On the use of factorial experiments for optimizing inhibition effect of acid extract of *Gnetum Africana* on copper corrosion

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Abstract

The inhibition of copper corrosion by acid extract of *Gnetum Africana* using weight loss method under various independent variables of time, inhibitor concentration and temperature was studied. The data used was retrieved from a three-level factorial experiment by Nkuzinna *et al*. (2014) and reanalyzed. The re-analysis was necessary to provide an alternative model for weight loss due to corrosion of copper in the acid extract of *Gnetum Africana*. The original three-level factorial design was employed to fit a model describing the weight loss due to corrosion. Equally, a two-level factorial design and Box–Behnken design were alternatively considered for the design constructions, analysis, and modeling the process of the original data. The optimum factor settings for corrosion inhibition were determined to be 24 h, 0.003 g/L and 303 K for time, inhibitor concentration and temperature respectively. It was concluded that the Box–Behnken design was the most efficient, followed by the two-level factorial design, while the three-level factorial design was the least efficient in predicting the weight loss. Indeed, this is a great improvement over the three-level factorial experiment earlier used by Nkuzinna *et al*. (2014) in relation to resources, time and above all the statistical efficiency, precision and reliability of the model.

Keywords: *designs of experimental, factorial designs, Box–Behnken design, optimization, corrosion, Gnetum Africana, corrosion inhibition.*

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1. Introduction

Corrosion is the destructive attack of a material by chemical reaction with its environment. Corrosion is an electrochemical reaction, *i.e*. it comprises both cathodic and anodic half reactions:

$$\text{Anodic reaction: } Fe_{(s)} \rightarrow Fe^{3+}_{(aq)} + 3e^{-}$$

$$\text{Cathodic reaction: } 2H^{+}_{(aq)} + 2e^{-} \rightarrow H_{2(g)}$$

$$\tfrac{1}{2}O_{2(g)} + H_2O_{(l)} + 2e \rightarrow 2OH^{-}_{(aq)}$$

The serious consequences of the corrosion process have become a problem of worldwide significance. In addition to our everyday encounters with this form of degradation, corrosion causes plant shutdowns, waste of valuable resources, loss or contamination of product, reduction in efficiency, costly maintenance, and expensive over-design; it also jeopardizes safety and inhibits technological progress (Roberge, 2000).

Copper and its alloys have good corrosion resistance in water and have excellent heat conductivity, but they corrode easily in acid solutions. They are broadly used in heating systems and condensers. However, these systems should be regularly cleaned due to inlays of carbonates and oxides (which can) diminish their heating transmission. Diluted acids are normally used to clean these surfaces. For preventing of copper corrosion, additives such as inhibitors are generally added to acid (Ravari *et al*., 2009).

Corrosion inhibitors are defined as chemical substances that, when added in small concentration to an environment, effectively decreases the corrosion rate of metals or alloys (Roberge, 2000). The use of corrosion inhibitors has considerably increased in recent years due to increased awareness of corrosion worldwide. Inhibitors play an important role in controlling corrosion of metals (Quraishi *et al*., 2001). Various organic and inorganic compound have been used as corrosion inhibitors in aggressive environment, *e.g*. pyridazine derivatives (Mashuga *et al*., 2017); quinoxaline derivatives (Olasunkanmi *et al*., 2016) and selected dyes (Peme *et al*., 2015). Various Schiffbase (3-acetyl-4-hydroxy-6-methyl-(2*H*)pyran-2-one and 2,2′-(ethylenedioxy)diethylamine) as corrosion inhibitors of mild steel in acidic medium have been reported (Jonnie *et al*., 2015). Phthalocyanines derivatives containing heteroatoms such as O, N, S, and P are found to have higher basicity and electron donating ability and have also been used as corrosion inhibitors and some ionic liquids (Yesudass *et al*., 2015).

