# Modelling and Optimization of Corrosion Inhibition of Mild Steel in Phosphoric Acid by Red Pomegranate Peels Aqueous Extract

**Khalid Hamid Rashid**
Lecturer
Chemical Engineering Department
University of Technology
email: 80053@uotechnology.edu.iq

**Zaidoon Muhsen Shakor**
Assistant Professor
Chemical Engineering Department
University of Technology
email: 80063@uotechnology.edu.iq

**Ayad Bahaddin Ahmed**
Lecturer
Med. Tech. Institute/AL- Mansour
Middle Technical University
email: ahmed.ayad2002@gmail.com

## ABSTRACT

Taguchi experimental design (TED) is applied to find the optimum effectiveness of aqueous Red Pomegranate Peel (RPP) extract as a green inhibitor for the corrosion of mild steel in 2M $H_3PO_4$ solution. The Taguchi methodology has been used to study the effects of changing, temperature, RPP concentration and contact period, at three levels. Weight-loss measurements were designed by construction a $L_9$ orthogonal arrangement of experiments. Results of the efficiencies of inhibition were embraced for the signal to noise proportion & investigation of variance (ANOVA). The results were further processed with a *MINITAB-17* software package to find the optimal conditions for inhibitor usage. Second order polynomial model was used for experimental data fitting. Optimum conditions for achieving the maximum corrosion inhibition efficiency are obtained from optimizing the above model and are found as follow: 39.66 °C temperature of acidic media, 38.29 ml/L inhibitor concentration and 2.95 h contact period. Results demonstrated that rate of corrosion was increased with temperature increasing & decreasing inhibitor concentration. It was concluded that the Taguchi design was adequately useful in the optimization of operating parameters and that RPP sufficiently inhibited the corrosion of steel at the range of variables studied.

**Keywords:** corrosion, inhibition, red pomegranate peel, optimization, weight loss, taguchi experimental design

# نمذجة وإيجاد الظروف المثلى لتثبيط تآكل الحديد الكاربوني الواطيء الكاربون في حامض الفسفوريك بالمستخلص المائي لقشور الرمان الاحمر

**م.د. خالد حامد رشيد**
قسم الهندسة الكيمياوية
الجامعة التكنولوجية

**أ.م.د. زيدون محسن شكور**
قسم الهندسة الكيمياوية
الجامعة التكنولوجية

**م.د. اياد بهاء الدين احمد**
قسم التحليلات المرضية
المعهد الطبي التقني – المنصور
الجامعة التقنية الوسطى

## الخلاصة

تم إستخدام تقنية تاكوجي لتصميم التجارب لإيجاد الظروف المثلى لإستخدام المستخلص المائي لقشور الرمان الاحمر كمآنع لتآكل الحديد الواطئ الكاربون في محلول 2 مولاري لحامض الفسفوريك. تم أستخدام هذه التقنية لدراسة التأثير المشترك لتغيير درجات الحرارة، تركيز المثبط و فترة التلامس وعلى ثلاثة مستويات. تم تصميم تجارب قياس معدل التآكل بتقنية فقدان الوزن. نتائج كفاءة التثبيط المستحصلة أستخدمت في دراسة نسبة الاشارة الى التشويش وتحليل التباين بواسطة أستخدام البرنامج الأحصائي *MINITAB-17* لإيجاد الظروف المثلى لإستخدام المثبط ، أعطى تعبير رياضي من الدرجة الثانية لتمثيل النتائج. بينت النتائج أن الظروف المثلى لإستخدام المثبط هي 38.29 مل / لتر لتركيز المثبط عند درجة حرارة 39.66 م° للوسط الحامضي ولوقت تلامس مقداره 2.95 ساعة. بينت النتائج معدل التآكل يزداد بزيادة درجة الحرارة ونقصان تركيز المثبط في الظروف تحت الدراسة . من خلال البحث تم أستنتاج أمكانية أستخدام المستخلص المائي لقشور الرمان كمآنع جيد لتآكل الحديد الكاربوني في محلول 2 مولاري حامض الفسفوريك وتم تأكيد فعالية تقنية تاكوجي في إستنباط الظروف المثلى لعملية التثبيط.

**الكلمات الرئيسية :** تآكل ، تثبيط ، قشور الرمان الأحمر ، إيجاد الظروف المثلى ، فقدان الوزن ، تصميم تاكوجي العملي.

## 1. INTRODUCTION

Mild steel (MS) is a standout amongst the most generally utilized building materials, in spite of its moderately constrained corrosion resistance it is an essential material of decision because of minimal effort & simple fabrication. Corrosion is one of the fundamental worries in the solidness of metallic materials & their structures. Many efforts have been made to build up a corrosion inhibition procedure to draw out the life of existing structures and limit consumption harms & damages of corrosion, **Bavarian**, and **Reiner**, **2003**.

To connect to one another they consider variables, a huge number of runs must be done to cover all the conceivable variable blends which are exceptionally uneconomical & troublesome. The routine enhancement optimization methods include modification of one variable & retain the other variables at a fixed level, which allows studying the influence of specific parameter on the whole execution of the procedure. This strategy is tedious, bulky & requires an extensive number of exploratory informational indexes so it is hard to give data about the common associations between parameters in the traditional improvement strategies, **Beg**, **et al.**, **2003**.

Statistical tools and experimental design make it easier to optimize such processes. Taguchi dynamic approach encourages the investigation of communication of a substantial number of factors spread over by variables & their settings with few examinations prompting to impressive time spare and cost for the procedure advancement, **Murthy**, **et al.**, **2012**. Many studies utilized the technique of Taguchi procedure as a gadget to assess the effect of different variables on the characteristics of the framework beneath thought, **Oktem**, **et al.**, **2007**. Yajid et al., have concentrated the inhibitive characteristic of aqueous extracted of Murray Koenig & Cymbopogon citrates aqueous extracted in 0.25-1.0 M sulfuric acid solution utilizing Taguchi technique, **Yajid**, **et al.**, **2013**.

