

Determination of the Optimum Conditions in Evaluation of Kiwi Juice as Green Corrosion Inhibitor of Steel in Hydrochloric Acid

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ABSTRACT

The corrosion protection of low carbon steel in 2.5 M HCl solution by kiwi juice was studied at different temperatures and immersion times by weight loss technique. To study the determination of the optimum conditions from statistical design in evaluation of a corrosion inhibitor, three variables, were considered as the most dominant variables. These variables are: temperature, inhibitor concentration (extracted kiwi juice) and immersion time at static conditions.

These three variables are manipulated through the experimental work using central composite rotatable Box – Wilson Experimental Design (BWED) where second order polynomial model was proposed to correlate the studied variables with the corrosion rate of low carbon steel alloy to estimate the coefficients by nonlinear regression analysis method based on Rosenbrock and Quasi-Newton estimation method in as few experiments as possible to determinate of the optimum conditions of the proposed polynomial adopted via *STATISTICA* software. The parametric study on corrosion inhibition process using response surface methodology (RSM) is presented in this paper.

The study shows that the immersion time and temperature of corroding medium had shown negative dependence of great significance in increase the corrosion rate while the other studied variable (i.e. inhibitor concentration) had shown large positive dependence in reduce the corrosion rate of low carbon steel alloy.

Optimum conditions for achieving the minimum corrosion rate are obtained from optimizing the above correlation and are found as follow: 42.86 °C temperature of corroding medium, 29.29 cm^3/L inhibitor concentration and 2.65 h immersion time. In these circumstances, the value of inhibition efficiency obtained was 96.09 %. It could be concluded that Box-Wilson experimental design was adequately applicable in the optimization of process variables and that kiwi juice sufficiently inhibited the corrosion for low carbon steel at the conditions of the experiment.

Keywords: RSM; corrosion inhibition; kiwi juice; hydrochloric acid solution

إيجاد الظروف المثلى في تقييم عصير الكيوي كمانع تآكل طبيعي للحديد في حامض الهيدر وكلوريك

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الخلاصة

تم دراسة حماية تآكل حديد منخفض الكاربون في محلول حامض الهيدروكلوريك (2.5 مولاري) بأستخدام عصير الكيوي كمثبط لعملية التاكل عند مختلف درجات الحرارة وفترات زمن غمر بأستخدام تقنية فقدان الوزن لحساب معدلات التآكل. لدراسة تحديد الظروف المثلى من التصميم الأحصائي في تقييم أداء مثبط عملية تآكل أُخذت بنظر الأعتبار ثلاثة متغيرات

أعتبرت الأكثر تأثيرًا على عملية التآكل. العوامل المؤثرة هي درجة الحرارة و تركيز المثبط (عصير الكيوي) و فترة زمن غمر عند ظروف ساكنة

المتغير إت الثلاثة تم معالجتها من خلال تصميم التجارب العملية بإستخدام طريقة التصميم الدوار المركب المركزي -Box) (Wilson لربط المتغيرات أعلاه بمعادلة متعددة الحدود من الدرجة الثانية التي تشير الى العلاقة بين المتغيرات الثلاثة ومعدل التآكل لسبيكة الحديد منخفض الكاربون من خلال إيجاد المعاملات بأستخدام طريقة تحليل التراجع والأرتداد اللاخطي المعتمدة على طريقة حساب (Rosenbrock and Quasi-Newton) بأقل عدد تجارب ممكن لتحديد الظروف المثلق للمعادلة المفترضة المتعددة الحدود المعتمدة بواسطة (STATISTICA software). ذكرت در اسة العوامل على عملية تثبيط التاكل بأستخدام منهج سطح الأستجابة في هذه المقالة.

