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Water Treatment: Evaluation of Maleic Acid-Acrylamide Copolymer Inhibitor Efficiency on Calcite Scale by Response Surface Methodology

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ABSTRACT

Mineral scales of calcite are common in the oil field and pose a serious integrity problem in the wellbore, flow lines, and equipment. It is also a challenge faced by industries such as refineries and power plants. Scale deposition is a complex process depending on various factors such as concentration of scaling species, temperature, pH, and flow rates. Deterministic models are used to predict the scale formation from the level of supersaturation of the scaling species in the water at the operating conditions. However, due to the complexity of the interaction of variables affecting the scaling and inhibition by chemicals, it is suitable to be represented by statistical models. This work focused on applying statistical analysis techniques such as response surface methodology to understand the effect of different operating parameters on the inhibition efficiency of maleic acid-acrylamide copolymer on $CaCO_3$ scales. The copolymer was synthesized, and its inhibition efficiency on the calcite scale was tested using static jar tests at different pH, temperature, and inhibitor concentrations. The effect of the critical parameters on the inhibition efficiency was analyzed using the statistical technique of Response Surface Methodology (RSM). The design of experiments (DoE) was created using a Box–Behnken design with three levels for each factor. The linear and the quadratic effects of the factors were studied and the interaction effects were analyzed using analyses of variance (ANOVA) and RSM. A desirability function was used to optimize the performance for the combination of the variables. The analysis showed that the linear effect of the parameters had the highest impact on the inhibition efficiency. Significant interaction effects were also identified between the operating variables. A transfer function was used to model the experimental data of inhibitor performance.

INTRODUCTION

Formation of mineral scales in flowlines and equipment is a major challenge faced by industries such as power plants and oil and gas production systems. Carbonates and sulfates of calcium typically deposit as scale in the wells, flowlines, and surface facilities, leading to restricted flow, increased pressure drops, and blockage in the oil field (Olajire 2015, Li et al. 2017). It can also lead to under-deposit corrosion and loss of metal. Scaling is typically controlled by a continuous dosage of scale inhibitors, which hinder the formation of deposits (Fink 2012). Scale formation depends on various parameters such as the water quality, concentration of the scaling ions, suspended solids, pH, temperature, flow rate, etc. Deterministic models have been developed to predict the formation and quantitative severity of scales as a function of the operating parameters (Kan & Tomson 2012, Mackay & Sorbie 1999). These models calculate the extent and amount of scaling from the level of supersaturation and the solubility of the scaling species in the water at the operating conditions. Some of the models also include the mitigating effect of common scale inhibitors. Scaling and the mitigation through chemical action are controlled by the complex interaction between the critical parameters and hence, it is suitable to be modeled using statistical methods. This paper describes the modeling and optimization of the efficiency of a threshold scale inhibitor on a calcium carbonate scale using response surface methodology (RSM).

RSM is a statistical method for analyzing and modeling a process where the outcome depends on several parameters and their interactions. RSM is specifically useful in modeling systems where the effect of the interaction of the independent variables is complex and a deterministic model cannot be developed satisfactorily. RMS is used in the design and development of new products/processes and also in the improvement of existing products and processes. It can be used to identify the critical parameters that affect the process.

The model can also be used to predict what-if scenarios of the process variables (Myers & Montgomery 1995, Martí-Calatayud et al. 2010). The design of experiments and RSM has been used widely in process and manufacturing industries and research laboratories to understand the effect of the operating parameters and optimize the process. It could be used to model the efficiency of chemicals such as corrosion inhibitors (Goh et al. 2008). RSM is applied in the oil industries in areas such as reservoir modeling and production forecasts (Manceau et al. 2002), uncertainty quantification on reserves estimation (Busby & Chugunova 2015), and estimating the wax deposition in the pipeline (Adeyanju & Oyekunle 2015). The use of statistical modeling to predict and optimize scale formation is very limited.