Environmentally friendly corrosion inhibitors are compounds showing good inhibition efficiency and low environmental risk. Among the so-called "green corrosion inhibitors" are organic compounds that act by adsorption on the metal surface, such as caffeine, **Fallavena**, **et al.**, **2006**, succinic acid, **Amin**, **et al.**, **2007**, ascorbic acid, **Ferreira**, **et al.**, **2004** and extracts of natural substances, **Raja**, and **Sethuraman**, **2008**. The inhibitors are organic structures with hetero- atoms such as sulfur, nitrogen and oxygen, **Rani**, and **Basu**, **2012**. The adsorption of these natural atoms on metal-solution interface happen by four mechanisms : (i) electrostatic attraction between the charged particles & charged metal, (ii) interaction of uncharged electron pairs in the particle with metal, (iii) Association of Л-electrons with metal, & (iv) mix of (i) & (iii). Among these natural compounds are fruits and their waste. Fruit and its waste is the opulent provenance of chemicals, for example, minerals, vitamins, organic (natural) acids & phenolic mixes. The pomegranate peel contains various levels of poly phenolic mixes, flavonoid, anthocyanin, tannins and metals, **Orak**, **et al.**, **2012**.

Taguchi introduced the Taguchi technique, **Taguchi**, **et al.**, **2005** to acquire the coveted execution properties by enhancing the outline parameters. In Taguchi method, three-phases, for example, framework plan, parameter outline, & resistance configuration are utilized. Framework configuration comprises of the utilization of logical & designing data required for creating a section. Resilience configuration is utilized to decide & dissect resistances about the ideal blends recommended by parameter plan. Parameter configuration is utilized to acquire the ideal levels of process parameters for building up the quality attributes & to decide the item parameter values relying upon the ideal procedure parameter values. In view of orthogonal arrangements, the number of trials which may build the time & cost can be lessened by utilizing Taguchi system. Taguchi utilizes S/N proportion keeping in mind the end goal to distinguish the quality attributes connected for building outline issues. The S/N proportion attributes can be separated on the premise of three criteria: bring down the better (LB), higher-the-better (HB) and ostensibly the best (NB). The parameter level blend that augments the fitting S/N proportion is the ideal level setting.

Minitab software (Minitab Inc., Minitab Statistical Software, version 17) was utilized for the spontaneous outline of tests in light of Taguchi method. Minitab programming can be utilized to automate $L_9$ empirical orthogonal arrangement with 3 parameters at their three various levels, **Ugur**, **2010**.

$$S/N_j = -10 \log \left[ \frac{1}{N_j} \sum_{a=1}^{N_j} \frac{1}{y_a^2} \right] \qquad (1)$$

Where $N_j$ is the quantity of attempts for trial number $j$, $a$ is the number of experiments & y is the estimation of experimental response $IE_E$(%).

In order to minimize the number of experiments, the experiments are planned against a three level Taguchi's orthogonal arrangements that required 9 tests in total to be achieved. The cause for choosing the three levels of each parameter is that the third level for a parameter simplifies investigation of a quadratic relationship between the response and effect of each parameter, **Bikash**, and **Prasanta**, **2014**.

In element Taguchi methodology, the impact factors are divided into flag parameter, control parameter & commotion parameter. The concentration of $Fe^{2+}$ and roughness of surface are appointed as the commotion parameters. The control component is a variable that is relied upon to give influence on the reaction. The temperature, concentration of aqueous pomegranate peel extracts and contact period were chosen as the control parameters.

Maintaining the level of flag & commotion parameters fixed, the levels of control parameter was varied.

The aim of the present work was to conduct the process optimization and to investigate the effect of the optimal operating conditions, (temperature, inhibitor concentration and contact period) on the inhibition efficiency of aqueous Red Pomegranate Peel (RPP) in the corrosion of mild steel alloy in phosphoric acid solution. The experimental design was done through Taguchi (TED) analysis by using the statistical *MINITAB-17* software for design and analysis of experiments to perform response surface methodology (RSM), then evaluating experimentally the outcome of using optimum operating parameters on the inhibition efficiency of the inhibitor under study.

## 2. EXPERIMENTAL WORK

Throughout the present investigation 2 M phosphoric acid solution was used as the corrosive media. Experiments were conducted under static conditions in the absence and presence of red pomegranate peel (RPP) aqueous extracts.

Inhibitor concentrations were 20 and 40ml of RPP added per litre of 2M $H_3PO_4$ solution. Temperatures used were 30, 40 and 50 °C.

A mild steel coupon (supplied by engineering Lab. and Inspection department, Ministry of Science and Technology) was used as working electrode. The chemical composition of the mild steel coupon is shown in **Table 1**. Test specimens of rectangular shape with 2 cm (width), 4.0 cm (length) and 0.15 cm (thickness) were used for weight loss measurements.

Phosphoric acid (supplied by BDH lab supplies, England), was diluted with distilled water to get the required concentration. The aqueous extract of red pomegranate peels was extracted as follows: red pomegranate was purchased from the local market (Baghdad-Iraq). 250 g of completely dried peel powder was weighed and boiled with double distilled water. The

particle size of peel powder (50 µm). The aqueous extract of pomegranate peel was filtered to remove suspending impurities and made up to 250 ml, **Rani**, and **Basu**, **2012**.
The three parameters at three various levels behold for the empirical outline are described in **Table 2**. Every one of the parameters has been specified with three levels perform in an array of $L_9$ empirical plan as shown in **Table 3**, with eight grades of opportunity (number of tests less one).

Weight loss technique includes samples preparation of coupons were strengthened in a vacuum at 595 °C for 1.25 h & were allowed to cool to 25 °C in the furnace in order to remove mechanical stresses. An annealed sample was abraded in sequence under running tap water using emery paper of grade 120, 220, 320, 400 and 600 respectively, washed with running tap water followed by distilled water, dried on clean tissue, immersed in benzene for 10 seconds and dried with clean tissue, immersed in acetone for 10 seconds and dried with clean tissue and then kept in desiccators over silica gel bed until time of use.
The procedure of weight loss technique includes:

1. The measurements of each specimen were measured with a vernier to the second decimal of millimetre & weighed precisely to the fourth decimal of gram before using.
2. Specimens were totally drenched in 400 ml of corroding medium contained in (500 ml) glass test cell at 2 M $H_3PO_4$, 30, 40 and 50 °C, and 20 and 40 ml/L pomegranate peel aqueous extract concentration for a contact period 1, 2 and 3 h. The test cell was submerged in a water bath to maintain the temperature of the cell at a desired thermal level. Controlling on temperature by using heater plate with the thermostat. The weight loss measurements was carried out in a cell described in **Fig. 1**.