الدراسة تبين بأن زمن الغمر ودرجة حرارة محلول التأكل لها تأثيرا سلبيا في زيادة معدل التأكل في حين أن المتغير الآخر (تركيز المثبط) كان له تأثير ا أيجابيا كبير ا في تقليل معدل تآكل سبيكة حديد منَّخفض الكاربون . تم إستخر اج الظروف المثلي للتجارب للحصول على أقل معدل تأكل و هي : درجة حرارة محلول التأكل C° 42.86 , تركيز المثبط 29.29 و 29.29 و زمن غمر h 2.65 h. وعند هذه الظروف في قيمة كفاءة المادة المانعة للتاكل كانت % 96.09. أستنتج من النتائج بأن التصميم العملي Box-Wilson قابل للتطبيق على نحو ملائم في أختيار الأفضل لمتغير ات العملية و عصير الكيوي لـه التأثير على نحو كافي في تثبيط تأكل الحديد منخفض الكاربون عند ظروف التجربة

الكلمات الرئيسيه: RSM ، تثبيط تأكل ، عصير كيوى ، محلول حامض الهيدروكلوريك

1. INTRODUCTION

In the chemical and engineering industry experimental designs are particularly applied to study of process variables and how they affect on the product, **Perry**, and **Green**, 2000.

Three basic types of statistically designated experiments are most often used in chemical and engineering industry. These are 1. Fractional-Factorial design. 2. Box-Wilson design. 3. Factorial design. Box-Wilson designs and, in particular, the two-level Box-Wilson design, have been treated in the literature both theoretically and from application point of view, **Box** and **Hunto**, **2005**. However, it's desirable here to review briefly their major characteristics:

Consider the weight loss resulting from corrosion of a low carbon steel specimen has to be determined as function of temperature, inhibitor concentration and immersion time, and let the two levels of temperature, inhibitor concentration and immersion time. The experiments carried out under these conditions are presented in the experimental design.

Many metals and alloys which used in different human activities are susceptible to different mechanisms of corrosion due to their exposure to different corrosive media. Among these, low carbon steel is very important. One of the methods used to reduce the rate of metallic corrosion is addition of inhibitors. Many studies have been carried out to find suitable compounds to be used as corrosion inhibitors for this metal in different aqueous solutions. These studies reported that there are a number of organic and inorganic compounds which can inhibit corrosion of steel, Musa, et al., 2010, Khadom, et al., 2009, Yaro, et al., 2000. Many researchers were conducted to examine some naturally occurring substances as corrosion inhibitors for different metals in various environments, El-Etre, et al., 2005, El-Etre, 2003.

The use of inhibitors is an important method of protecting materials against deterioration. Inhibitors are chemicals that often work by adsorbing themselves on the metallic surface by forming a film, Ebenso, et al., 2009, Noor, 2008, Ebenso, et al., 2008, Oguzie, 2006, Gazquez, 2006. Most corrosion inhibitors are either synthesized from cheap raw materials or are chosen from organic compounds containing electronegative functional groups and π-electrons in triple or conjugated double bonds. The sites of aromatic rings and hetro atoms (such as S, N, O and P) are the major adsorption centers for these inhibitors, Dubey, and Sing, 2007. Broad spectra of organic compounds are available as corrosion inhibitors. Of these, only very few are actually used in practice. This is partly due to the fact that desirable properties of an inhibitor usually



extend beyond those simply related to metal protection. Considerations of cost, toxicity, availability and environmental friendliness are of considerable important. Accordingly, the replacement of some toxic, expensive chemical inhibitors by inhibitors obtained from natural sources is necessary. Apart from being readily available, cheap and a renewable source of materials, naturally occurring substances are eco-friendly and ecologically acceptable. Naturally occurring substances are biodegradable and do not contain heavy metals or other toxic compounds. Among the so-called "green corrosion inhibitors" are organic compounds that act by adsorption on the metal surface, such as extracts of natural substances, Raja, and Sethuraman, 2008. The efficiency of these organic corrosion inhibitors is related to the presence of polar functional groups with S, O or N atoms in the molecule, heterocyclic compounds and лelectrons. The polar function is usually regarded as the reaction center for the establishment of the adsorption process, Roberge, 1999. One of these natural compounds is fruits. Fruit is a rich source of chemicals such as vitamins, minerals, organic acids and phenolic compounds. The kiwi juice includes different level of phenolic compounds. The hydroxybenzoic acids and hydroxycinnamic acids are the most common compounds in kiwi fruits, Keith, 2012. Note that the kiwi juice extract use for the first time as natural inhibitor in this article to the corrosion of low carbon steel alloy in hydrochloric acid solution. Alaneme, and Olusegun, 2012 have studied the inhibitive property of lignin extract of sun flower (tithonia diversifolia) as green corrosion inhibitor in 1 M H₂SO₄ solution using experimental design. Da Rocha, et al., 2012 have studied the inhibitive action of grape pomace extract as green corrosion inhibitor against the corrosion of carbon steel in a 1 mol L¹ HCl solution using weight loss measurements. Afia, et al., 2014 have studied the inhibitive property of acid garlic essential oil as a green corrosion inhibitor in a 1 M HCl solution by weight loss method of monitoring corrosion rate.