In this paper, the application of RSM for investigating the effect of operating conditions such as pH, temperature, and polymer concentration on scale inhibition efficiency is explained. Low molecular weight copolymers of maleic acid–acrylamide have shown good inhibition of $CaCO_3$ and $CaSO_4$ scales (Senthilmurugan & Ghosh 2009, Senthilmurugan et al. 2010, Senthilmurugan et al. 2019). In this paper, RSM methodology is used to design a new set of experiments for $CaCO_3$ inhibition and analyze the results.

MATERIALS AND METHODS

Experimental Methods

Maleic acid (HOOCHC=CHCOOH) and acrylamide (CH_2=CHCONH$_2$) were copolymerized in an aqueous medium through free radical polymerization using an initiator in an inert atmosphere. The reaction product was concentrated using vacuum distillation. The scale inhibition efficiency of the synthesized copolymer was tested using static jar tests according to the NACE standard test method TM0374-2016-SG (NACE 2016). Tests were conducted at various levels of pH of 7-8.5 and temperature of 50-70°C with an inhibitor concentration of 1-70 ppm. Cationic (Ca^{2+}) and anionic (CO_3^{2-}) solutions were prepared separately to maintain a stoichiometric proportion of 300 ppm each. CO_2 gas was bubbled through both the brines before mixing. The two solutions were mixed at a ratio of 1:1. Measured quantity of the scale inhibitor was added to the anionic solution before mixing with the cationic solution. The cells were placed in a shaker water bath at constant temperature for 12 h and then allowed to cool for 12 h. The residual calcium ion concentration in the solution was measured using the EDTA titration method before and after precipitation. The tests were performed for various combinations of pH, temperature, and inhibitor concentration, as shown in Table 1. The scale inhibition efficiency of the inhibitor (as a percentage) was calculated using the following equation:

$$\% \text{ Inhibition Efficiency} = 100 \times \frac{C_a - C_b}{C_c - C_b} \quad \ldots(1)$$

Where:

$C_a = Ca^{2+}$ ion concentration in the treated solution after precipitation

$C_b = Ca^{2+}$ concentration ion in the untreated solution after precipitation

$C_c = Ca^{2+}$ ion concentration in the untreated solution before precipitation

Experimental Design

The experimental conditions need to be selected based on the field conditions to obtain practical inhibition values. Formation of scale, especially of calcite, is a strong function of temperature and pH of the solution. However, it is affected by minor factors such as flow rates, surface metallurgy, the presence of other ions, and suspended impurities. The CO_2-HCO_3-CO_3 equilibrium in water depends on the pH, and the solubility of $CaCO_3$ depends on pH and temperature. Hence pH, temperature, and the inhibitor concentration are selected to be the primary parameters of the study of inhibition efficiency. Based on the experimental constraints and field experience, the following ranges were selected for the operating variables: pH 7-8.5, Temperature 50-70°C, and copolymer concentration 1-70 ppm, as shown in Table 1. This system had three independent variables (pH, temperature, and copolymer concentration, denoted by X) and one dependent response (inhibition efficiency, denoted by Y). The numeric ranges of the dependent variables are coded between -1 (minimum) and 1 (maximum). The design of experiments was formulated according to the Box-Behnken design (Montgomery 2013) with three discrete levels (-1, 0, 1) for each input parameter. The design is composed of 15 runs with three central points (runs 13, 14, and 15 at levels 0,0,0) and 19 error degrees of freedom. The experimental design matrix with the actual and coded values of pH, temperature,

Table 1: Range of parameters used in calcite scale inhibition efficiency measurement experiments.

Operating Parameters	Code	Levels		
		-1	0	1
pH	X_1	7	7.75	8.5
Temperature (°C)	X_2	50	60	70
Copolymer concentration (ppm)	X_3	1	35.5	70

concentration, and the measured inhibition efficiency are given in Table 2.