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 importance (Nkuzinna *et al*., 2014). Plants are sources of naturally occurring compounds, some with complex molecular structures and having different chemical, biological, and physical properties. The naturally occurring compounds are mostly used because they are environmentally acceptable, cost effective, and have abundant availability. These advantages are the reasons for the use of extracts of plants and their products as corrosion inhibitors for metals and alloys under different environments (Patni *et al*., 2013). Among natural plants extract as corrosion inhibition of mild steel and aluminum alloy in acidic medium are extracts from *Ficus asperifolia* (Fadare *et al*., 2016) and *Newbouldia leavis* (Lebe *et al*., 2011). Similarly, the inhibiting effect of *Chenopodium* extract against the copper corrosion in 1 M HNO_3 was studied by weight loss, potentiodynamic polarization, electrochemical impedance spectroscopy (EIS) and electrochemical frequency modulation (EFM) techniques (Motawea, 2019). Inhibitory effect of African Pumpkin (*Momordica balsamina Linn*.) leaf extract on copper corrosion in acidic media was also studied and reported (Maibulangu *et al*., 2017).

With concerns about the harmful effects of inorganic inhibitors on the environment, several organic corrosion inhibitors have been developed to avoid those problems. The extract of *Gnetum Africana* is one of such inhibitors. *Gnetum Africana* (also called African Jointfir) is a forest vine that grows abundantly in Africa, South America, and tropical and subtropical Asia. Its leaves are widely used as an ingredient in soups and stews and they are much in demand for their nutritional and therapeutic properties (Ali *et al*., 2015). In recent years, the use of *Gnetum Africana* extract as a corrosion inhibitor has been explored. It has been found to reduce corrosion significantly in carbon, mild steel and stainless steel (Nnanna *et al*., 2013; Obiukwu *et al*., 2015; Obiukwu *et al*., 2016). The phenolic extract has also been shown to inhibit corrosion of copper in ammonium hydroxide (Aliyu and Onyedikachi, 2014). The rate of corrosion of a metal is determined by several factors such as temperature and time. It is therefore imperative to simplify, model and optimize the corrosion inhibition process of *Gnetum Africana* extract on copper.

In recent time, a full-factorial experiment studying the inhibition effect of acid extract of *Gnetum Africana* was conducted (Nkuzinna *et al*., 2014), for the experiment, pure copper sheets (99.9%) were employed and HNO_3 was used to extract the inhibitor from *Gnetum Africana* leaves. The weight losses due to corrosion were recorded under three different factors (inhibitor concentration, time and temperature), each factor was measured at three levels, making it a 3-level full factorial design with 27 runs.

The study presented a polynomial model describing the process in terms of denoted factors. The regression model having an R^2 value of 0.8227 is expressed in Equation 1.

$$Y_{\text{weight loss}} = 2.16 - 1.04X_1 - 0.17X_1^2 + 0.33X_2 - 0.12X_2^2 - 0.64X_3 - 0.22X_3^2 + 0.27X_1X_2 + 0.13X_1^2X_3 + 0.068X_1X_3^2 + 0.11X_1^2X_3^2 \quad (1)$$

Where X_1, X_2 and X_3 are time, inhibitor concentration and temperature, respectively and the optimal (minimum) factor settings obtained are 24 h, 0.003 g/L, and 303 K for time, inhibitor concentration and temperature respectively. Importantly, however, we found that using the statistical model in Equation 1, the predicted weight loss at those settings is 3.30 g, which is much higher than the experimental weight loss value of 0.62 g in Run 7 of Table 2.

This paper aims to reanalyze the data in order to provide alternative models with better optimal setting as an improvement over the model provided by (Nkuzinna *et al*., 2014). In addition, to also investigate and identify the most efficient alternative designs (two-level factorial design, three-level factorial design, and Box–Behnken design), in studying the inhibition effect of acid extract of *Gnetum Africana* on copper corrosion.

2. Overview of the original 3^3 factorial experiment

In 1937, Frank Yates wrote an article titled, the design and analysis of factorial experiments and universally known as "TC35" which designed and analyzed factorial experiments with regard to main and interaction effects was introduced. Factorial designs are frequently used in experiments involving several factors where it is necessary to investigate the joint effect of the factors on a response (Montgomery, 2013). The D-optimal minimax criterion with a suitable tool that minimizes the variance, covariance and bias of the estimations of the optimal fractional factorial split plot designs in which the efficiency was felt when compared with both A- and D-optimal designs was introduced (Saka, *et al.*, 2019).