Therefore, the present study of inhibition of kiwi juice on low carbon steel corrosion and the optimization of the independent variables is necessary, as well as important to the environment.

The experiments discussed in this work were designed to optimize the inhibition of low carbon steel by extracted kiwi juice where several variables, including temperature, inhibitor concentration and immersion time would influence the corrosion rate.

Design of experiment is a statistical procedure that can reduce significantly the number of experiment, keeping however, the reliability of the conclusions at high standard. The traditional experimental method, one factor at a time approach can hardly be used to establish relationships among all the experimental input factors and the output responses. Even though the traditional approach can be useful in finding predominate factors in this situation, it is difficult to observe the optimum value of the working parameters as no interaction among them is considered. To solve this problem and obtain a probable optimum, design of experiment (DOE) offers a better alternative to study the effect of variables and their responses with minimum number of experiments, **Giovanilton**, et al., 2011, Montgomery, 2005. A Box-Wilson is used to evaluate two or more factors simultaneously. The treatment is combination of level of the factors. The advantages of Box-Wilson design over one-factor-at-a-time experiment are that they are more efficient and they allow interactions to be detected.

The aim of the present work is to investigate the influence of the operating parameters (temperature, inhibitor concentration of extracted kiwi juice and immersion time) on corrosion rate of low carbon steel alloy in hydrochloric acid solution under optimum conditions that are obtained by using Box-Wilson techniques, then estimation of the inhibition efficiency of extracted kiwi juice as green corrosion inhibitor in order to find a naturally, cheap and environmentally safe substance that could be used for inhibition purposes.

2. EXPERIMENTAL WORK

2.1 Metal Preparation

The material of working electrodes used in this study is low carbon steel in foil coupons of a tag shape were used as specimens. This material is cut of specimens by dimensions of $(2\times3\times0.2 \text{ cm})$. Analysis of these specimens was carried out at National Center for Construction Laboratories and Research /Baghdad/Iraq. **Table 4** shows the nominal and the analytical chemical compositions of material used in this work. This analysis indicates that the main elements of material are within the standard limits.

2.2 Extraction of Inhibitor

Completely fresh ripe kiwi fruit weighing 2 kg was purchased from the local market (Baghdad-Iraq). The fruits washed in cold running tap water followed by distilled water, dried with clean tissue, and extracted the crust from it and then was squeezed mechanically to get corresponding juice. The resulting juice nomination in order to get a homogeneous solution. In this process, 1/2 liter of kiwi juice was obtained. The extracted juice was kept frozen at - 4 °C in glass graduated cylinder until further experiment. The concentrations of inhibitor were closer as 5, 15, 25, 35 and 45 cm³/L kiwi juice concentration.

2.3 Box-Wilson Experimental Design (BWED) and Optimization of Variables

The running expense for each experiment is relatively high which leads to need of design of experiment. The design of experiment is a statistical tool which helps to minimize the number of experiments so that appropriate data will be collected, the minimum number of experiments will be performed to acquire the necessary technical information and suitable statistical methods will be used to analyze the collected data. The initial task of this stage is to find out the key process control parameters with their ranges and performance evaluation parameter (output) that is to be measured. The levels of each variable represent the range for which the effect of that variable is desired to be known.