After each test, the sample was washed with running faucet water, scoured with a brush to evacuate corrosion items, washed with faucet water took after by distilled water & dried on a spotless tissue, inundated in benzene, dried, submerged in $C_2H_6O$, dried & left in a desiccators over silica gel for one hour some time as of late weighting.

## 3. RESULTS AND DISCUSSION

### 3.1 Weight Loss Data:

The rate of corrosion of mild steel was resolved in presence and absence of inhibitor at different operating conditions utilizing the formula:

$$C.R = \frac{\Delta m\ (g)}{S\ (m^2)\ x\ t\ (day)} \tag{2}$$

$C.R$: Corrosion rate (g/m$^2$. day) (gmd)
Where $\Delta m$ the loss of mass (g), $S$ *is* the area (m$^2$) & $t$ is the contact time (day).
The percentage inhibition efficiency (IE (%)) was computed as follows, **Alaneme**, and **Olusegun**, **2012**:

$$IE\% = \frac{C.R_0 - C.R}{C.R_0} \times 100 \tag{3}$$

Where $C.R_0$ and $C.R$ are the corrosion rate without and with inhibitor, respectively.

**Table 4** shows a variation of inhibition efficiency and corrosion rate with temperature and contact period in presence and absence of inhibitor. Unmistakably at a certain temperature, the corrosion rate of steel decreases with an increase in the concentration of inhibitor and contact period. Without inhibitor, the corrosion rate increases with a rise in temperature at different contact periods, obeying the Arrhenius equation. It was accounted for

that the rate of corrosion of iron in acidic solutions roughly doubles for each 10 °C ascend in temperature, **Uhlig**, and **Winston**, **2008**. The efficiency of the inhibitor increased with increasing concentration of inhibitor at different periods of contact.
Nonlinear regression for the Data in **Table 4** is done to estimate the coefficients of the proposed model. **Table 5** shows the experimental inhibition efficiency values and the. predicted ones

### 3.2 Response Surface Methodology (RSM):

Response surface methodology is a statistical technique based on simple multiple regressions. By this method, the effect of two or more factors on quality criteria can be investigated and optimized, **Montgomery**, **2005**. Accordingly for the surface technique, there ought to be no less than three levels for each variable. The effect of component values that are not really tried utilizing less exploratory blends can be estimated, **Neseli**, **et al.**, **2011**. It was accounted for that temperature, inhibitor concentration and exposure time fundamentally affected the rate of corrosion of mild steel alloy in 1 M $H_3PO_4$ solution using natural apricots juice as the inhibitor, **Yaro**, **et al.**, **2013**. After the ID of the vital parameters, the test range was classified in Table 2. A Taguchi experimental measurable outline with three components & three levels was utilized to understand second request polynomial model & exponential model was utilized to represent these parameters. These models consider the impact of temperature, inhibitor concentration, contact period & the cooperation of them on inhibition efficiency. Minitab software (Minitab Inc., Minitab Statistical Software, version 17) was utilized to produce an outline of test appeared in Table 3, which will be utilized for both model's examination & streamlining. In most RSM issues, there is a useful connection amongst reactions & free factors & this formula can be clarified utilizing the model, **Salam, et al.**, **2014**. The primary & intuitive values between the free factors & response (predicted inhibition efficiency$IE_P$ (%)) were assessed by producing the first second order model in the form represented in Eq. (4).

$$IE_P\,(\%) = b_0 + b_1T + b_2C + b_3t + b_{11}T^2 + b_{22}C^2 + b_{33}t^2 + b_{12}TC + b_{13}Tt + b_{23}Ct \quad (4)$$

One more second exponential model was additionally proposed, which depended on the accompanying presumptions:

i. The rate of corrosion can be identified with temperature by Arrhenius equation, which gives a sign that the corrosion rate depends exponentially on temperature:

$$C.R \propto Exp\,[\frac{-1}{T}]$$

ii. The corrosion inhibition increases as the inhibitor concentration increases and contact period decreased, so;

$$IE_P\,(\%) \propto \frac{C^{b_5}}{t^{b_6}}$$

These two assumption respect propose the second exponential model:

$$IE_P\,(\%) = b_4C^{b_5}t^{-b_6}Exp\left[\frac{-1}{T^{-b_7}}\right] \quad (5)$$

Where: $IE_P$ (%), $T$, $C$ and $t$ are predicted corrosion inhibition percentage, temperature (°C), inhibitor concentration (ml/L) and contact period (h) respectively. Nonlinear minimum squares regression investigation in view *Rosenbrock and Quasi-Newton* estimation method employing the Minitab software can be used for estimation of coefficients $b_0$, $b_i$, $b_{ij}$ and $b_{ii}$, the results showed in **Table 6**. And creating the accompanying condition for mild steel alloy with 1.000 correlation coefficient for first model Eq. (4):

$$IE_P\,(\%) = 18.45 - 3.34\,T + 1.71\,C + 69.26\,t + 0.05\,T^2 - 0.06\,C^2 - 7.03\,t^2 + 0.05\,T \times C - 0.96\,T \times t + 0.26\,C \times t \qquad (6)$$

The regression results of coefficients$b_4$, $b_5$, $b_6$ and $b_7$ for another estimated model Eq. (5), showed in **Table 7**.

And producing another evaluated display Eq. (5) with 0.992 coefficient of correlation:

$$IE_P\,(\%) = 13.965\,C^{0.474} t^{0.106} Exp[-T^{-8.784}]$$

**Fig. 2** shows the performance plot of percentage predicted inhibition efficiency by Eq. (4) and Eq. (5) versus experimental values. The figure demonstrates that the both models represent the inhibition efficiency information with coefficients of high correlation.

