Experimental correlation using ANOVA and DOE studies on corrosion behavior of Fe and Ni-based alloy under different media

Ghalia A Gaber✉, Lamiaa Z Mohamed✉ and Mahmoud M Tash✉

1 Department of Chemistry, Faculty of Science (Girls), Al-Azhar University, Cairo, 11754, Egypt
2 Mining, Petroleum and Metallurgical Engineering Department, Faculty of Engineering, Cairo University, Giza, 12613, Egypt
E-mail: ghaliaaaid@azhar.edu.eg

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Abstract
The present study was undertaken to evaluate the effect of temperature, immersion time, and different corrosive media such as HCl, H2SO4 and KOH at different time intervals at 30 °C, 40 °C and 60 °C on the corrosion behavior of Fe and Ni-based alloy. The use of the design of experiment (DOE) and the analysis of variance (ANOVA) can be a useful methodology to reach this research. The analysis of the effects of each variable and their interaction on the corrosion of Fe and Ni-based alloys important role in selective best materials choice. The corrosion rate differs with different time intervals and different acid-base environments and increased with an increase in temperature from 30 to 60 °C. The study further reveals that the corrosion rate in different environment follows the order: HCl > H2SO4 > KOH.

1. Introduction
Exposing of metal to different aggressive environments (such as acid cleaning, etching, cleaning of oil refinery equipment, acid descaling, etc) lead to the degradation and damages due to corrosion [1–4]. In an industrial process like pickling, etching, acid cleaning, acid de-scaling, Fe-base alloy is subjected to aggressive solutions such as acidic (HCl, H2SO4) and basic solutions (KOH) which caused corrosion attack. The corrosion magnitude is related to the acid and basic concentration, operating temperature and contact period [4]. The Fe-base and Ni-base alloys are applied for a large range of industrial applications due to their excellent thermo-mechanical processing, corrosion resistance, and high temperature oxidation resistance and strength. Also, they have excellent resistance to dissolution in many industrial aqueous environments especially within the range 36% to 46% Ni [5–7].

Several researchers have studied the corrosion and corrosion mitigation of Fe-Ni alloys in different aggressive media [8–10]. They found that increasing the Ni content in the alloy improves its corrosion resistance [10]. Statistical design of experiments (DOE) and fractional factorial design are efficient, well-established techniques and are applied to study and control the properties and behavior of an alloy system. It allows for reducing the work required to achieve the results. Ping Li et al [11] have performed a quantitative analysis to investigate the effects of a single factor (i.e. temperature, rotation speed, sulfuric acid concentration, and synergistic actions) and their interaction effect on the weight loss and erosion-corrosion rate of 304 stainless steels using a factorial experimental design method (DOE). They reported that the temperature has the most significant effect followed by sulfuric acid concentration. The synergies of the parameters all accelerate the corrosion rate. Similar analyses have been successfully applied to Al-Si alloys in previous studies by Tash et al [12, 13].

The weight loss measurement is a very reliable more accurate technique to study the corrosive behavior of the Fe and Ni-based alloys and also very useful to corroborate the obtained data from the electrochemical analysis [14, 15]. Almost all theories for the initiation and development of pitting concentrate on the way a pit can initiate on what is close to a perfect surface, with material imperfections, inclusions and local alloy constituents producing only very small differences in local potential to drive dissolution leading to pit initiation.
16. Initial pitting usually is considered to initiate at multiple sites on the surface of metal than some stable pits able to propagate with time. The present work provides a useful quantitative technique for estimating/evaluating the effects of environmental factors and material composition/alloying additions in practical engineering applications. An attempt has been made using design of experiments (DOEs) and analysis of variables (ANOVA) to evaluate the effect of temperature, immersion time, and different corrosive media such as HCl, H2SO4 and KOH at different time intervals at 30°C, 40°C and 60°C on the corrosion behavior of Fe and Ni-based alloys. By developing regression equations between the response variable (properties) and the factor varied (composition/alloying additions, time and temperature, and pH value) these equations may be used to predict the conditions required to achieve the desired properties.

2. Materials and methodology

The experimental works were done on four based alloys. The chemical composition is illustrated in table 1. These alloys have the shape of a cylindrical coupon that was ground with 320, 400, 800, 1000 and 1200 grade of emery paper, polished and then dipped into HCl acid solution for 15 min. They were washed first with tap water and with double distilled water, dried in air and then washed with acetone. Dried in hot air and weighed on an analytical balance to accuracy. The specimen weights were noted and then dipped into the test solution at different temperatures 30°C, 40°C and 60°C. At regular time intervals up to 30 min, the specimen were removed from the test solution and washed with tap, double distilled water, dried in the air, washed with acetone and again dried in hot air. Finally, the weights registered after 200 min. The differences in weights at each interval were observed and the corrosion rates and specific reaction rates were measured. A stock solution of analytical grade hydrochloric acid (37%, 1.18 g l⁻¹), sulfuric acid (98%, 1.84 g l⁻¹) and KOH were prepared by using double distilled water. The acid solutions of required concentrations of 1M were prepared by appropriate dilutions. All the chemicals utilized in the current investigation, for the preparation of solutions were of analytical grade.