Box-Wilson experimental design is easy to apply to many engineering situations, making it a powerful yet simple tool and a series of tests for characterizing a physical mechanism. These series of experiments have been developed which efficiently serve as a basic deriving the mathematical model of process, **Jeff**, and **Michael**, **2009**. For three variables, the quadratic polynomial equation can be represented as follows:

$$Y = B_0 + B_1 X_1 + B_2 X_2 + B_3 X_3 + B_{11} X_1^2 + B_{22} X_2^2 + B_{33} X_3^2 + B_{12} X_1 X_2 + B_{13} X_1 X_3 + B_{23} X_2 X_3$$
(1)

A preliminary step is to set up the relationships between the coded level and the corresponding real variables, which are required in the determination of experimental range by the following equation.

$$X_{coded} = \frac{X_{real} - X_{center}}{\frac{X_{center} - X_{min}}{\sqrt{k}}}$$
(2)

The coded variables take the value between -2 and 2 in accordance with the central composite rotatable suggested by Tarantino, **Tarantino**, **2010**.

The range of real variables for the system could be represented in **Table 1**. The expression of coded level for the system is estimated from Eq. (2)



$$X_{1} = \frac{T - 45}{8.66}$$
(3)
$$X_{2} = \frac{C - 25}{11.55}$$
(4)

$$X_3 = \frac{t-3}{1.15}$$
(5)

Where:

T = Temperature of corroding medium (°C) C = Inhibitor Concentration (cm³/L) t = Immersion Time (h)

Table 2 shows the relationship between the coded level and corresponding real variables.

The number of experiments that must be done shown in Table 3.

2.4 Experimentation

The cleaning procedure was as follows: The specimens were first degreased with analar benzene and acetone at 25 °C, and then annealed in a vacuum oven at 550 °C for 1.5 h and cooled to room temperature. Specimens were abraded in sequence under running tap water using emery paper of grade numbers 220,320,400 and 600, rinsed with running tap water followed by distilled water, dried with clean tissue, immersed in acetone and benzene, and kept in desiccator over silica gel bed until time of use. The specimens were completely submerged in 200 cm³ corrosion solution at 2.5 M HCl, 30, 37.5, 45, 52.5 and 60 °C, and 5, 15, 25, 35 and 45 cm³/L kiwi juice concentration for a period of time 1, 2, 3, 4 and 5 h. After each experiment the specimens were washed with running tap water followed by distilled water, dried with clean tissue, immersed in desiccators over silica gel bed to dry, then weighed by high accuracy electronic balance.

3. RESULTS AND DISCUSSION

The corrosion of low carbon steel in 2.5 M HCl solution containing various concentrations of inhibitor at different temperatures and immersion times was studied by weight loss measurments. The corrosion rate of low carbon steel was determined using the relation:

$$C.R = \frac{Weight loss (g)}{Area (m^2) x Time (day)}$$
(6)

C. *R*: Corrosion rate $(g/m^2, day)$ (gmd)

The percentage inhibition efficiency (IE (%)) was calculated at optimum conditions using the relationship, Alaneme, and Olusegun, 2012:

$$IE\% = \frac{W_{uninhibt} - W_{inhibit}}{W_{uninhibit}} \times 100$$
⁽⁷⁾

Where $W_{uninhibt}$ and $W_{inhibit}$ are the corrosion rates in absence and presence of inhibitor respectively.

Table 5 will be first fitted through nonlinear regression analysis to estimate the coefficients of the proposed model. **Table 5** also shows the corrosion rate of the specimens that reached through the experimental work and the predicated corrosion rate, which are designed according to the central composite rotatable design method.

3.1 Response Surface Methodology:

Response Surface Methodology (RSM) can be regarded as a statistical technique for optimizing the objective functions through some mathematical methods. Basically, this involves doing several experiments. Using the result of one experiment, direction for what to do next is provided, **Lenth**, **2010**. The field of RSM consists of the experimental strategy for exploring the space of the process or independent variables, empirical statistical modeling to develop an appropriate approximating relationship between the yield and process variables and optimization methods for finding the values of process variables that produce desirable values of the response, **Acherjee**, **et al.**, **2009**. The main objective of RSM is to find the combination of factor levels to achieve the optimal response.