### 3.3 Analysis of variance (ANOVA)

The measurable system known as an investigation of variance (ANOVA) can be to determine the significance of differences that exist among the methods for a few gatherings of observations. Two-way ANOVA has been utilized as a part of a request to decide the impact of temperature, the concentration of inhibitor and contact period on inhibition efficiency.

**Table 8** gives the analysis of variance ANOVA for inhibition efficiency for mild steel alloy. Fisher's F-test at 95% level of confidence can also be used to determine which process factors have a significant effect on performance, **Jeff Wu**, and **Michael**, **2009**. When F value is large, it means that the change of the process factor has a significant effect on characteristics of performance. It can be observed from **Table 8** that inhibitor concentration has large F value which suggests that the inhibitor concentration is the major factor which affects the efficiency of inhibition at 98.09 % contribution.

The accuracy of an empirical model can also be tested by means of statistical factors, such as the correlation coefficient. The correlation coefficient ($R^2$) 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: $R^2$ = 1.000, which suggests a full compatibility.

### 3.4 Experimental Variables & their Main Influences - S/N proportion Analysis

**Fig.3** shows the influence of input variables, for example, temperature, the concentration of inhibitor & contact period on inhibition efficiency. It can be observed that efficiency increases with increase in concentration and contact period and generally decreases with increase in temperature. **Fig. 4** demonstrates the effect of every individual component on the efficiency of inhibition as far as S/N proportions. Increment the concentration of element RPP & contact period has brought about the increment in inhibition behaviour mirroring the high positive S/N proportions for level 2 and 3. The maximum inhibition in temperature is obtained at level 1 and increased in temperature has also decreased the inhibition efficiency with further increment in variable levels efficiency of inhibition increased. The efficiency of inhibition diminishes with increment in temperature, **Bodude**, and **Sanni**, **2014**.

### 3.5 Optimization of Maximum Inhibition Efficiency

In **Table 9**. It can be seen from the table that RPP have a huge part in the corrosion inhibition than the other chose components. The consequences of the S/N & examination of variance unmistakably demonstrate that the level of concentration of the inhibitor builds; the S/N proportion additionally increments. The bigger S/N proportion as the main effect is watched for RPP in level 3 as shown in **Fig. 4**. Temperature and contact period are the other basic components which influence the utilization method & subsequently the efficiencies of inhibition. RPP concentration exhibits beneficial outcome on the considered parts. The effect of the factors main, square and interaction, levels 1.98, 2.93 and 2.87 for temperature, RPP concentration and contact period, respectively, are the optimum conditions realizing higher percentage inhibition for the inspected system. For the RPP concentration level 2.93 in coded factor, i.e., 38.29 ml/L in real factor produce better results.

According to Eq. (6), using *POLYMATH* software version 4.02 in terms of maximum percentage inhibition efficiency, the optimum values were obtained. The optimum values of the studied independent factors in real and coded form are listed in **Table 9** below for mild steel alloy. An approval trial was led by the predetermined upgraded values & the outcomes were utilized to compute the response. The experimental value of the inhibition efficiency at the optimum conditions was comparable with the theoretical value and the result was closed are listed in **Table 10**.

### 3.6 Effect of Process Factors on Inhibition Efficiency

The parametric influences of corrosion inhibition process factors on inhibition efficiency at the optimum circumstances are presented as response surface plots and can also be interpreted with the contour plot diagram and these are given in **Figs. 5** through **7**.

To show the combined influence of any two inhibition factors on the inhibition efficiency at the optimum value of the third factor. For example, the effect of temperature and concentration of inhibitor (aqueous pomegranate peel extract) on inhibition efficiency is shown in **Fig. 5** A response surface plot while keeping constant contact period at the optimum value 2.95 h. From **Fig. 5** describes the surface behaviour of up growth of the inhibition efficiency as a function of the temperature and the concentration of inhibitor and demonstrate the collaboration between shifting estimations of temperature & concentration at an optimum estimation of the contact period. Maximum inhibition efficiency of 87.08 % was observed when concentration of inhibitor was 2.93 in coded variable, i.e., 38.29 ml/L in real variable at optimum circumstance at 39.66 °C. There was a slight decrease in inhibition efficiency when temperature expanded to 39.66 °C while sharp increase when concentration of inhibitor increased to 38.29 ml/L. Considering simultaneous effects of temperature and inhibitor concentration is presented in **Fig. 5**. Contour plot shows the variation of corrosion inhibition efficiency with temperature and concentration of RPP. This plot reveals that concentration of RPP has a greater effect on corrosion inhibition process. The better inhibition is achieved in minimum temperature (30 °C) when the concentration of RPP was increased. This is also in close agreement with a previous work, **Yaro**, **et al.**, **2013**.

**Fig. 6** describes the relationship between different values of temperature and contact period at optimum inhibitor concentration. Optimum inhibition efficiency of 87.08 % was recorded at contact period 2.95 h and temperature 39.66 °C, and inhibition efficiency was expanded as the temperature was decreased. Even though inhibition efficiency was upper in high contact period however it didn't avert dynamic increment in inhibition efficiency as the temperature was decreased. Analysis of inhibition efficiency as effects of interaction between temperature and contact time is shown in **Fig. 6** contour plot, demonstrates the variety of efficiency of inhibition with contact period and temperature. It was seen the higher contact period and lower temperature gave better performance in terms of inhibition efficiency at the optimum condition of RPP concentration 38.29 ml/L and this in agreement with another study, **Banerjee**, **et al.**, **2012**.

Surface conduct of various estimations of the concentration of inhibitor & contact period at the optimum value 39.66 °C in **Fig. 7**. Optimal inhibition efficiency of 87.08 % was experienced at optimum contact period (2.95 h) with optimum RPP concentration (38.29 ml/L). This estimation diminished with decreased in the estimation of RPP concentration to 20 ml/L, beyond 20 ml/L there was decreased in inhibition efficiency to zero at absence inhibitor. **Fig. 7** Contour plot demonstrates the variety of efficiency of inhibition with a concentration of RPP & contact period. At higher values of RPP concentration and contact period responsible for corrosion inhibition and depicts a methodology polynomial surface response relating to the effect of RPP concentration and contact period on inhibition efficiency. Contact period increased inhibition efficiency from 12-72 % along the contact period setting (1-3 h). The concentration of RPP from 0-40 ml/L has increased inhibition efficiency from 12-72 %. This is also in close agreement with the research done by, **Al-Moubaraki**, **et al.**, **2015**.