The three factors considering in this study are time, inhibitor concentration and temperature with their respective levels are presented in Table 1 while Table 2 shows the full experimental runs in a standard form for the original 3^3 factorial design with actual measurements obtained for the factors and the responses.

Table 1. Experimental factors and their respective levels.

Factors	Lower limit (–1)	Moderate (0)	Upper limit (+1)
Time (h)	24	72	120
Inhibitor Concentration (g/L)	0.001	0.002	0.003
Temperature (K)	303	318	333

Table 2. The 3^3 full factorial design showing runs in standard form and responses.

Runs	Time X_1 (h)	Inhibitor concentration X_2 (g/L)	Temperature X_3 (K)	Weight loss (g)
1	24	0.001	303	0.90
2	24	0.001	318	0.96
3	24	0.001	333	1.86
4	24	0.002	303	0.70
5	24	0.002	318	1.09
6	24	0.002	333	1.72
7	24	0.003	303	0.62
8	24	0.003	318	0.85
9	24	0.003	333	1.36
10	72	0.001	303	1.68
11	72	0.001	318	2.02
12	72	0.001	333	3.54

Runs	Time X_1 (h)	Inhibitor concentration X_2 (g/L)	Temperature X_3 (K)	Weight loss (g)
13	72	0.002	303	1.40
14	72	0.002	318	1.95
15	72	0.002	333	1.40
16	72	0.003	303	1.32
17	72	0.003	318	1.64
18	72	0.003	333	2.88
19	120	0.001	303	2.84
20	120	0.001	318	3.18
21	120	0.001	333	5.37
22	120	0.002	303	2.18
23	120	0.002	318	3.07
24	120	0.002	333	4.84
25	120	0.003	303	1.96
26	120	0.003	318	2.67
27	120	0.003	333	4.20

*This is the original data given by Nkuzinna *et al*., 2014.

2.1. Results and discussions of the original 3^3 factorial experiment

Here, the results of the analysis carried out on the dataset presented in Table 2 are discussed.

Table 3. Analysis of variance of original 3^3 factorial experiment.

Source	Sum of squares	DF	Mean square	*F*-value	*p*-values
Model	36.167	4	9.042	46.526	<0.0001
Error	4.275	22	0.194		
Total	40.442	26			

Table 4. Parameter estimates of the original 3^3 factorial experiment.

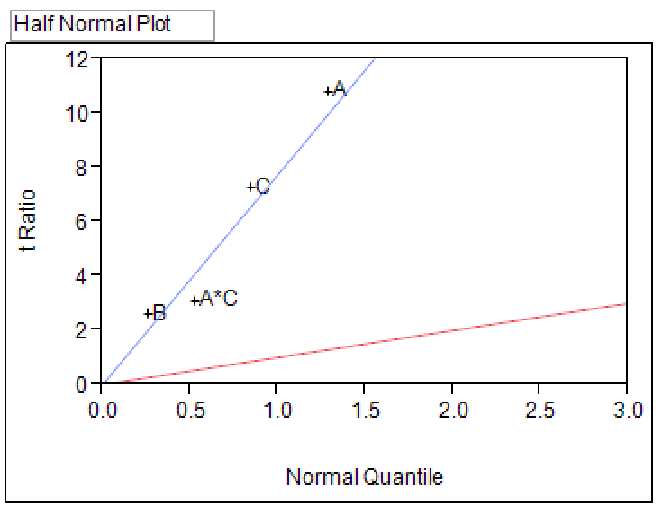
Term	Estimate	*t*-value	*p*-values
Intercept	2.1555556	25.41	<0.0001*
Time	1.125	10.83	<0.0001*
Conc.	–0.269444	–2.59	0.0166*

Term	**Estimate**	***t*-value**	***p*-values**
Temp	0.7538889	7.26	<0.0001*
Time*temp	0.3925	3.08	0.0054*

From Table 3, the analysis of variance confirms that there is sufficient evidence to reject the null hypothesis (*F*-value of 46.53 and a *p*-value of 0.0001), meaning that at least one of the model parameters is significant and useful in explaining variability in the weight loss. Also, the coefficient of determination, R^2, and adjusted R^2 of the model are 0.894 and 0.875 respectively, which is an indication that an approximately 90% of the variability in the weight loss is explained or accounted for by the model in Equation 2.