In the present work the RSM is used to study the parametric effect of process parameters on corrosion rate. The result of experiment is used to develop the regression model as discussed in the following section. The effect of temperature, inhibitor concentration and immersion time on corrosion rate is shown graphically in **Fig. 3** through **Fig. 5** by constructing the response surface and contour diagram.

Multiple regression analysis (MRA) is one of the most widely used statistical techniques for analyzing multifactor data, **Davim**, **et al.**, **2008**. In the present work a regression model is developed by establishment of correlation between the process control parameters such as temperature, inhibitor concentration and immersion time with the output parameter namely corrosion rate, one can use this relationship in various algorithms. A quadratic regression model for response corrosion rate (*C* . *R*) is developed based on experimental results using the coded data of the central composite rotatable design, **Table 6** the coefficients of the 2^{nd} order polynomial were estimated by implementing nonlinear least squares regression analysis technique based on Rosenbrock and Quasi-Newton estimation method via the *STATISTICA* software of version 10.1 can be used for estimation of coefficients *B*₀, *B*₁,..., *B*₂₃. This model will help to predict the response as a function of independent variables and their interactions.

In order to obtain the second-order response surface model equation the following equation may be assumed:

$$Y = B_0 + \sum_{i=1}^n B_i X_i + \sum_{i=1}^n B_{ii} X_i^2 + \sum_{i< j}^n B_{ij} X_i X_j + e_i$$
(8)

Where Y is the predicted response, B_0 the intercept coefficient, B_i the linear terms, B_{ii} the squared terms, B_{ij} the interaction terms, X_i and X_j are coded levels of the process control variables, the residual, e_i measures the experimental error of the observation number and n is the total number of designed variables, **Jian-Ping**, et al., 2007, Montgomery, 2005. The coefficients of the model for the corresponding response are estimated using multiple regression analysis technique included in RSM. The response surfaces of corrosion rate can be expressed by the following quadratic equation in terms of coded values and real units.

$$C.R_{weight \, loss \, (gmd)} = 70.41 + 8.73 \, T - 18.44 \, C + 10.42 \, t + 10.24 \, T^2 + 22.34 \, C^2 + 12.56 \, t^2 - 0.34 \, T \, \times \, C + 7.42 \, T \, \times \, t + 1.11 \, C \, \times t$$
(9)



(13)

Where, T = Temperature (°C) C = Concentration of Inhibitor (cm³/L) t = Immersion Time (h)

3.2 Statistical and Mathematical Analysis:

From **Table 5**:

 $e_i = (C.R_E - C.R_P)$ An estimation of the experimental error variance (S_r^2) is obtained by dividing the residual sum of squares (e_i) by number of degree of freedom (γ) ; where:

$$\gamma = N_i - N_{\text{coff}}$$
(10)
Where:
 N_i : No. of experiments

 $N_{\text{coff}}: \text{ No. of coefficients in the model}$ $\gamma = 18 - 10 = 8$ $S_r^2 = \sum e_i^2 / \gamma$ (12)

The estimated variance of coefficients (S_b^2) is then calculated by the following formula: $S_b^2 = S_r^2 / \sum X^2$

The significant coefficient can be estimated by comparing the value of (B^2/S_b^2) to the critical value $F_{0.95(1,8)} = 0.29$ of the F-distribution at 95% level of confidence, **Jeff**, and **Michael**, **2009**. The results of these calculations are shown in **Table 7** for low carbon steel alloy. The final form of the proposed model was as follows:

$$C.R_{weight \, loss \, (gmd)} = 70.41 + 8.73 \, T - 18.44 \, C + 10.42 \, t + 10.24 \, T^2 + 22.34 \, C^2 + 12.56 \, t^2 + 7.42 \, T \times t$$
(14)

The accuracy of an empirical model can also be done by means of statistical parameters, for example, correlation coefficient. The correlation coefficient (\mathbb{R}^2) is a statistical measure of the strength of correlation between the predicted and measured values, **Devore**, **2005**. For the current problem, the following result is obtained: $\mathbb{R}^2 = 0.895$. Fig. 2 shows predicted corrosion rate by Eq. (14) against experimental one.