The correlation between the operating variables (X_1, X_2, X_3) and the response variable (Y = Inhibitor Efficiency) was fitted using a quadratic polynomial (Hyder et al. 2009) using the least squares method. This equation captures most of the linear and interaction effects of the parameters. The response model is given by:

$$Y_k = b_0 + \sum_{i=1}^{3} b_i X_i + \sum_{i=1}^{3} b_{ii} X_i^2 + \sum_{i=1}^{2} \sum_{j=1}^{3} b_{ij} X_i X_j + \varepsilon \quad \ldots(2)$$

Where X_i is the dimensionless coded input variable (X_1 = pH, X_2 = temperature, and X_3 = copolymer concentration), Y_k is the response or output variable (Y_1 for inhibition efficiency), b_0 the intercept or constant, b_i the linear coefficients, b_{ii} the quadratic coefficients, b_{ij} the interaction coefficients and ε the residual (noise or error) term. The coefficients of the equation are determined using the least squares method. The difference between the measured and the predicted value ($\varepsilon = y_i - \hat{y}$) is called the residual or error and denotes the deviation of the model predictions from the actual observations. In the least squares method, the coefficients (b) are selected so that the sum of squares of the residual is minimized. (Baş & Boyaci 2007). Analyses of variance method (ANOVA) were used to determine the linear and quadratic effects of the parameters and the effect of interaction on the response variable. The statistical analyses were performed using the software Design Expert 11 from Stat-Ease (Stat-Ease 2018).

RESULTS AND DISCUSSION

Calcite Inhibition Efficiency

The methods of characterization of the synthesized co-polymers and the typical characteristics of maleic acid-acrylamide copolymers are explained in the earlier paper (Senthilmurugan & Ghosh 2009). The average molecular weight of the copolymer was around 2000 g/mol (2000 Da). Lower molecular weight polymeric scale inhibitors are found to be more effective than higher ones. The optimum range of molecular weight of polymers in terms of scale inhibition efficiency is between Mw ~ 1000-4000, and the performance decreases at higher MW ranges (Farooqui et al. 2015).

The results of the static jar tests for calcite scale inhibition are given in Table 2. It is observed that the inhibition efficiency is very high at medium and high concentrations of the inhibitor. The ANOVA of the results is shown in Table 3. The Model F-value of 23.3 implies the model is significant. There is only a 0.02% chance that an F-value this large could occur due to noise. The quadratic polynomial model, as fitted, explains the R-squared of 95.89 of the variability in the percentage of inhibition efficiency. The residual analysis to establish if there is any significant correlation was carried out with the Durbin-Watson (DW) statistic tests, which showed a

Table 2: Design of experiments and results of $CaCO_3$ inhibition tests.

Runs	Actual Values			Coded Values			Inhibition efficiency [%] Y_1
	pH [-] x_1	Temperature [°C)] x_2	Copolymer concentration [ppm] x_3	pH X_1	Temperature X_2	Copolymer concentration X_3	
1	7	50	35.5	-1	-1	0	100
2	8.5	50	35.5	1	-1	0	100
3	7	70	35.5	-1	1	0	100
4	8.5	70	35.5	1	1	0	84
5	7	60	1	-1	0	-1	89
6	8.5	60	1	1	0	-1	66
7	7	60	70	-1	0	1	100
8	8.5	60	70	1	0	1	92
9	7.75	50	1	0	-1	-1	90
10	7.75	70	1	0	1	-1	76
11	7.75	50	70	0	-1	1	100
12	7.75	70	70	0	1	1	100
13	7.75	60	35.5	0	0	0	97
14	7.75	60	35.5	0	0	0	100
15	7.75	60	35.5	0	0	0	100

value greater than 5.0% (P=0.9599). There is no indication of serial autocorrelation in the residuals at the 5.0% significance level. The value of variance inflation factor (VIF) was used to prove if this design is orthogonal. The value of VIF was 1 for all the factors included in the analysis, and then the model is adequate for the observed data at a 95% confidence level. The model coefficients were selected based on the p-value. For a 95% confidence level, a factor significantly affects the result if the p-value is less than 0.05. Anova was performed step by step by eliminating the insignificant terms that have a p-value greater than 0.05 to improve the accuracy of the model. The final p-value of the model is 0.0002, which indicates that the fitted model is statistically significant. The model results and the coefficients of the transfer function are shown in Table 3.