## 4. CONCLUSION

The multivariable regression model of the impact of temperature, the concentration of inhibitor & contact period on the inhibition efficiency of mild steel alloy was effectively considered with least number of trial runs utilizing reaction surface methodology (RSM) gives Eq. (6) depicts the behaviour of the inhibition process with high accuracy (correlation coefficient = 1.000). The (RSM) of three levels, Taguchi experimental design (TED) was shown to be very useful for the effect of process parameters as the main, quadratic and combined effect on the response. From the analysis of variance it is experienced that the three parameters effect on the inhibition efficiency in following order:

Red pomegranate peel extract concentration > Temperature > Contact period.

The optimization result shows optimum circumstances as predicted from Eq. (6) is 39.66 °C temperature of acidic media, 38.29 ml/L inhibitor concentration and 2.95 h of the contact period for mild steel alloy. Aqueous RPP extract is effective acts as a green corrosion inhibitor and provides good inhibition of corrosion of mild steel alloy in 2 M $H_3PO_4$ solution with maximum RSM predicted inhibition efficiency 87.08 % and experimentally 88.64 % under the mentioned optimum circumstances. There is a close agreement between the predicted and experimental results of inhibition efficiency at optimum circumstances which portrays the precision of the statistical model & can be utilized in the test conditions. The inhibition action of RPP extract increases with the increase of inhibitor concentration and contact period and decreases with increase in temperature.

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**Table 1.** Average chemical composition of mild steel coupon (% by weight).

| Component | Weight % |
|---|---|
| C | 0.069 |
| Si | 0.009 |
| Ni | 0.026 |
| Mn | 0.441 |
| S | 0.005 |
| Iron | Remainder |

**Table 2.** Experimental range and levels of control independent Factors for mild steel corrosion inhibition.

| Control independent factor | Symbol | Unit | Range and Level | | |
|---|---|---|---|---|---|
| | | | Level 1 (Low) | Level 2 (Middle) | Level 3 (High) |
| Temperature | $T$ | (°C) | 30 | 40 | 50 |
| Inhibitor Concentration | $C$ | (ml/L) | 0 | 20 | 40 |
| Contact Period | $t$ | (h) | 1 | 2 | 3 |

**Table 3.** Sequence of experiments according to Taguchi experimental design of the three factors.

| Exp. No. | Coded Factor | | | Real Factor | | |
|---|---|---|---|---|---|---|
| | $T$ | $C$ | $t$ | Temp. (°C) | Inhibitor Conc. (ml/L) | Contact Period (h) |
| 1 | 1 | 1 | 1 | 30 | 0 | 1 |
| 2 | 1 | 2 | 2 | 30 | 20 | 2 |
| 3 | 1 | 3 | 3 | 30 | 40 | 3 |
| 4 | 2 | 1 | 3 | 40 | 0 | 3 |
| 5 | 2 | 2 | 1 | 40 | 20 | 1 |
| 6 | 2 | 3 | 2 | 40 | 40 | 2 |
| 7 | 3 | 1 | 2 | 50 | 0 | 2 |
| 8 | 3 | 2 | 3 | 50 | 20 | 3 |
| 9 | 3 | 3 | 1 | 50 | 40 | 1 |

**Table 4.** Experimental layout using an $L_9$ orthogonal arrangement.

| Exp. No. | Factor's Level | | | $C.R_{uninhibit}$ (gmd) | $C.R_{inhibit}$ (gmd) | $IE_E$ (%) |
|---|---|---|---|---|---|---|
| | *T* | *C* | *t* | | | |
| | Temperature (°C) | Inhibitor Concentration (ml/L) | Contact Period (h) | | | |
| 1 | 1 | 1 | 1 | 44.29 | 44.29 | 0 |
| 2 | 1 | 2 | 2 | 50.19 | 14.61 | 70.88 |
| 3 | 1 | 3 | 3 | 133.49 | 11.25 | 91.57 |
| 4 | 2 | 1 | 3 | 74.39 | 74.39 | 0 |
| 5 | 2 | 2 | 1 | 50.95 | 25.11 | 50.72 |
| 6 | 2 | 3 | 2 | 77.23 | 15.05 | 80.51 |
| 7 | 3 | 1 | 2 | 76.05 | 76.05 | 0 |
| 8 | 3 | 2 | 3 | 79.03 | 29.20 | 63.05 |
| 9 | 3 | 3 | 1 | 130.73 | 19.21 | 85.31 |

**Table 5.** $L_9$ orthogonal arrangement of experiments with the observed, predicted values and experimental error for the inhibition efficiency.

| Exp. No. | Coded Factor | | | Real Factor | | | Exp. Inhibition Efficiency | Predicted Inhibition Efficiency | Corresponding Residual |
|---|---|---|---|---|---|---|---|---|---|
| | $T$ | $C$ | $t$ | Temp. (°C) | Inhibitor Conc. (ml/L) | Contact Period (h) | $IE_E$ (%) | $IE_P$ (%) | $e_i = IE_E - IE_P$ |
| 1 | 1 | 1 | 1 | 30 | 0 | 1 | 0 | 0 | 0.00 |
| 2 | 1 | 2 | 2 | 30 | 20 | 2 | 70.88 | 70.88 | 0.00 |
| 3 | 1 | 3 | 3 | 30 | 40 | 3 | 91.57 | 91.57 | 0.00 |
| 4 | 2 | 1 | 3 | 40 | 0 | 3 | 0 | 0 | 0.00 |
| 5 | 2 | 2 | 1 | 40 | 20 | 1 | 50.72 | 50.72 | 0.00 |
| 6 | 2 | 3 | 2 | 40 | 40 | 2 | 80.51 | 80.51 | 0.00 |
| 7 | 3 | 1 | 2 | 50 | 0 | 2 | 0 | 0 | 0.00 |
| 8 | 3 | 2 | 3 | 50 | 20 | 3 | 63.05 | 63.05 | 0.00 |
| 9 | 3 | 3 | 1 | 50 | 40 | 1 | 85.31 | 85.31 | 0.00 |