The parameter estimates of the treatment effects: time, temperature, time·temperature and inhibitor concentration that are significant and sensitive to the weight loss due to corrosion are presented in the Table 4 above. This is eventually represented as a model in Equation 2, which is clearly supported by the half normal plot in Figure 1.

$$Y_{\text{weight loss}} = 2.1556 + 1.125X_1 - 0.2694X_2 + 0.7539X_3 + 0.3925X_1X_3 \quad (2)$$



A: Time, B: Inhibitor concentration and C: Temperature

Figure 1. Half-normal plot on 3^3 factorial experiments for the independent variables.

2.2. Optimal evaluation of 3^3 factorial experiments

The Figures 2 and 3 are used to evaluate the model obtained in Equation 2 and also to obtain the optimal values. As such, the optimum values of the factors that minimize weight loss due to corrosion are shown in Table 5.

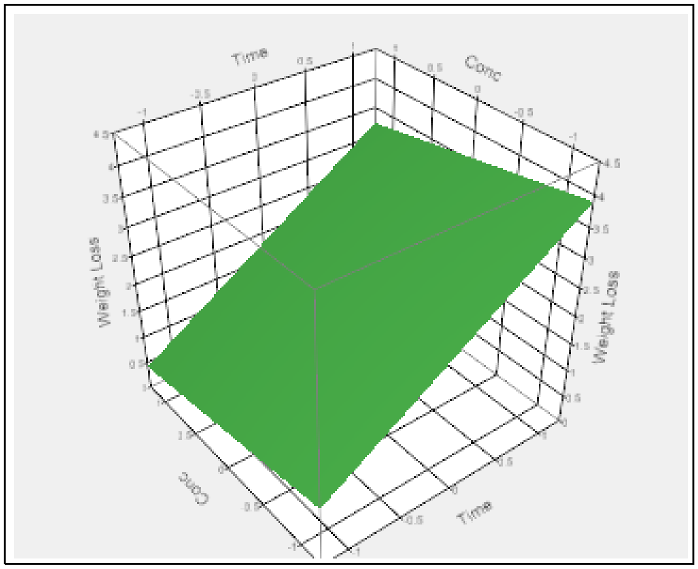


Figure 2. Response surface plot of time against inhibitor concentration.

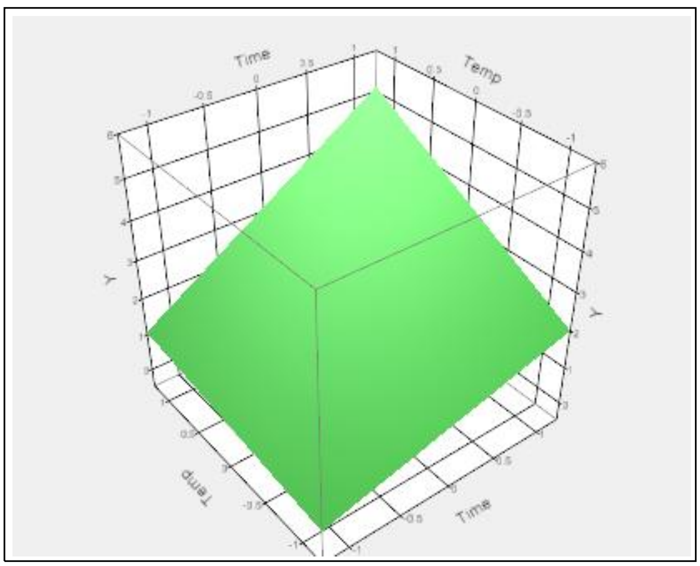


Figure 3. Response surface plot of time against temperature.

Table 5. Optimum values of 3^3 factorial experiments.

Parameters	Optimum values
Weight loss (g)	0.400
Time (h)	24
Inhibitor concentration (g/L)	0.003
Temperature (K)	303

3. The 2^3 factorial experiment

Full two-level factorial designs are carried out to determine whether certain main factors or interactions (two or more) factors have effects on the response variable and also to estimate the magnitude of such effects. The designs require an experiment to be carried out on all possible combinations of the two levels of each of the *k* factors considered.

