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Optimization of factors influencing corrosion inhibition performance of N- Maleyl chitosan/ poly vinyl alcohol blend for mild steel in acid medium – Taguchi method

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A water soluble chitosan derivative [N- Maleyl chitosan] was prepared and blended with PVA for evaluation of its corrosion inhibition performance towards mild steel in 1 M HCl. The parameters employed to realize the corrosion inhibition viz., N- Maleyl chitosan, PVA, exposure time and temperature were optimized using Taguchi method, in order to achieve a better inhibition efficiency. Four influencing factors and their three levels were constructed in sequence to achieve a L9orthogonal array. The experiments were carried out to analyze the effect of variables on corrosion inhibition and optimal conditions. The results obtained from orthogonal array experiments were further processed with Qualitek-4 software at "bigger is better" as quality character. Taguchi statistical experiments resulted in evaluating the effects of the factors individually and in combination. The optimized results were validated through electrochemical techniques.

Introduction

Mild steel a common form of steel widely used as a chief construction material in most of the industries because of its low cost and excellent mechanical strength. Mild steel pipes are used in petroleum industries, storage tanks, reaction vessels and chemical batteries [1]. Acids are widely used for various industrial processes like acid cleaning, ore production, pickling, descaling and other applications. High corrosive nature of acids may cause severe damage to the system components in the industries. Since corrosion of metals remains a serious challenge to world economy, different methods are used to reduce the destruction of metals in aggressive media. The use of corrosion inhibitors is one among the different methods and is most economical and practical method.

Polymers [2-6] have been reported as effective corrosion inhibitors because of their inherent stability and cost effectiveness. Basically polymers are known for their strong adsorption on the metal surface with their multiple adsorption sites. From the literature survey, polymeric mixtures and polymeric blends were reported to produce effective corrosion inhibition efficiency. The use of polymer blends as acid inhibitors offer multiple services (including synergistic effect) for effective corrosion inhibition. Some of the studies highlighted the effect of polymer- polymer mixtures or blends on corrosion inhibition of metals [7,8]. Blending of polymers considered to be an attractive approach and provides an individual characteristic of homopolymer to achieve a better corrosion inhibition.

The aim of the study is to investigate a inhibitive action of Poly vinyl alcohol and N-maleyl chitosan blends for mild steel corrosion in 1M HCl. Polyvinyl alcohol (PVA) is a water-soluble, biodegradable polymer, used in many applications because of its excellent chemical resistance and physical properties. Chitosan is the N-deacetylated product of chitin rich in hydroxyl and amino groups found to exhibit good inhibiting potential. The application of chitosan is limited because of its insolubility in neutral and high pH region. To improve the solubility of chitosan, the attempts are made to synthesis new derivatives [9]. Chitosan and its derivatives gained useful attention among many researchers because of its properties such as immunological activity, wound healing, biocompatibility, low toxicity, and biodegradability [10–12]. These compounds also used widely in many applications such as medicine, cosmetics, textile, paper and food industries [13]. Only few studies have been reported the inhibitive action of the chitosan derivatives [14-17]. So the blend of PVA /NMC was drawn in to picture for the inhibition process of mild steel.

Taguchi method

Corrosion inhibition of mild steel using the polymer blends PVA/ N-Maleyl chitosan in 1M HCl depends on the parameters such as concentration of PVA, concentration of N-Maleyl chitosan, exposure time and temperature. Practically optimization of all these parameters to achieve a best combination for the better performance and to study the interrelationship between the parameters is a time and labor consuming process. To optimize the affecting variables on inhibition efficiency of mild steel, Taguchi method can be used [18]. Taguchi technique is a powerful tool in optimization of the processes and in quality control. It provides a simple, efficient and systematic approach to find the optimum condition of the process which provides a best inhibition performance. An efficient way to study the effect of parameters and optimal conditions of parameters is by designing the orthogonal array of experiments [19]. Taguchi method determines the parameter optimization using signal to noise [S/N] ratio. Taguchi method is the combination of mathematical and statistical techniques used in many studies [20-23].

In this work, Taguchi L_9 orthogonal array is employed to analyze and optimize the inhibition efficiency of mild steel in 1M HCl using PVA/N-Maleyl chitosan blend. This process of optimization enables to study the influence of individual factors, relationship between variables and the performance at the optimum levels based on the concept of S/N ratio.