Fig. 1 shows a Pareto chart of the linear, quadratic, and interaction coefficients to compare the effect of parameters on the inhibition efficiency. The linear coefficients of copolymer concentration (b_3) show the most significant effect ($p<0.0001$, F-ratio 64.22) to increase the inhibition efficiency. In addition, the interaction factors pH - temperature (b_{12}) and pH - copolymer concentration (b_{13}) also showed a significant effect ($p \leq 0.05$) to increase the inhibition efficiency. On the other hand, the quadratic effect of copolymer concentration (b_{33}), the linear coefficients of pH (b_1), and temperature (b_2) showed a significant effect ($p \leq 0.05$) to decrease the inhibition efficiency. The interaction factor b_{23}, which represents the product of temperature and concentration, and the quadratic term of temperature (b_{22}), did not produce a significant effect ($P \geq 0.05$) on the inhibition efficiency; therefore, these factors are not included in the regression model equation of the inhibition efficiency.

The resulting transfer function is given by:

$$Y_1 = 100 - 5.88X_1 - 4.13X_2 + 8.5X_3 - 4.0X_1X_2 + 3.75X_1X_3 - 4.0X_1^2 - 9.25X_3^2 \quad \ldots(3)$$

where Y_1 is the predicted calcium carbonate inhibition percentage. X_1, X_2 and X_3 are the dimensionless coded input

Table 3: ANOVA for a quadratic model for calcite scale inhibition efficiency of the copolymer.

Source	Sum of Squares	DF	Mean Square	F-value	p-value	Coefficient (Coded)
Model	1469.93	7	209.99	23.33	0.000242	
Intercept						100
A-pH	276.13	1	276.13	30.68	0.00087	-5.875
B-Temp	136.13	1	136.13	15.13	0.00598	-4.125
C-Conc	578	1	578	64.22	9.01E-05	8.5
AB	64	1	64	7.11	0.0321	-4.0
AC	56.25	1	56.25	6.25	0.0410	3.75
A^2	59.43	1	59.43	6.60	0.037	-4.0
C^2	317.80	1	317.80	35.31	0.000574	-9.25
Residual	63	7	9			
Lack of Fit	63	5	12.6			
Pure Error	0	2	0			
Cor Total	1532.93	14				

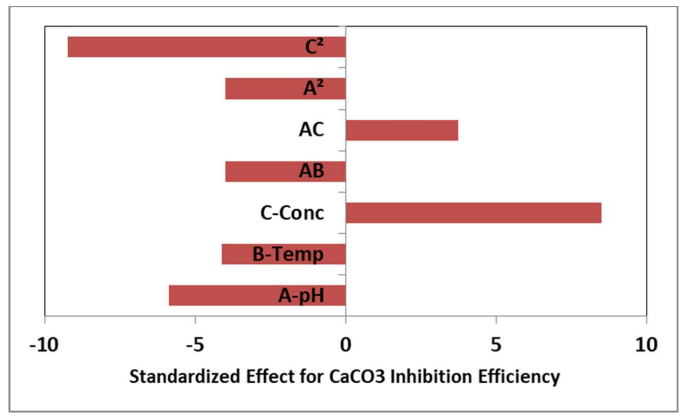

Fig. 1: Standardized Pareto chart for $CaCO_3$ inhibition efficiency.