Here, Table 6 presents the 2^3 factorial experimental runs obtained from the 3^3 factorial experimental dataset in Table 2, in which the levels were measured at either low (–1) or high (+1). This exercise becomes necessary to create an opportunity to compare, identify and recommend the most efficient model that minimizes weight loss due to corrosion among various alternative models.

Table 6. The 2^3 full factorial design showing runs in actual factors and responses.

Runs	Time (X_1, h)	Inhibitor concentration (X_2, g/L)	Temperature (X_3, K)	Weight loss (g)
1	24	0.001	303	0.90
2	120	0.001	303	2.84
3	24	0.003	303	0.62
4	120	0.003	303	1.96
5	24	0.001	333	1.86
6	120	0.001	333	5.37
7	24	0.003	333	1.36
8	120	0.003	333	4.20

3.1. Results and discussion

Here, the results of the analysis carried out on the dataset presented in Table 6 are discussed.

Table 7. Analysis of variance of 2^3 factorial experiment.

Source	Sum of squares	DF	Mean square	*F*-value	*p*-values
Model	19.004	4	4.751	60.719	0.0033
Error	0.235	3	0.078		
Total	19.239	7			

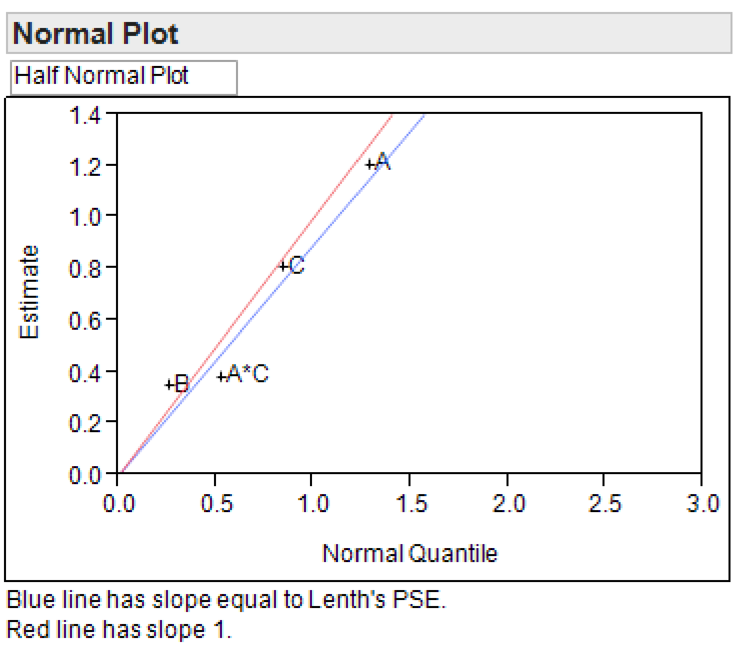
Table 8. Parameter estimates for the 2^3 factorial experiment.

Term	Estimate	*t*-value	*p*-values
Intercept	2.38875	24.15	0.0002*
Time	1.20375	12.17	0.0012*
Conc	–0.35375	–3.58	0.0374*
Temp	0.80875	8.18	0.0038*
Time·temp	0.38375	3.88	0.0303*

From Table 7, the analysis of variance confirms that there is sufficient evidence to reject the null hypothesis (*F*-value of 60.719 and a *p*-value of 0.0033), meaning that at least one of the model parameters is significant and useful in explaining variability in the weight loss. Also, the coefficient of determination, R^2, and adjusted R^2 of the model are 0.988 and 0.972 respectively, which is an indication that an approximately 99% of the variability in the weight loss is explained or accounted for by the model in Equation 3.

The parameter estimates of the treatment effects: time, temperature, time*temperature and inhibitor concentration that are significant and sensitive to the weight loss due to corrosion are presented in the table 7 above. This is eventually represented as a model in Equation 3, which is clearly supported by the half normal plot in Figure 4.

$$Y_{\text{weight loss}} = 2.3888 + 1.2038X_1 - 0.3538X_2 + 0.8088X_3 + 0.3838X_1X_3 \quad (3)$$



A: Time, B: Inhibitor concentration and C: Temperature

Figure 4. Half-normal plot on 3^3 factorial experiments for the independent variables.