Materials and methods

Chitosan (75% deacetylated) was purchased from Sigma aldrich, PVA of molecular weight 15000 from Rankem, maleic anhydride (loba chem.), acetone (finar), acetic acid, HCl, were used for the study.

Preparation of N-Maleyl chitosan

N-Maleyl chitosan was prepared according to the procedure given elsewhere [24]. 2 g chitosan was dissolved in 100 mL of 2% acetic acid, and 2.44 g maleic anhydride was dissolved in 10 mL acetone solution added dropwise into solution with stirring. The mixture was stirred continuously for 18 h at room temperature. The product was precipitated in acetone, filtered, washed with acetone again to remove unreacted compounds, and then dried to obtain the product of NMC.

Design of experiments – Taguchi method

The steps involved in the Taguchi method of optimization are as follows:

Selection of factors and levels
Selection of orthogonal array
Conducting experiments
Documentation of experimental results
Analysis of results
Prediction of optimum performance
Confirmation of results

Experimental design: selection of orthogonal arrays and assignment of factors

A standard Taguchi L9 orthogonal array table was prepared for this study by choosing four parameters that affects the inhibition efficiency of mild steel. Factors and levels selected for this experiment are given in Table 1.

The statistical experimental design can determine the effect of the four influencing parameters on the corrosion inhibition process and also the optimal conditions. In this method, the influencing parameters are categorized into signal factor, noise factor and control factor. Signal factor is a variable which initiates the process and its action sustained throughout. As HCl is responsible for the corrosion process, HCl concentration is selected as the signal factor. The noise factor is a variable that known to affect the response but it is difficult to control. Fe^{2+} concentration and surface roughness are assigned as noise factors.

Table 1. Experimental parameters and its different levels

Factors	Level 1	Level 2	Level 3
PVA	100 ppm	300 ppm	500 ppm
N- Maleyl Chitosan	100 ppm	300 ppm	500 ppm
Exposure Time [h]	½	1	3
Temperature [K]	303	313	323

The control factor is a variable that is expected to give impact on the response [21]. The exposure time, temperature, PVA concentration and N-Maleyl chitosan concentration are selected as control factors. The four factors at three different levels were considered for L9 orthogonal array which resulted in nine set of experiments [Table 2].

Conducting experiments - Gravimetric measurements

Mild steel of composition of Mn -0.196, C-0.106, P-0.027, Cr-0.022, S-0.016, Ni-0.012, Si-0.006, Mo-0.003 and remaining Fe was cut into rectangular pieces of area 1x5 cm^2. The polished specimens were thoroughly dried at room temperature. Weight loss measurements were performed thrice with mild steel specimens for the nine experiments following the ASTM G31 standard procedure [25]. Inhibition efficiencies are calculated from the weight loss values using the relation,

$$IE = \frac{Wo-Wi}{Wo} X\ 100 \quad \text{---------}\ 1$$

where, W_o and W_i are the weight loss of the specimen in the absence and presence of inhibitor.

Electrochemical studies for the optimized conditions

The electrochemical experiments were carried out using frequency response analyser (Solartron 1280B) The conventional three- electrode assembly with saturated calomel electrode (SCE) as reference electrode, platinum foil as counter electrode and mild steel strips (1 cm^2 exposed area) as working electrode was used for polarization and impedance studies.

In potentiodynamic polarization studies, specimens were polarized over a range of from -0.1 V to -1 V at a scan rate of 2 mV/sec. The corrosion current densities (I_{corr}) were determined by extrapolating the linear portion of the corrosion potential (Ecorr) in the graph plotted between applied potential versus current. The IE_{Icorr} is calculated using the following equation 2.

$$IE(\%) = 100\ X \left[1 - \frac{corr}{{}^{\circ}corr}\right] \quad \text{---------}2$$

where, I_{corr} is corrosion current without inhibitor and I^{o}_{corr} is corrosion current with inhibitor.

Impedance measurements were carried out using AC signals of 10 mV amplitude and sweeping the frequency from 20 kHz to 0.1 Hz.

Table 2. L9 orthogonal array of experiments.