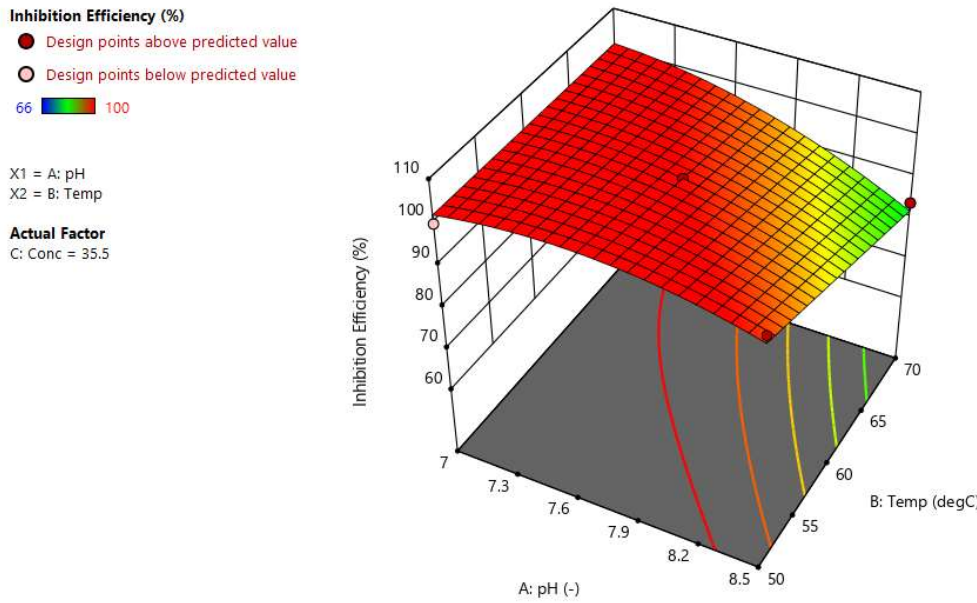

Fig. 2: 3D response surface with contour plot for the effect of pH and temperature on calcite scale inhibition efficiency.

parameters (X_1 = pH, X_2 = Temperature, X_3 = Inhibitor Concentration). A positive coefficient indicates that the parameter has a direct effect on the inhibition efficiency, and a negative coefficient indicates that the parameter has an inverse effect. The equation indicates that the inhibition efficiency increases with an increase in copolymer concentration. In contrast, it decreases with an increase in either pH or temperature, which is also observed from the data (Table 2). In linear terms, the effect of the various parameters on the efficiency is in the order of Concentration > pH > Temperature.

The response surface of the inhibition efficiency is plotted against two input parameters, while the third parameter is kept constant (at level 0 in Table 1). The 3D plot (Fig. 2) generated using equation (3) shows the effect of temperature and pH on the inhibition efficiency. Temperature has a linear effect in the range studied (50-70°C), where the inhibition efficiency decreases when the temperature increases (denoted by the absence of quadratic term of temperature in equation 3). The effect of pH is non-linear due to the presence of a quadratic term of pH in the equation. The data shows that at the maximum inhibitor concentration, the inhibition efficiency is always higher irrespective of the values of pH or temperature. This indicates that the calcite scale can be controlled by MA-AD copolymer even in adverse conditions by increasing the dosage.

Change in the pH affects the precipitation of calcium carbonate by altering the carbonate – bicarbonate - carbon dioxide balance in water. The solubility of calcium carbonate increases with a decrease in pH due to the conversion of carbonate ions into soluble bicarbonate ions and carbon dioxide gas. This aids in the increased efficiency of the scale inhibitor at lower pH. A similar effect is observed for the temperature, where an increase in temperature produces a decrease in the inhibition efficiency. At higher temperatures, the liquid layer adjacent to the hot surface acts as a super-saturation zone, leading to the precipitation of the compounds. However, these compounds may be soluble in the bulk liquid. This will lead to reduced inhibition efficiency at higher temperatures. Another factor affecting the efficiency at higher temperatures is the thermal stability of the inhibitors. The activity and stability of the inhibitors decrease with increasing temperature, leading to a reduction in efficiency.