**Table 6.** The regression analysis coefficient values of the predicted correlation (polynomial model).

| Coeff. | $b_0$ | $b_1$ | $b_2$ | $b_3$ | $b_{11}$ | $b_{22}$ | $b_{33}$ | $b_{12}$ | $b_{13}$ | $b_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|
| Value | 18.45 | -3.34 | 1.71 | 69.26 | 0.05 | -0.06 | -7.03 | 0.05 | -0.96 | 0.26 |
| Correlation Coefficient ($R^2$) | | 1.000 | | | Proportion of Variance | | | 1.000 | | |
| Final Value of Loss Function | | 0.000 | | | | | | | | |

**Table 7.** The regression analysis coefficient values of the suggested correlation (exponential model).

| Coefficient | $b_4$ | $b_5$ | $b_6$ | | $b_7$ |
|---|---|---|---|---|---|
| Value | 13.965 | 0.474 | -0.106 | | -8.784 |
| Correlation Coefficient ($R^2$) | | | 0.992 | Proportion of Variance | 0.983 |
| Final Value of Loss Function | | | 205.081 | | |

**Table 8.** Numerical results of analysis of variance (ANOVA).

| Control Independent Factors | Degree of Freedom (DF) | Sum of Squares (Adj.SS) | Mean Squares (Adj.MS) | F-ratio (F) | Probability Percent P (%) | (%) Contribution |
|---|---|---|---|---|---|---|
| Temperature | 2 | 162.96 | 81.48 | 4.139 | 19.5 | 1.36 |
| RPP Conc. | 2 | 11737.37 | 5869.69 | 298.12 | 0.3 | 98.09 |
| Contact Period | 2 | 65.77 | 32.89 | 1.671 | 37.4 | 0.55 |
| Total | 6 | 11966.1 | | | 57.2 | 100 |

**Table 9.** Optimum conditions of the control independent factors.

| Control Independent Factor | Optimum Conditions (mild steel alloy) | |
|---|---|---|
| | Code | Real |
| $T$. Temperature (°C) | 1.98 | 39.66 |
| $C$. Inhibitor Concentration (ml/L) | 2.93 | 38.29 |
| $t$. Contact Period (h) | 2.87 | 2.95 |
| RSM Predicted Inhibition Efficiency, $IE_P$ (%) | 87.08 | |

**Table 10.** Experimental validation at objective optimization adjusting.

| Response | Objective Optimization of Percentage Inhibition Efficiency | | Percentage of Prediction Error |
|---|---|---|---|
| | Result of RSM Predicted at Optimum Conditions, $IE_P$ (%) | Result of Experiment at Optimum Conditions, $IE_{O.C}$ (%) | |
| Inhibition Efficiency | 87.08 | 88.64 | 1.759 % |

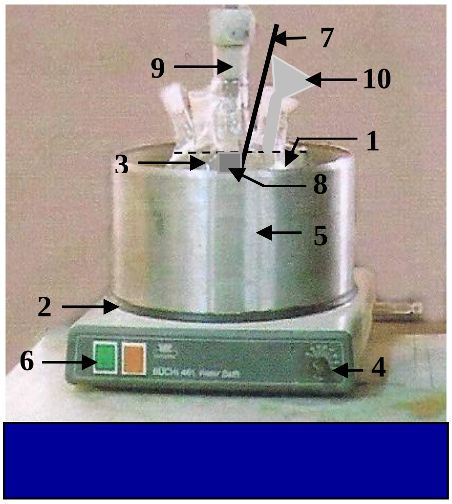



**Figure 1.** Experimental set-up for weight loss investigation.

| | | | |
|---|---|---|---|
| 1. | Corrosive Solution | 6. | Switch |
| 2. | Heater and Controller | 7. | Thermometer |
| 3. | Corrosion Cell | 8. | Sample |
| 4. | Thermostat | 9. | Teflon Rod |
| 5. | Water Bath | 10. | Glass Funnel |