Experiments	Combination of Factors and Levels			
Trial 1	PVA [100 ppm]	NMC [100 ppm]	½ h	303 K
Trial 2	PVA [100 ppm]	NMC [300 ppm]	1 h	313 K
Trial 3	PVA [100 ppm]	NMC [500 ppm]	3 h	323 K
Trial 4	PVA [300 ppm]	NMC [100 ppm]	1 h	323 K
Trial 5	PVA [300 ppm]	NMC [300 ppm]	3 h	303 K
Trial 6	PVA [300 ppm]	NMC [500 ppm]	½ h	313 K
Trial 7	PVA [500 ppm]	NMC [100 ppm]	3 h	313 K
Trial 8	PVA [500 ppm]	NMC [300 ppm]	½ h	323 K
Trial 9	PVA [500 ppm]	NMC [500 ppm]	1 h	333 K

Analysis of results

For the analysis of the results and optimization of the factors, Qualitek-4 software (Nutek Inc., MI) was used. The obtained experimental results were processed in the Qualitek-4 software with "bigger is better" quality characteristics for the optimum condition of corrosion inhibition efficiency. The resulted data helped to identify the influence of individual factors on corrosion inhibition performance and also to estimate the optimum performance conditions. Taguchi method determines the optimum condition in terms of S/N ratio using bigger the better quality characteristic equation,

$$S/N_i = -10 \log \left[\frac{1}{N_i} \sum_{u=1}^{N_i} \frac{1}{y_u^2} \right]$$

Prediction of optimal conditions

ANOVA is a statistical technique used for determining the influence of factors from the Taguchi experimental results of corrosion inhibition process [22]. The optimum conditions for the process can be interpreted from the experimental results using ANOVA table. The table consists of sum of the square (SS), the degree of freedom (*D*), the variance (*V*), corrected sum of squares (SS′) and the percentage contribution of each parameter (*P*). From the percentage contribution values, the optimum conditions can be predicted and compared with experimental results.

Validation of results

Taguchi method of experiments was validated by conducting the corrosion inhibition experiments at optimized conditions. The individual performance of PVA and N-Maleyl chitosan were observed under optimized conditions. The optimum conditions were also validated through electrochemical studies.

Results and discussion

Determination of the optimum conditions

Weight loss corrosion experiments were conducted as per L9 orthogonal array, assigning various values of the levels to the process parameters. The individual experiments for each set of values were conducted on mild steel and their inhibition efficiency was calculated using the equation (1).

Table 3. Experimental results of Inhibition efficiency and their S/N ratios

Experiments	Inhibition of efficiency			S/N ratio for inhibition efficiency
	Trial 1	Trial 2	Trial 3	
1	51.16	69.18	56.97	35.23
2	80.63	83.18	80.7	38.22
3	77.04	77.1	75.85	37.69
4	74.19	73.18	72.17	37.28
5	82.61	82	83.72	38.35
6	87.41	85.81	86.39	38.74
7	87.11	85.9	83.72	38.64
8	81.8	83.24	80.42	38.25
9	86.84	85.94	87.52	38.76

The obtained inhibition efficiency values were converted into S/N ratio using Qualitek software with "bigger is better" as quality characteristic to determine the optimum conditions for better corrosion performance. The experimental results of inhibition efficiency and their corresponding S/N ratios were given in Table 3.

The relative influence of each factor can be determined by the difference between the maximum and minimum S/N ratio of the levels. The higher difference in value indicates that the factor exhibits stronger relative influence. The factors with maximum S/N ratio at a particular level considered to be an optimum performance conditions for corrosion inhibition. The mean S/N ratio and the significance of influencing factors (the difference between max and min values) were tabulated in Table 4. The table includes ranks based on the significance values. From the Table, it can be seen that the maximum influence is given by the concentration of PVA followed by the concentration of N-Maleyl chitosan and temperature while the minimum influence is by the exposure time. Significance of influencing factors indicated that concentration of PVA is contributing more towards inhibition efficiency as the difference value is high. The main effect plot of each factor influencing on the inhibition efficiency is represented in Figure 1.

Taguchi dynamic method accounts for interaction of each factor with all of the other factors to estimate the interaction between the factors which influences more for the corrosion inhibition process. Interaction between the factors can be determined by non-parallelism effects of the factors. If the interaction plot consists of non-parallel lines, interaction exists between the factors and if the lines cross, strong interaction occurs between the factors [22]. Figure 2 shows the interaction plot which consists of non-parallel lines, that infers interaction exists between all the factors. From Taguchi results, the optimal factors for corrosion inhibition process is obtained as 500ppm of concentration of PVA (level 3), 500 ppm of concentration of NMC (level 3), 3 hours of exposure time (level 3) and temperature of 313 K (level 2).