Fig. 3 shows the combined effect of pH and copolymer concentration on the inhibition efficiency. The curvature in this 3D plot arises due to the quadratic effect of copolymer concentration. The inhibition efficiency decreases while the pH value increases from 7 to 8.5. The copolymer concentration effect is the opposite because the inhibition efficiency increases while the copolymer concentration is increasing.

The effect of temperature and copolymer concentration is shown in Fig. 4, where it is evident that an increase in the temperature produces a decrease in the inhibition efficiency in the range investigated (50-70°C). On the other hand, the copolymer concentration shows a quadratic dependence with an increase in the inhibition efficiency when the copolymer

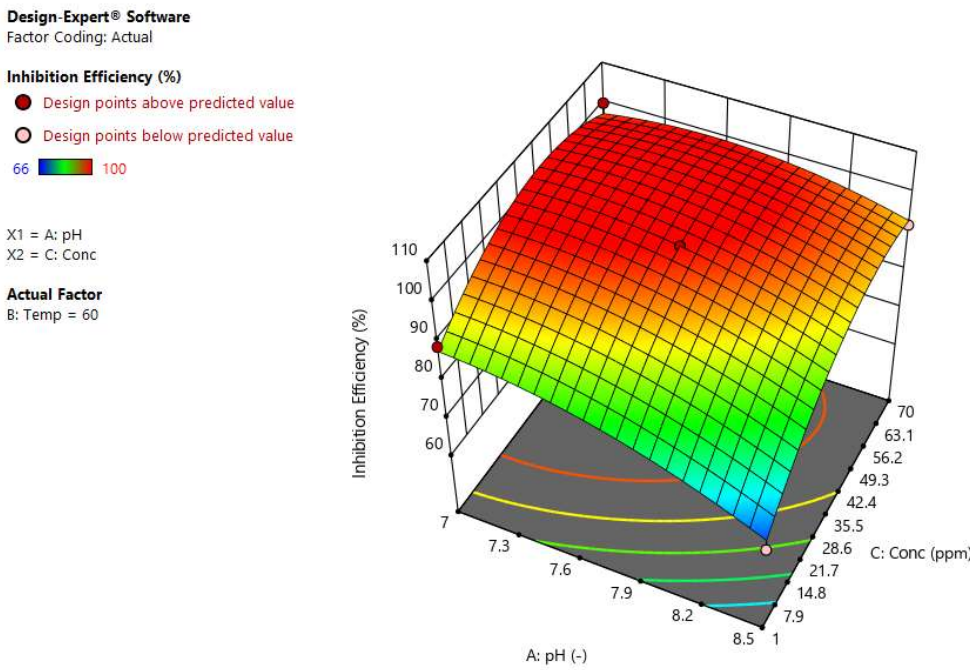

Fig. 3: 3D response surface with contour plot for the effect of pH and copolymer concentration on calcite scale inhibition efficiency.