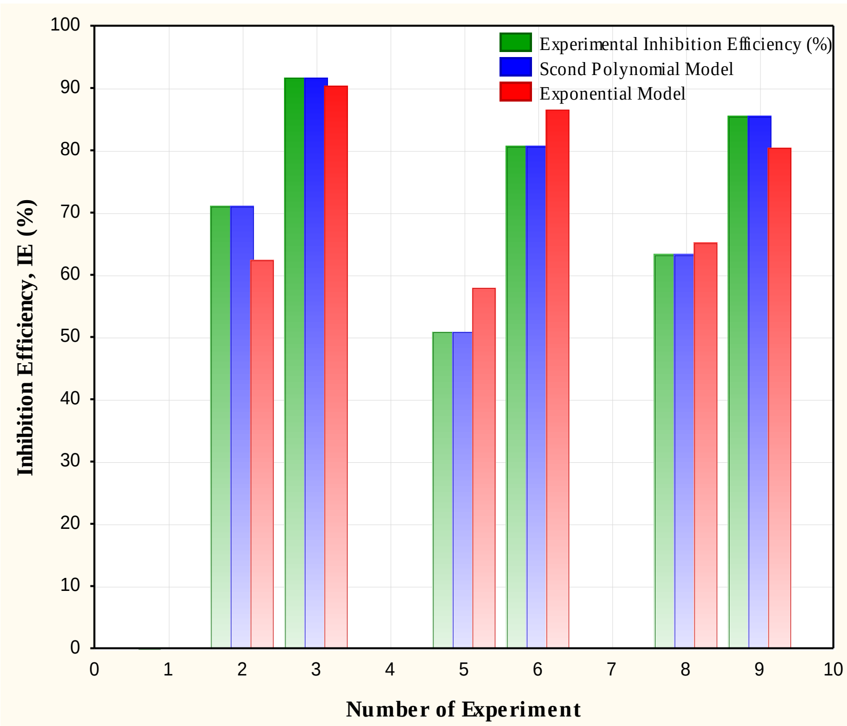



**Figure 2.** The performance plot of experimental and predicted values.

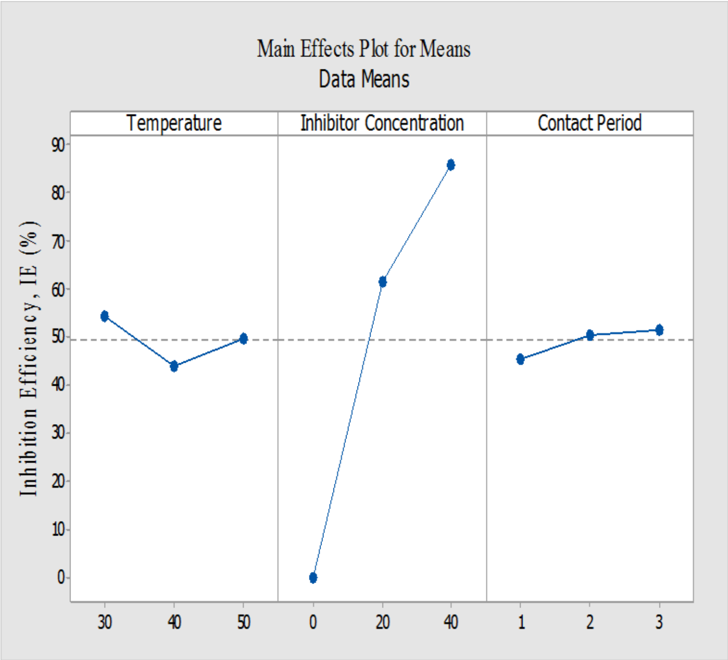



**Figure 3.** Main effect plot.

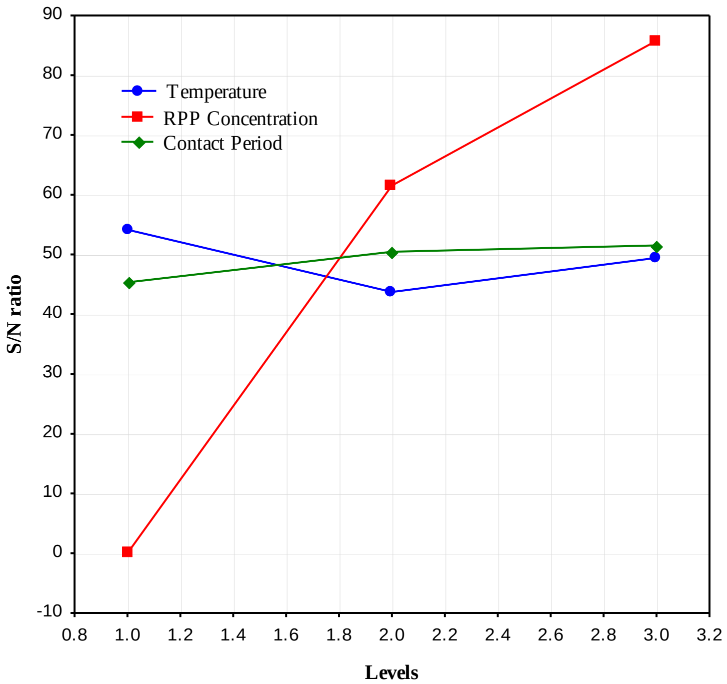



**Figure 4.** Main effects of control independent factors at their different levels.

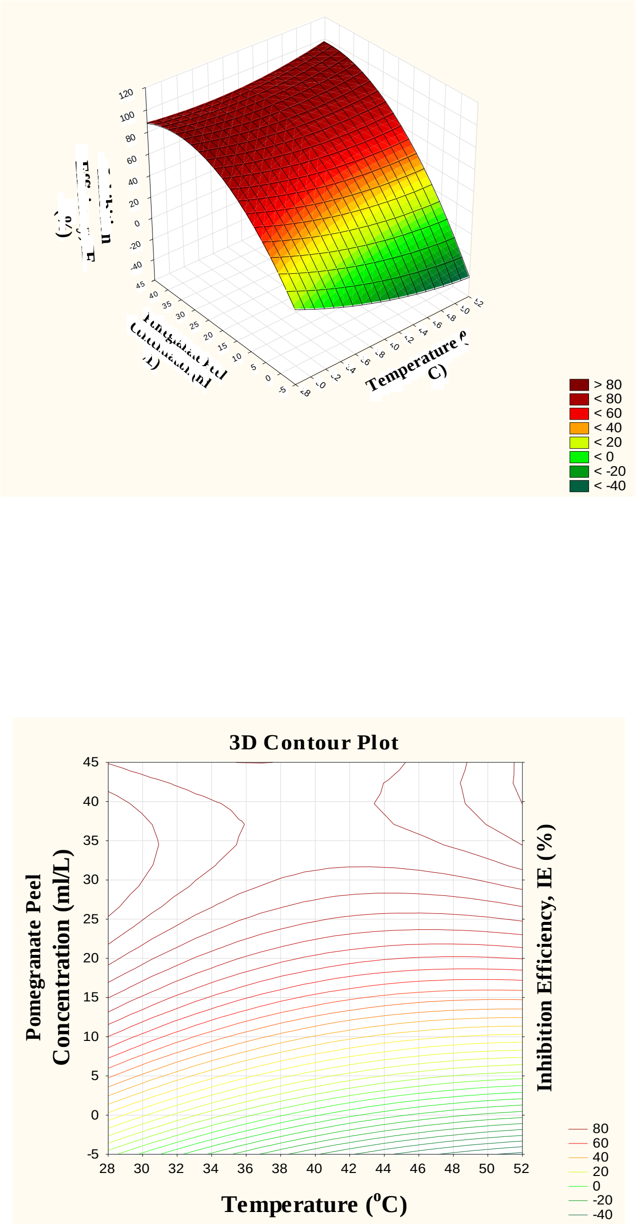



**Figure 5.** Response surface contour for interaction on mild steel corrosion inhibition between temperature and pomegranate peel concentration at the optimum value (2.95 h).