3.3 Inhibition Optimization Result and Estimating the Percentage Inhibition Efficiency:

According to Eq. (14), using *POLYMATH* software version 4.02 in terms of minimum corrosion rate, the optimum values were obtained. The optimum values of the studied variables in coded and real form are listed in **Table 8** below for low carbon steel alloy. A validation experiment was conducted at the specified optimized values and the results were used to calculate the response (corrosion rate). The practical value of the corrosion rate at the optimum conditions was comparable with the theoretical value and the result was closed.

The percentage inhibition efficiency at the optimum conditions can be calculated from the Eq. (7).

$$IE\% = \frac{0.1049 - 0.0041}{0.1049} \times 100 = 96.09\%$$

3.4 Influence of Process Parameters on Corrosion Rate:

The aim of this study was to find a corrosion rate whose features would have been previously defined from the operative conditions extracted from the quadratic mathematical model.

Because the direct exploitation of the equation was delicate, it was convenient to restore it under a graphic representation; while fixing one of the three factors of the survey, it was possible to represent the response surface materializing the surface of regression in a three-dimensional space. It was also possible to project the equation in a design under isoresponse curves, interpreted as card curves level.

A. Evolution of corrosion rate as a function of the temperature and the concentration of corrosion inhibitor:

Fig. 3 shows the evolution of the corrosion rate as a function of the temperature and the concentration of corrosion inhibitor (extracted kiwi juice). It can be seen that the inhibitor concentration has a strong influence on the tentative response. The minimal corrosion rate is obtained for an inhibitor concentration of 0.37 in coded variable, i.e., 29.29 cm³/L in real variable. Considering simultaneous effects of temperature and inhibitor concentration is presented in **Fig. 3** contour plot. The figure shows, in high temperature (60 °C), the increase of corrosion rate is higher than in lower temperature (30 °C). This is also in close agreement with the research done by, **Da Rocha, et al., 2012**.

B. Evolution of corrosion rate as a function of the temperature and the immersion time:

Fig. 4 shows the synergism between the two factors: the temperature and immersion time in corrosion rate at inhibitor concentration 29.29 cm³/L. It can be noted that the effect of the immersion time differed according to the corrosion rate's variation. This effect becomes positive and even more important when the corrosion rate is degraded (reduced). Analysis of corrosion rate as effects of interaction between temperature and immersion time is shown in **Fig. 4** contour plot. The model shows a decrease followed by growing of corrosion rate due to temperature and immersion time at the optimum condition of inhibitor concentration 29.29 cm³/L and this in agreement with, **Alaneme**, and **Olusegun**, **2012**.

C. Evolution of corrosion rate as a function of the concentration of corrosion inhibitor and the immersion time:

Fig. 5 represents the evolution of the corrosion rate as a function of the inhibitor concentration and the immersion time. This figure shows that the corrosion rate initially decreased when the inhibitor concentration and immersion time increased. This evolution was however more accentuated up to a concentration of 29.29 cm³/L kiwi juice until it reaches a minimum value of corrosion rate at 2.65 h, which contributes to the great tendency of corrosion protection causes decrease in corrosion rate then it begins to increase again with increasing the inhibitor concentration and immersion time. **Fig. 5** contour plot presents a polynomial surface response relating to effect of inhibitor concentration and immersion time on corrosion rate. It is shown that different corrosion rate is observed for inhibitor concentration and immersion time. Immersion time increased corrosion rate from 70-220 gmd along the immersion time setting (1-5



h). Concentration of inhibitor from 5-45 cm^3/L has decreased corrosion rate from 270-70 gmd. This is also in close agreement with the research done by, **Afia**, et al., 2014.

4. CONCLUSION

- 1. The results of experiment are extended to develop the second order polynomial regression model of the objective function (corrosion rate) in terms of three variables (i.e., temperature of corroding medium, inhibitor concentration and immersion time) using response surface methodology (RSM) gives Eq. (14), which adequately describes the behavior of the process throughout the studied range.
- 2. The three variables effect on the corrosion rate in following order:

Inhibitor concentration > Immersion time > Temperature.