3.2. Optimal evaluation of 2^3 factorial experiments

The Figures 5 and 6 are used to evaluate the model obtained in Equation 3 and also to obtain the optimal values. As such, the optimum values of the factors that minimize weight loss due to corrosion are shown in Table 9.

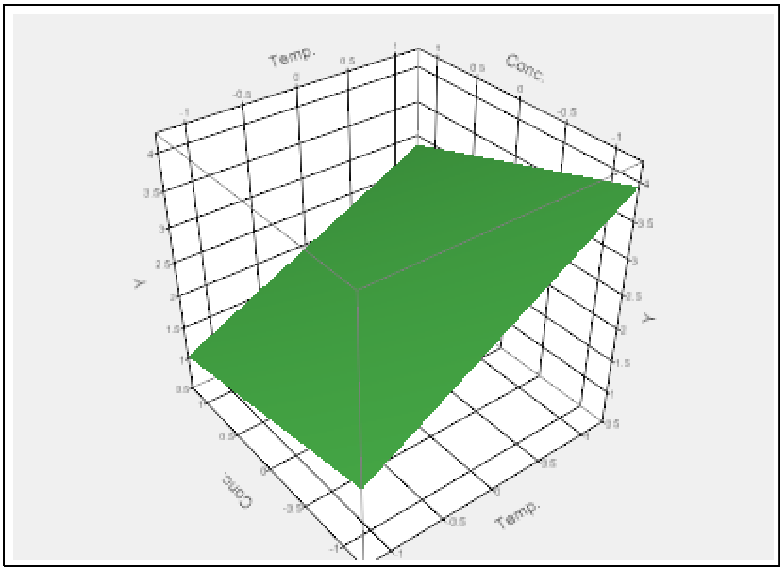


Figure 5. Response surface plot of time against inhibitor concentration.

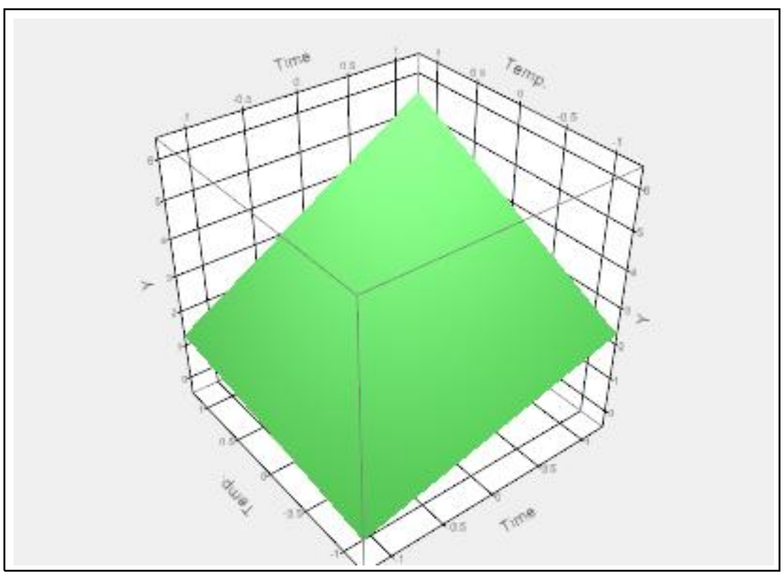


Figure 6. Response surface plot of time against temperature.

Table 9. Optimum values of 2^3 factorial experiments.

Parameters	Optimum values
Weight loss (g)	0.410
Time (h)	24
Inhibitor concentration (g/L)	0.003
Temperature (K)	303

4. The Box–Behnken design (BBD)

The Box–Behnken was devised by George E. P. Box and Donald Behnken in 1960. It consists of a particular subset of the factorial combinations from the three-level factorial design. A minimum of three control variables can be used, with each variable having three levels (high, medium and low).

Table 10. Data presentation in Box–Behnken design format.

