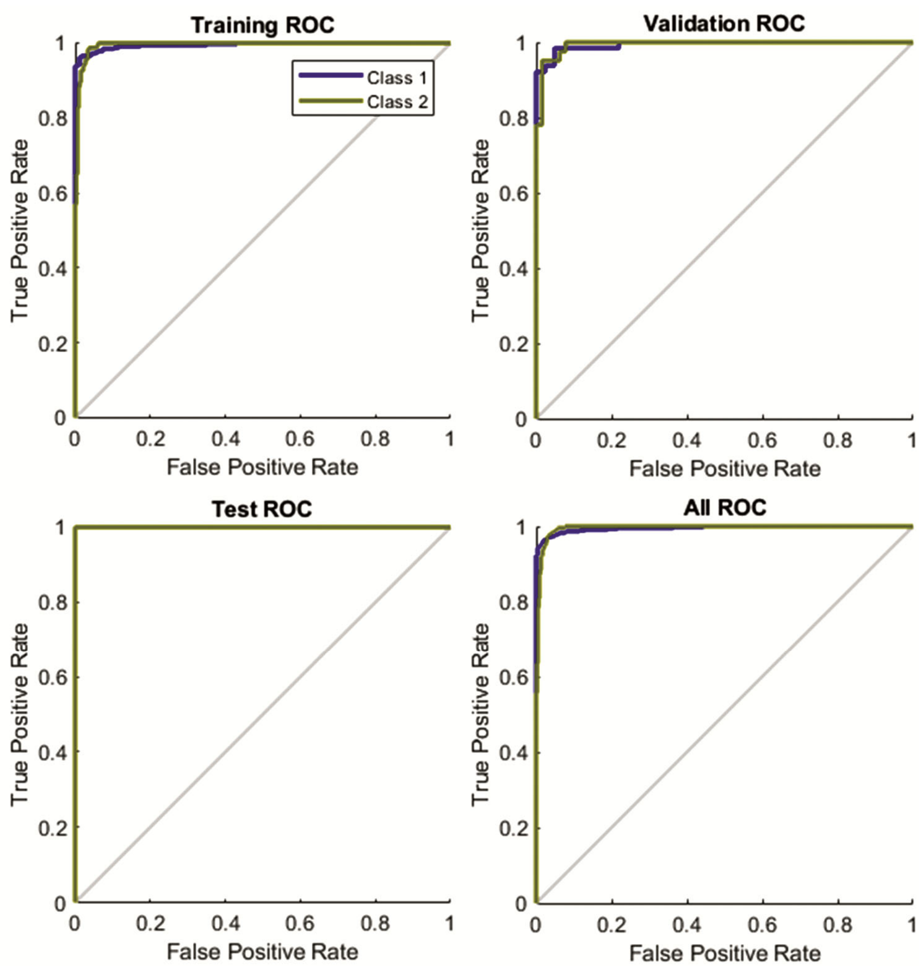

Fig. 10 — Receiver operating characteristics

performance of the neural network based prediction of corrosion inhibition study is well above the random classifier line, and almost nearer to the perfect classifier point value of 0.97. These results are in coherence with the performance parameters evaluated by Ude et al. for the performance and emission evaluation[33]. A good separation can be interpreted from the ROC of training, validation, testing and combined states. When the area under curve is 0.97, it clearly indicates the best performance of the predictor

Neural network based corrosion prediction

The features were fed as input for the neural network based predictor for pre training testing and validation. The outcomes of the neural network based predictors are depicted in the following figures. Confusion plot is one of key performance indicator plot of neural network predictors. In the proposed study the training, test, validation and combined confusion plots were obtained through the neural network tool box in the MATLAB. The training, validation and testing performance were measured in terms of sensitivity, specificity and accuracy, that are calculated using the formulae given below[34]

True Positive Rate (Sensitivity) = $\frac{TPs}{TPs+FNs}$

False Positive Rate (Specificity) = $\frac{FPs}{TNs+FPs}$

Accuracy = $\frac{TPs+TNs}{TPs+FPs+TNs+FNs}$

where TPs are number of true positive decisions taken by classifier, TNs are number of true negative decisions taken by classifier, FNs are number of false negative decisions taken by classifier and FPs are number of false positive decisions taken by classifier.

Fig. 11 represents the confusion plot of training, validation, testing and overall states of corrosion prediction performance of artificial intelligence based