- 3. The green corrosion inhibitor made up the kiwi juice extract successfully reduced the corrosion rates of low carbon steel alloy in 2.5 M HCl solution.
- 4. The optimum conditions as predicted from Eq. (14) is 42.86 °C temperature of corroding medium, 29.29 cm³/L inhibitor concentration and 2.65 h of immersion time for low carbon steel alloy.
- 5. Kiwi juice extract acts as a corrosion inhibitor of low carbon steel with inhibition efficiency of 96.09 % under the optimum conditions.
- 6. The corrosion rate of low carbon steel alloy in 2.5 M HCl solution in presence of kiwi juice extract, increases with increasing temperature and immersion time, and decreased with increasing concentration of inhibitor.
- 7. The analysis of statistical central composite rotatable Box–Wilson Experimental Design (BWED), generally, shows that the interaction effects on the corrosion rate by weight loss technique (within the studied range) is less pronounced compared with the main and square variables except the interaction effect of $(T \times t)$ in presence of extracted kiwi juice.

ACKNOWLEDGEMENT

The author would like to thank National Center for Construction Laboratories and Research /Baghdad/Iraq for financial support.

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Table 1. The experimental range of variables.							
Temperature (°C)	Inhibitor Concentration (cm ³ /L)	Immersion Time (h)					
30-60	5-45	1-5					

Table 2. Real and coded of the independent variables used in RSM study.

Variables	Levels					
X ₁ , X ₂ , X ₃	-2	-1	0	1	2	
$X_1 = \text{Temperature (°C)}$	30	37.5	45	52.5	60	
X_2 = Inhibitor Concentration (cm ³ /L)	5	15	25	35	45	
X_3 = Immersion Time (h)	1	2	3	4	5	

Table 3. Sequence of experiments according to central composite rotatable experimental design.

Evn		Code ariab		Real Variables			
Exp. No.	<i>X</i> ₁	<i>X</i> ₂	<i>X</i> ₃	Temp. (°C)	Inhibitor Conc. (cm ³ /L)	Immersion Time (h)	
1	-1	-1	-1	37.5	15	2	
2	1	-1	-1	52.5	15	2	
3	-1	1	-1	37.5	35	2	
4	1	1	-1	52.5	35	2	
5	-1	-1	1	37.5	15	4	
6	1	-1	1	52.5	15	4	
7	-1	1	1	37.5	35	4	
8	1	1	1	52.5	35	4	
9	-2	0	0	30	25	3	
10	2	0	0	60	25	3	
11	0	-2	0	45	5	3	
12	0	2	0	45	45	3	
13	0	0	-2	45	25	1	
14	0	0	2	45	25	5	
15	0	0	0	45	25	3	
16	0	0	0	45	25	3	
17	0	0	0	45	25	3	
18	0	0	0	45	25	3	



	composition (wt. %) of low carbon steel alloy.										
Metal	Fe	С	Mn	Si	Р	S	Cr	Mo	Ni	Al	
Nominal	Rem.	0.02-0.13	0.3-0.6	0.002	0.004	0.005	0.024	0.0008	0.014	0.003	
Analytical	Rem.	0.040	0.309	0.004	0.005	0.007	0.021	0.0009	0.010	0.004	

Table 4. A nominal, **Astm**, **2004** and analytical chemical
composition (wt. %) of low carbon steel alloy.

Table 5. Two-level central composite rotatable experimental design of the independent variables with the observed, predicted values and experimental error for the response.