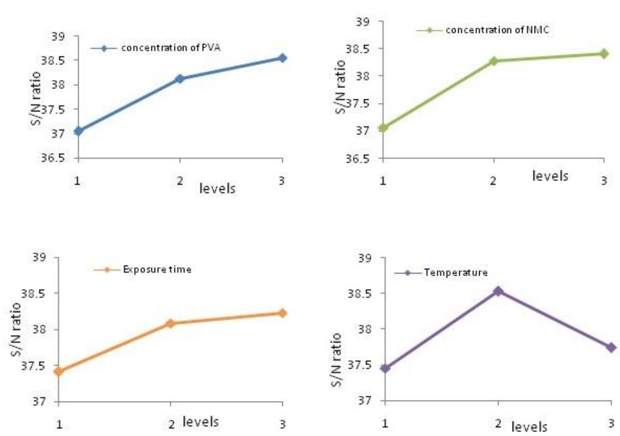

Fig.1. Effects of factors at their different levels.

Analysis of variance (ANOVA)

The Percentage contribution of individual factor to the inhibition efficiency can be well determined by performing analysis of variance (ANOVA). The results of ANOVA are summarized in Table: 5. The data in the table provides the percentage contribution of the four factors viz., Concentration of PVA, Concentration of NMC, Exposure time, Temperature as 36.233, 33.278, 11.582, 18.901 respectively. It is evidently concluded that, among the considered four factors, concentration of PVA has the major influence and considered to be the most significant factor for the corrosion inhibition process. By ranking the percentage contribution, concentration of PVA attains first in corrosion inhibition followed by concentration of NMC and temperature, finally exposure time acquires least position in the sequence. It is to be mentioned that in the ANOVA analysis, there will not be any missing of significant factor in the experimental design if the percentage error contribution is less than 15 %. On the other hand, if the percentage error contribution exceeds 50%, factors should be verified and the experiment should be re-constructed [26]. From the table, 0% percent error contribution (P_e) value clearly suggested that there is no missing of significant factor in the experimental design. Similar ANOVA results were discussed by Hosseini and Bodaghi for corrosion behavior of electroless Ni-P-TiO2 nanocomposite coatings [22]. Since the P_e is 0% the F- value for the all the four factors is infinity. F-ratio is an important tool traditionally used to determine the significance of the factors. High F-values suggest the greater significance of the factors. The F-values for the corrosion inhibition process was calculated and found to be infinity for all the factors. The infinity value of the F-ratio also confirmed that all the four factors are significant in the corrosion mitigation process [27].

Table 4. Significance of influencing factors for inhibition efficiency.

Influencing Factors	Mean S/N Ratio Level 1	Level 2	Level 3	Significance of factors Max-Min	Rank
Concentration of PVA	37.048	38.128	38.554*	1.506	1
Concentration of N-Maleyl chitosan	37.054	38.277	38.4*	1.345	2
Exposure time	37.41	38.091	38.23*	0.82	4
Temperature	37.452	38.535*	37.743	1.082	3

* more significant level of factors

Table 5. Results of ANOVA for corrosion inhibition efficiencies.

Factors	Degrees of freedom(f)	Sum of Squares (S)	Variance (V)	F- ratio (F)	% Contribution (P)	Rank
Concentration of PVA	2	3.617	1.808	∞	36.233	1
Concentration of NMC	2	3.322	1.661	∞	33.278	2
Exposure time	2	1.156	0.578	∞	11.582	4
Temperature	2	1.887	0.943	∞	18.901	3
Error	0					
Total	8	9.984			100%	

Optimum conditions

By ANOVA statistical method, optimum conditions for better inhibition performance in terms of contributions was evaluated using Qualitek software and the results are given in Table 6.

Table 6. ANOVA results of Optimum conditions for corrosion inhibition process

Factors	Level	Level description	Contribution
Concentration of PVA	3	500 ppm	0.644
Concentration of NMC	3	500 ppm	0.489
Exposure time	3	3 h	0.319
Temperature	2	313 K	0.625
Total contributions from all the factors			2.076
Average performance			37.91
Expected performance at their optimal conditions			39.987

It can be seen from the table, all the four factors have the significant effect still concentration of PVA and temperature contributes more significance than the other factors. Thus optimum conditions evaluated from ANOVA method closely match with the Taguchi results.