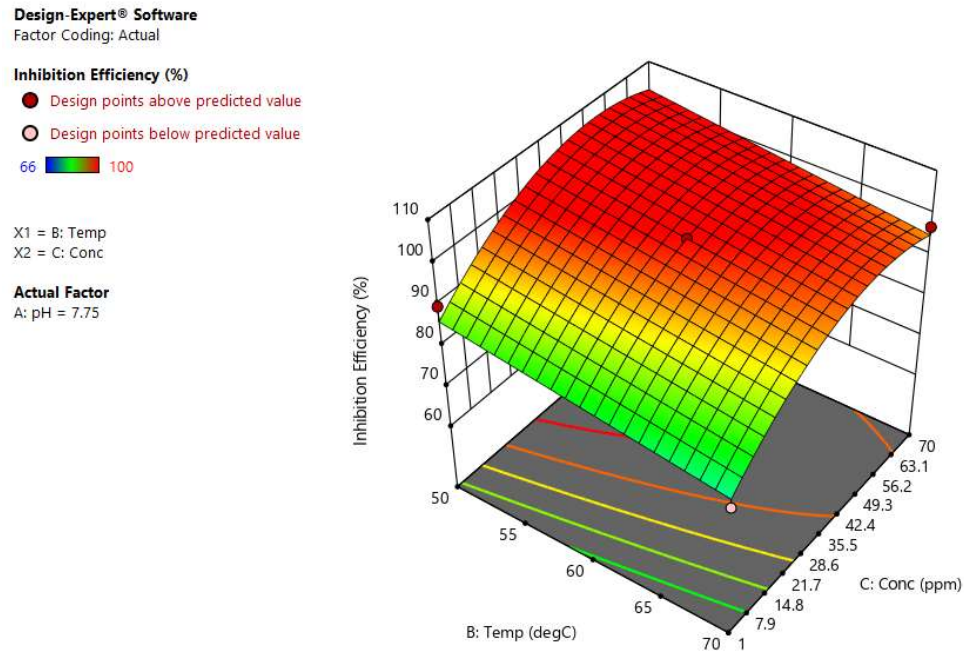

Fig. 4: 3D response surface with contour plot for the effect of temperature and copolymer concentration on calcite scale inhibition efficiency.

concentration is increased. The tested inhibitor (MA-AD) is a threshold or kinetic scale inhibitor that affects the kinetics of the nucleation and crystal growth of scaling species, thus allowing supersaturation without scale formation. Hence, threshold scale inhibitors can be dosed at low concentrations, far below the stoichiometric amounts required for inhibition through chelation.

Optimization of Responses

The desirability function (DF) is widely used in RSM to

determine the combination of input variables to optimize multiple responses (Kalil et al. 2000, Harrington, 1965). If y(x) denotes the response as a function of the input parameters (x), the desirability function is denoted by DF = $d_k(y_k)$ with a range in values between 0 and 1. A value of 0 for DF represents a completely undesirable or unfavorable value of y, and DF = 1 represents a completely desirable value of y. The desirability function is used to find the operating conditions (x_k) which result in the most desirable responses. This is done by maximizing the overall desirability as a function of input parameters. Depending on whether a particular response y_i has to be maximized, minimized, or assigned to a target value, Different desirability functions can be used depending on whether the observed response (y) need to be minimized, maximized, or set to a target value (Del Castillo 1996).

Optimization was done to obtain the operating conditions resulting in the maximum inhibition efficiency equal to 100%. In this case, the operating conditions should be 7.75, 60°C, and 35.5 ppm for pH, temperature, and copolymer concentration, respectively, to attain maximum calcium carbonate inhibition. With sufficient data sets, this model could be improved and used for determining minimum inhibitor concentration (MIC) for the given operating conditions. This approach can also be extended for other scale inhibitors and could be used to select the appropriate scale inhibitor for a given set of conditions.

CONCLUSIONS

This work focused on applying statistical analysis techniques such as response surface methodology to understand the effect of different operating parameters on the inhibition efficiency of maleic acid - acrylamide copolymer scale inhibitor on $CaCO_3$ scales. The copolymer was synthesized, and its inhibition efficiency on the calcite scale was tested using static jar tests at various levels of temperature, pH, and inhibitor concentration. The design of experiments was formulated using Box-Behnken design with three levels for each factor. The results were analyzed using ANOVA and response surfaces. The analysis showed that the linear effect of the variables had the highest impact on inhibition efficiency. The order of influence of the parameters for the calcite scale was copolymer concentration > pH > temperature. There was also a significant effect of the interaction between pH – concentration, and temperature-concentration factors, indicating that scale inhibition involves a complex interaction of operating parameters. The linear factors had the highest influence, followed by the quadratic term of temperature and the interaction effects. A quadratic transfer function was fitted to the data by selecting the significant factors using backward elimination regression. This model can be used to predict the performance of the scale inhibitor for different operating conditions. Statistical methods could be used to model and optimize complex phenomena such as scale inhibition and aid in optimum mitigation of inorganic scaling in industrial systems.

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