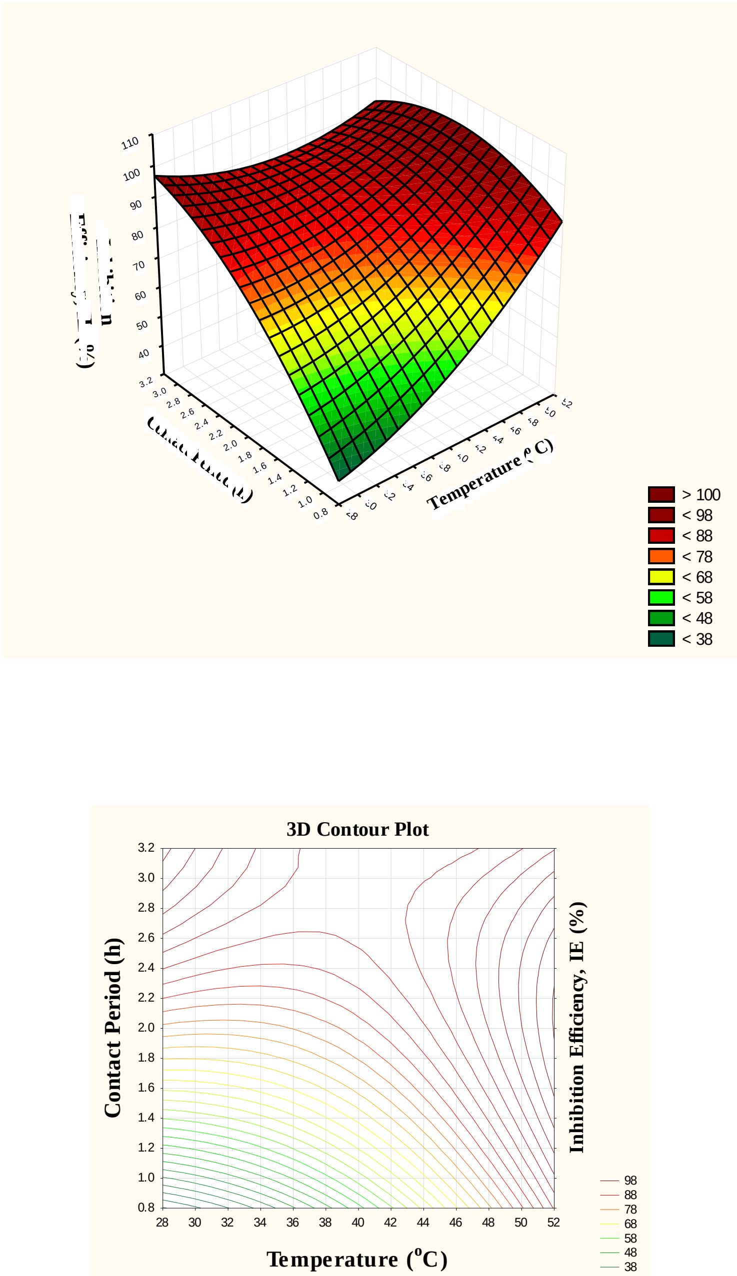
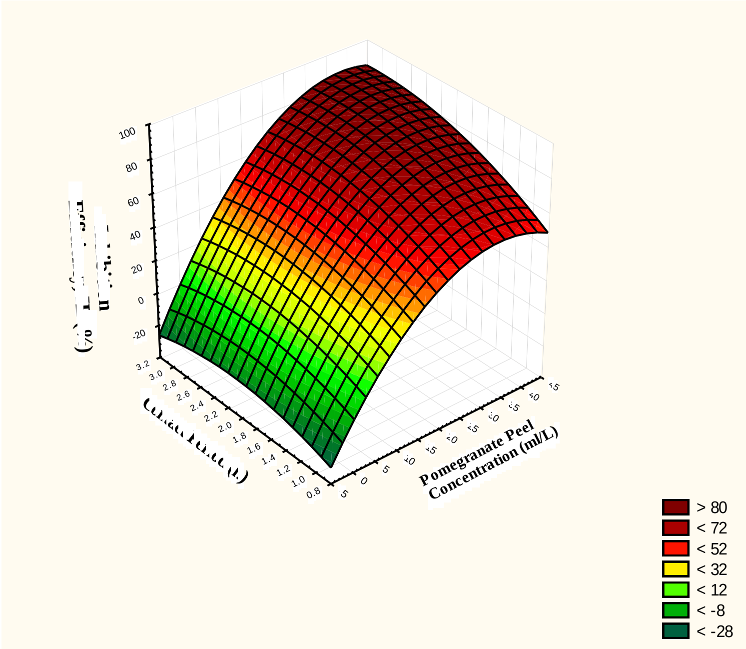



**Figure 6.** Response surface contour for interaction on mild steel corrosion inhibition between temperature and contact period at the optimum value (38.29 ml/L).



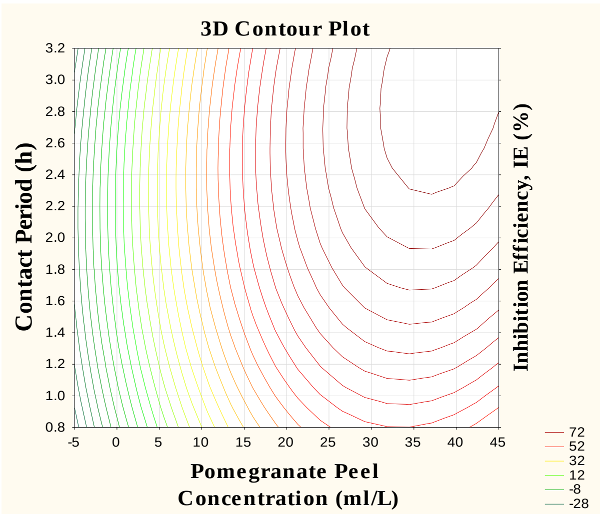




**Figure 7.** Response surface contour for interaction on mild steel corrosion inhibition between pomegranate peel concentration and contact period at the optimum value (39.66 °C).