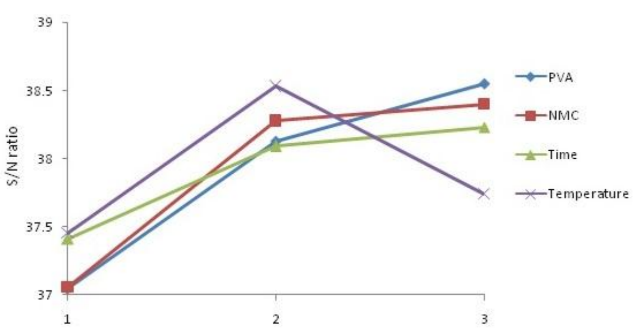

Fig. 2. Interaction plot of all factors at their levels.

Validation of the experiment and confirming the results

After identifying the best level of factors for inhibition process, validation and confirmation is the final step in verifying the results of proposed method. To validate the experimental results weight loss experiment was carried out for mild steel specimens at optimized conditions (Table 6). The validation experiment was also performed at the same experimental conditions for the individual polymer solutions. The inhibition efficiencies observed for the validation was presented in Table 7. The inhibition efficiency of the polymer blend was found to be reliable with Taguchi experimental method. Thus the mixture of PVA and NMC was optimized as a better corrosion inhibitor for mild steel in 1M HCl.

Effect of factors on corrosion inhibition mechanism

Effect of concentration of PVA and NMC

From the experimental results, it could be seen that the corrosion of mild steel was inhibited by the blend solutions of PVA and N-Maleyl chitosan. The corrosion inhibition of mild steel was studied by varying the ratio between the blend solutions. Since the concentration of PVA has the maximum S/N ratio value, it plays a vital role in the inhibition efficiency of mild steel. Also the experimental condition with high concentration of PVA yields maximum inhibition efficiency. The effect of inhibition may be attributed to the strong hydroxyl bridge formation between the polymer and metal surface. By ranking, the concentration of NMC has second maximum S/N ratio contributes in the corrosion inhibition mechanism. The significant inhibition efficiency was achieved on blending PVA and N-Maleyl chitosan in equal amount of 500ppm. The increase in the inhibitor concentration increases the inhibition efficiency because of the adsorption of the polymeric species through their active centre [17].

Effect of temperature and time

For all the concentration of inhibitor, inhibition efficiency increases with increase in exposure time. This may be attributed to the stability and persistence of the polymeric film formed on the metal [21]. Inhibition efficiency increases with temperature and decrease at 323 K resulted in desorption of adsorbed molecules.

Thus the corrosion behavior of mild steel in PVA/NMC blend solution (500 ppm of concentration of PVA, 500 ppm of concentration of NMC) along with 1M HCl studied for 3 hours of immersion at 323 K. The increase in the inhibition efficiency could be attributed to the adsorption of stable polymeric film formed on the metal surface.

Table 7. Results of validation experiment

Inhibitor at 313 K for 3 h	Inhibition efficiency (%)
500 ppm of PVA	63.67
500 ppm of NMC	66.16
500 ppm of PVA + 500 ppm of NMC	88.12

Electrochemical measurements for optimum conditions

Potentiodynamic polarization studies

Figure 3 shows the anodic and cathodic polarization curves for mild steel in 1 M HCl in the presence and absence of the inhibitor system at the optimum conditions obtained from the Taguchi method. The electrochemical parameters such as corrosion potential (Ecorr), Corrosion current (Icorr), cathodic Tafel slope (bc), anodic Tafel slope (ba), inhibiton efficiency (IE) derived from the polarization curves are summarized in Table. The inhibition efficiency of the PVA/NMC blend compared to the individual polymer solution is more indicating the effective inhibition on corrosion of mild steel. The difference in the E_{corr} values between inhibited and uninhibited solution is less than 12mV indicating that the inhibitor system belongs to mixed type. The decrease in the current density value also evidences the inhibition of mild steel corrosion by PVA/NMC blend. The inhibitor system does not alter the mechanism of corrosion process which is represented by the values of Tafel slopes. The increase in the inhibition efficiency in the case of PVA/NMC blend is attributed to the adsorption of the inhibitor on the metal surface. The film forming nature of the NMC may be responsible for the higher inhibition efficiency.

AC impedance studies

The corrosion behavior of the mild steel in presence of the PVA/NMC blend was studied using impedance spectroscopy. Figure represents the Nyquist plot and impedance responses of mild steel in 1M HCl and inhibitor system were given in Table. Double layer capacitance is calculated using the following formula: $CPE_{dl}=[\ Y_o.Rct^{1-n}]^{1/n}$. From the table, it can be seen that Rct value is high and Cdl value is low for the blend system. The higher value of Rct is responsible for the slower corroding system. The decrease in the Cdl value can be attributed to the enhanced surface coverage of the inhibitor on the metal. Thus the inhibitor system found to be effective inhibitor for mild steel corrosion in 1M HCl.