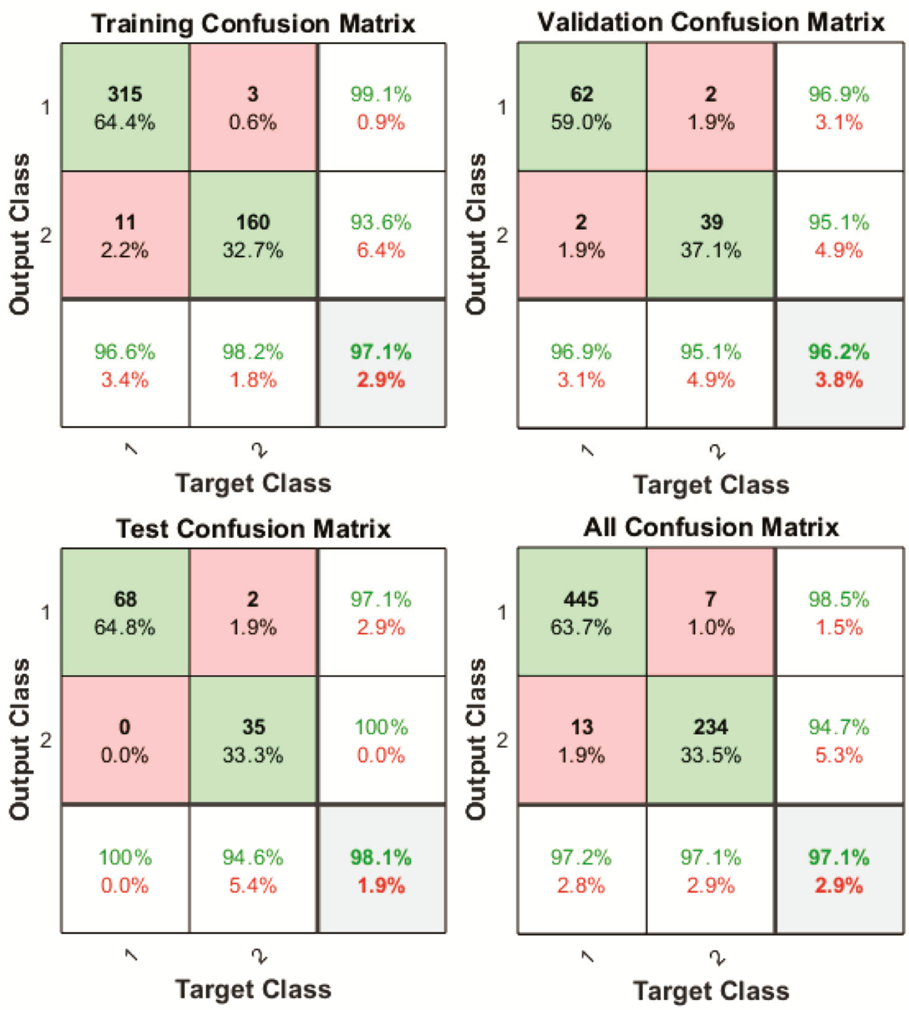

Fig. 11 — Confusion plot of training, validation and testing

system. The classification accuracy for corrosion of metals in biodiesel medium, for training, validation, testing and overall states were 97.1%, 96.2%, 98.1% and 97.1%, respectively. This is in consistent with the parameters obtained by Ude et al. in a performance analysis of engines with biodiesel through a MIMO based neural network classification study, in which the accuracy obtained were around 99% for all these states[33]. Similarly in another work done with artificial intelligence-based pipeline inspection of concrete structures by Chow et al.[35], an accuracy of 96.7% was obtained for all these states of classification by neural networks.

A CNN and RSM based performance and emission prediction of a compression ignition engine fuelled with biodiesel-diesel blends were done by Aydin et al.[36]. In this work, the regression parameters obtained were ranged between 0.86 and 0.98 for the three states namely testing, training and validation. These results are in coherence with the accuracy parameters for the three states obtained in this current work.

The testing phase of the neural network has a greater accuracy among the training and validation phases. The neural network based classification proposed in this current work achieved a high overall accuracy rate of 97.1%, which is substantially higher than the reported accuracy of 95.6% obtained by Wang et al.[35] and Chow et al.[37] in their works.

The specificity of the neural network system for classification of the corrosion behaviour is 93.6% in training state, 95.1% in validation state and 100% in testing state. The overall specificity of all the states is 94.7%.

The sensitivity of the system during the training phase is 99%, 96.9% in validation phase and 97.1% during the testing phase. It is interesting to note that the neural network system provided a high overall sensitivity of 98.5%, which seemingly makes this as a reliable technique for classification of corrosion behaviour of copper in biodiesel with and without inhibitors or general corrosion deterioration. The sensitivity obtained in this current work is in coherence with the sensitivity of 98.64% obtained by Zhang et al.[38], in a deep neural based metallic corrosion prediction work.

The neural network system used here helps the technologists to evaluate the corrosion-impact on the metal in multiple environments without performing any experimental work, but just by recording the surface images of the corroded surface. It also saves human resource, cost, time and energy.

The proposed corrosion inhibitor significantly arrests the corrosion of copper in biodiesel, therefore can be used as prospective inhibitor. Novel AI tool proposed here, potentially can serve for copper corrosion prediction not only in biodiesel, but in multiple environments as it has an excellent overall sensitivity.

Conclusion

Value added biodiesel is made by blending them with green coffee bean inhibitor in ppm levels. Weight loss evaluations clearly showed that the corrosion was significantly inhibited by the addition of green coffee bean inhibitor. As the temperature increases, weight loss was also found to increase, but green coffee bean extract significantly reduced the corrosion reaction and weight loss. Microscopic image obtained clearly demonstrated the protective nature of the green coffee bean inhibitor on the copper metal surface in biodiesel medium by preventing them from damages such as cracks and pits. The proposed novel neural network system was found to be reliable and robust method for surface corrosion behaviour prediction. The proposed model can be used for prediction of corrosion behaviour of copper in biodiesel and other environments consisting of various complex systems that has variable influential factors. Neural network system proposed here saves human resource, cost, time and material-resources in metallic corrosion evaluation not just in biodiesel but in multiple corrosive environments. The work can be extended to various metals and alloys with different type of fuels with a larger dataset. Further, a number of hybrid deep learning algorithms can be evaluated for the classification and prediction of corrosion behaviour in various metals and alloys with various blends of biodiesel with inhibitors.

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