Runs	Time (X_1, h)	Inhibitor concentration (X_2, g/L)	Temperature (X_3, K)	Weight loss (g)
1	72	0.001	303	1.68
2	24	0.002	303	0.7
3	120	0.002	303	2.18
4	72	0.003	303	1.32
5	24	0.001	318	0.96
6	120	0.001	318	3.18
7	72	0.002	318	1.95
8	72	0.002	318	1.95*
9	72	0.002	318	1.95*
10	24	0.003	318	0.85
11	120	0.003	318	2.67
12	72	0.001	333	3.54
13	24	0.002	333	1.72
14	120	0.002	333	4.84
15	72	0.003	333	2.88

Centre points were repeated*

4.1. Results and discussion

Here, the results of the analysis carried out on the dataset presented in Table 10 are discussed.

Table 11. Analysis of variance of factorial experiment using Box–Behnken design.

Source	Sum of squares	DF	Mean square	*F*-value	*p*-values
Model	17.324	5	3.465	235.692	<0.0001
Error	0.132	9	0.014		
Total	17.456	14			

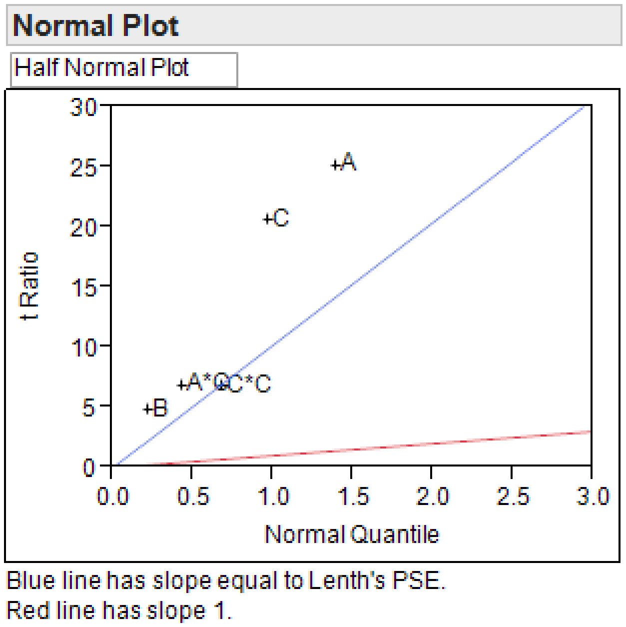
Table 12. Parameter estimates for the Box–Behnken model.

Term	Estimate	*t*-value	*p*-values
Intercept	1.93	42.12	<0.0001*
Time	1.08	25.19	<0.0001*
Conc.	–0.205	–4.78	0.0010*
Temp	0.8875	20.70	<0.0001*
Time·temp	0.41	6.76	<0.0001*
Temp·temp	0.4275	6.81	<0.0001*

From Table 11, the analysis of variance confirms that there is sufficient evidence to reject the null hypothesis (*F*-value of 235.692 and a *p*-value of 0.0001), meaning that at least one of the model parameters is significant and useful in explaining variability in the weight loss. Also, the coefficient of determination, R^2, and adjusted R^2 of the model are 0.992 and 0.988 respectively, which is an indication that an approximately 99.2% of the variability in the weight loss is explained or accounted for by the model in Equation 4.

The parameter estimates of the treatment effects: time, temperature, time*temperature and inhibitor concentration that are significant and sensitive to the weight loss due to corrosion are presented in the Table 12 above. This is eventually represented as a model in Equation 4, which is clearly supported by the half normal plot in Figure 7.

$$Y_{\text{weight loss}} = 1.93 + 1.08X_1 - 0.205X_2 + 0.8875X_3 + 0.41X_1X_3 + 0.4275X_3X_3 \quad (4)$$



A: Time, B: Inhibitor concentration and C: Temperature

Figure 7. Half-normal plot on Box–Behnken design for the independent variables.

4.2. Optimal evaluation on Box–Behnken design

The Figures 8 and 9 are used to evaluate the model obtained in Equation 4 and also to obtain the optimal values. As such, the optimum values of the factors that minimize weight loss due to corrosion are presented in Table 13.