Exp.	Coded Variables		Real Variables			Exp. Corrosion Rate	Predicted Corrosion Rate	Corresponding residual	
No.	<i>X</i> ₁	<i>X</i> ₂	<i>X</i> ₃	Temp. (°C)	Inhibitor Conc. (cm ³ /L)	Immersion Time (h)	C.R _E (gmd)	C.R _p (gmd)	$\mathbf{e}_{i} = \mathbf{C}.\mathbf{R}_{E} - \mathbf{C}.\mathbf{R}_{P}$
1	-1	-1	-1	37.5	15	2	100.53	123.04	-22.51
2	1	-1	-1	52.5	15	2	125.88	126.33	-0.45
3	-1	1	-1	37.5	35	2	110.17	84.61	25.56
4	1	1	-1	52.5	35	2	111.45	86.54	24.91
5	-1	-1	1	37.5	15	4	130.44	126.81	3.63
6	1	-1	1	52.5	15	4	162.76	159.79	2.97
7	-1	1	1	37.5	35	4	121.82	92.83	28.99
8	1	1	1	52.5	35	4	175.49	124.45	51.04
9	-2	0	0	30	25	3	90.35	93.92	-3.57
10	2	0	0	60	25	3	103.87	128.84	-24.97
11	0	-2	0	45	5	3	219.09	196.64	22.45
12	0	2	0	45	45	3	71.89	122.87	-50.98
13	0	0	-2	45	25	1	100.33	99.82	0.51
14	0	0	2	45	25	5	112.44	141.49	-29.05
15	0	0	0	45	25	3	63.45	70.41	-6.95
16	0	0	0	45	25	3	65.32	70.41	-5.09
17	0	0	0	45	25	3	61.87	70.41	-8.54
18	0	0	0	45	25	3	62.45	70.41	-7.96

Table 6. The coefficient values of the predicted correlation.

Coeff.	B_0	B_1	B_2	B_{3}	<i>B</i> ₁₁	<i>B</i> ₂₂	<i>B</i> ₃₃	<i>B</i> ₁₂	<i>B</i> ₁₃	<i>B</i> ₂₃
Value	70.41	8.73	-18.44	10.42	10.24	22.34	12.56	-0.34	7.42	1.11
Correlation Coefficient (R ²)			0.3	895		Proportion of Variance			0.67	
Final Value of loss function			1004	42.18						

Table 7. Analysis of variance (ANOVA) for the fitted model.									
Constant Estimated	ΣX^2	Estimated Coefficient (<i>B</i>)	Variance $Sb^2 = Sr^2 / \Sigma X^2$	F-value = B^2/Sb^2	$F_{0.95(1,8)} = 0.29$				
B_1	16	8.73	78.4539	0.97	\mathbf{S}^*				
B_2	16	-18.44	78.4539	4.33	S				
B_3	16	10.42	78.4539	1.38	S				
<i>B</i> ₁₁	16	10.24	78.4539	1.34	S				
<i>B</i> ₂₂	16	22.34	78.4539	6.36	S				
<i>B</i> ₃₃	16	12.56	78.4539	2.01	S				
B_{12}	8	-0.34	156.9078	0.001	NS ^{**}				
B ₁₃	8	7.42	156.9078	0.35	S				
B_{23}	8	1.11	156.9078	0.01	NS				
(*) a: :c:									

Table 7. Analysis of variance (ANOVA) for the fitted model

^(*) Significant ^(**) Non-significant

Table 8. Optimum values of the process variables for minimum corrosion rate.

Variables	^	Optimum Values (low carbon steel alloy)			
	Coded	Real			
$X_1 = \text{Temperature (°C)}$	-0.25	42.86			
X_2 = Inhibitor Concentration (cm ³ /L)	0.37	29.29			
X_3 = Immersion Time (h)	-0.30	2.65			
Function Minimum (Corrosion rate, gmd)	62.35				



Figure 1. Experimental set-up for weight loss investigation.



Figure 2. Predicted corrosion rate versus experimental corrosion rate.

Number 8



Figure 3. Response surface plot (top) and contour plot (bottom) showing the variation of corrosion rate as a function of the temperature and extracted kiwi juice concentration at the optimum value (2.65 h).



Figure 4. Response surface plot (top) and contour plot (bottom) showing the variation of corrosion rate as a function of the temperature and immersion time at the optimum value (29.29 cm³/L).





Figure 5. Response surface plot (top) and contour plot (bottom) showing the variation of corrosion rate as a function of the extracted kiwi juice concentration and immersion time at the optimum value ($42.86 \,^{\circ}$ C).