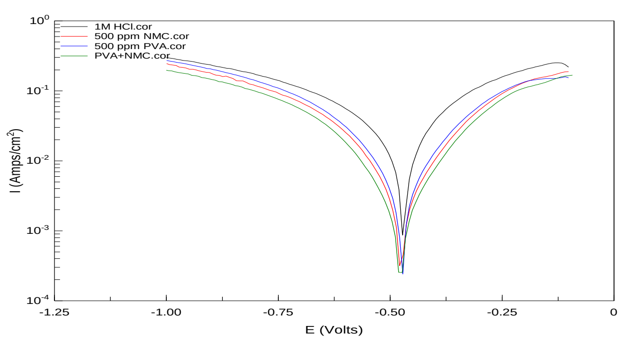

Fig. 3. Polarisation curve PVA/NMC blend system in 1M HCl.

Conclusion

Taguchi orthogonal array of experiments is employed to optimize the factors influencing the corrosion inhibition process. Taguchi method of experiment explains the influence of factors and also interaction between the factors. The experimental results were evaluated with Qualitek software for optimal conditions. Results obtained from Taguchi method are comparable with ANOVA results. The optimal level of factors was found to be 500 ppm of concentration of PVA, 500 ppm of concentration of NMC, 3 hours of exposure time at a temperature of 323 K. The experiment at the optimized condition inhibits mild steel corrosion in 1M HCl with 88% inhibiton efficiency. Electrochemical studies conducted for the optimized condition also revealed that the PVA/NMC blend acts as a mixed type inhibitor for the corrosion of mild steel in 1M HCl.

Table 8. Potentiodynamic polarisation parameters for PVA/NMC blend system in 1M HCl.

Inhibitor system	b_a (mV/dec)	b_c (mV/dec)	I_{corr} (μA/cm^2)	E_{corr} (V/dec)	IE (%)
Blank	140.79	110.62	0.0060998	-0.46248	
500ppm of PVA	138.82	125.58	0.0035384	-0.47017	42
500ppm of NMC	109.3	99.301	0.001638	-0.46899	73.14
500ppm PVA+500ppm NMC	111.11	99.424	0.0012348	-0.47126	80.15

Table 9. Impedance parameters for PVA/NMC blend system in 1M HCl.

Inhibitor system	R_s (Ohm /cm^2)	R_{ct} (Ohm/cm^2)	N	Y_o 10^{-3} ohm^{-1}cm^{-2}	Cdl
Blank	0.46247	6.84	0.71704	0.0010776	1.0776
500ppm of PVA	0.56214	16.19	0.83103	0.00028189	0.2818
500ppm of NMC	0.53704	27.2	0.79835	0.00036705	0.36705
500ppm PVA+500ppm NMC	0.56638	45.91	0.79431	0.00036532	0.3653

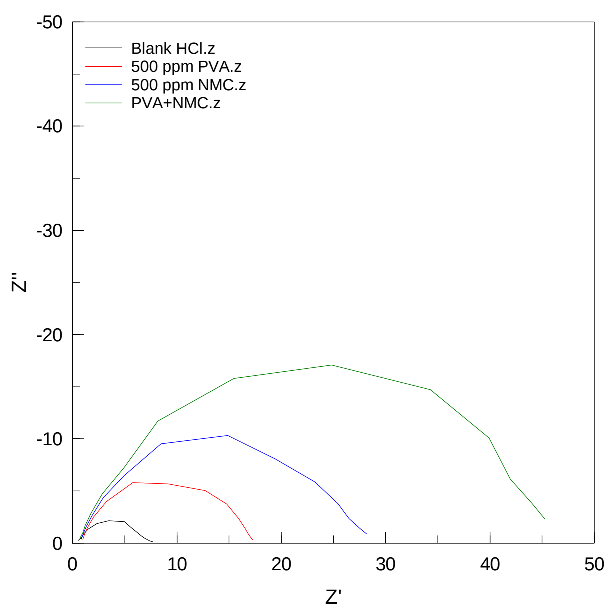

Fig. 4. Nyquist plot for PVA/NMC blend system in 1M HCl.

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