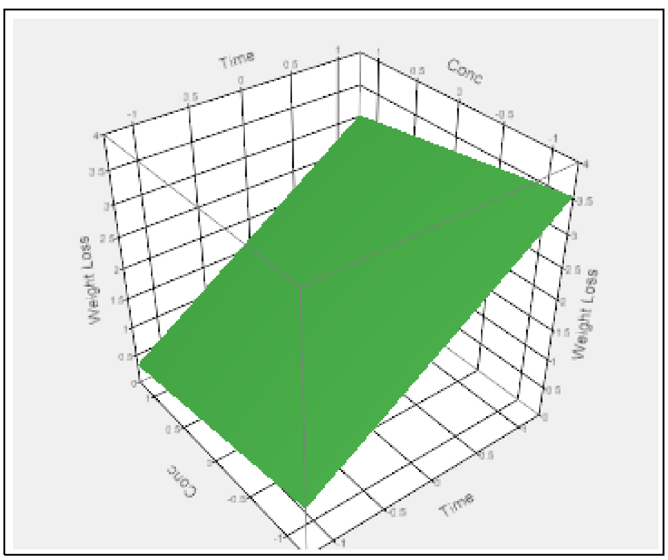


Figure 8. Response surface plot of time against concentration.

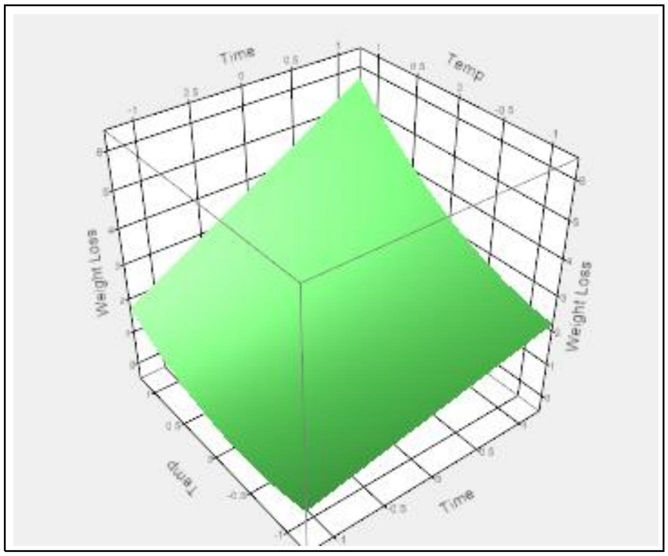


Figure 9. Response surface plot of time against temperature.

Table 13. Optimum values for Box–Behnken model.

Parameters	Optimum Values
Weight loss (g)	0.600
Time (h)	24
Inhibitor concentration (g/L)	0.003
Temperature (K)	303

5. Conclusion

This paper reanalyzed and presented alternatives models to the work of Nkuzinna *et al.* (2014) on the inhibition of copper corrosion using acid extract of *Gnetum Africana,* under three independent factors of time, inhibitor concentration and temperature. Nkuzinna *et al.* gave the optimal (minimum) factor settings to be 24 h, 0.003 g/L, and 303 K for time, inhibitor concentration and temperature respectively. However, it was found that using the statistical model in Equation 1, the predicted weight loss at those settings is 3.30 g, which is much higher than the experimental weight loss value of 0.62 g in Run 7 of Table 2.

Upon this discovery, this paper explored different factorial tools such as a two-level full-factorial design, a three-level full-factorial design and a Box–Behnken design to construct factorial designs and model inhibition of copper corrosion using acid extract of *Gnetum Africana*. Results obtained from the analysis in Section 2, 3 and 4 showed that the three-level full-factorial design earlier proposed by Nkuzinna *et al*. (2014) was relatively less reliable for predicting the effects of the independent factors on the weight loss.

As such, Box–Behnken design gave an efficient model with approximately 99.2% of the variability in the weight loss being explained or accounted for by the model in

Equation 4. On a final note, BBD also optimized the process with a minimum number of experimental runs, with an achievement of approximate values of weight loss when compared with other designs in Section 2 and 3 in the realization of the initial experimental dataset.

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