Weight loss, (mg) measurements were carried out on specimens prepared from Fe and Ni-based alloys. Once the responses, factors (5) and levels have been selected, see table 2, the next step is to design the experimental runs. After the parameters and the values input into the software (MINITAB 14), a DOE model will be automatically generated with the specific number of runs coupled with specific parametric settings. The main factors are %Ni eq., %Cr eq., and Temperature, Time and pH value of the corrosive media.

The %Ni and %Cr equivalents are calculated from the alloy composition data listed in table 1 for the given alloys by using equations developed by Pickering and Schneider [17, 18] as the following:

| Table 1. The chemical composition of Fe and Ni-based alloy. | Chemical composition, % |
|---|---|---|---|---|---|---|---|---|
| Alloy code | Ni | Cr | Mo | Fe | Si | Mn | Al | Ti | Nb |
| 1 Ni-24Cr-10Mo | 65.40 | 24.40 | 9.87 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 2 Ni pure | 99.90 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 3 Fe-35Ni-22Cr | 35.20 | 21.90 | 0.00 | 40.70 | 0.58 | 0.94 | 0.13 | 0.32 | 0.05 |
| 4 Fe-17Ni-18Cr | 17.00 | 17.70 | 1.26 | 60.90 | 0.35 | 1.45 | 0.01 | 0.00 | 0.75 |

| Table 2. Design of experiment (DOE) factors and their uncoded levels. | Level |
|---|---|---|---|---|
| No. | Parameters | Notation | Unit | Low | High |
| 1 | % Ni eq. | A | wt% | 17.7 | 99.9 |
| 2 | %Cr eq. | B | wt% | 0 | 39.2 |
| 3 | Temperature | C | (°C) | 30 | 60 |
| 4 | Time | D | (min,mm²) | 30 | 200 |
| 5 | pH | E | # | 1.01 | 12.8 |

The scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) are used to investigate the corroded surfaces of the alloys.
Experimental correlations of the corrosion rate results of the four alloys listed in Table 1 are analyzed using factorial analysis method. Correlation between responses (weight loss (mg) and corrosion rate) and factors studied (i.e. %Ni eq., %Cr eq., Temperature, Time and pH value) of the corrosive media are investigated, to quantify the effects of these variables on the corrosion behavior of Fe-base and Ni-base alloys. Design of experiment (DOE) and analysis of variance (ANOVA) are conducted and their results are shown in Figures 1–2. The levels of different factors studied are %Ni eq., (%Ni + %Co + 30(%C) + 25(%N) + 0.5(%Mn) + 0.3(%Cu)) (1) %Cr eq. = %Cr + 2%Si + 1.5(%Mo) + 5(%V) + 5.5(%Al) + 1.75(%Nb) + 1.5(%Ti) + 0.75(%W) (2)

Figure 1. ANOVA plots: (a) main effects plot for the mean values of corrosion rate and (b) interaction effect plot for the mean values of weight loss, (mg).

3. Results and discussion

3.1. Experimental correlation: ANOVA and DOE results (mathematical models)

Experimental correlations of the corrosion rate results of the four alloys listed in Table 1 are analyzed using factorial analysis method. Correlation between responses (weight loss (mg) and corrosion rate) and factors studied (i.e. %Ni eq., %Cr eq., Temperature, Time and pH value) of the corrosive media are investigated, to quantify the effects of these variables on the corrosion behavior of Fe-base and Ni-base alloys. Design of experiment (DOE) and analysis of variance (ANOVA) are conducted and their results are shown in Figures 1–2. The levels of different factors studied are %Ni eq., (%Ni + %Co + 30(%C) + 25(%N) + 0.5(%Mn) + 0.3(%Cu)) (1) %Cr eq. = %Cr + 2%Si + 1.5(%Mo) + 5(%V) + 5.5(%Al) + 1.75(%Nb) + 1.5(%Ti) + 0.75(%W) (2)

Analysis of variance (ANOVA) is used to detect the design parameters significantly influencing the corrosion rate. Using the limited data that were available at the time, it was supposed that the modal characteristic also valid to longer-term trends for maximum and average pit depths [16]. ANOVA is used to decide which model
parameters affect significantly the experimental outputs. Figures 1(a) and (b) show the main effect plot and interaction plot for weight loss, (mg), respectively. Figure 1(a) illustrates that increasing the temperature and/or time increases the weight loss, (mg) while increasing the %Ni eq., %Cr eq. and pH reduces the weight loss, (mg). It is observed that the weight loss, (mg) values were reduced with increasing %Cr eq. and %Ni eq., up to 65.4 percent. Increasing %Ni eq. up 99.9 percent increase the weight loss, (mg), figure 1. The weight loss, (mg) was increased with increasing temperature and time. Alloy 1(65.4%Ni and 24.4%Cr) show the best corrosion resistance at low temperature and time for different corrosive media. Alloy 2 (99.9% Ni) shows better corrosion resistance at low pH values. In figure 1(b), the interaction is visible as the two lines are not parallel. It is clear that the effect of temperature or time on increasing the weight loss, (mg), is reduced as the pH increases. The interaction effect between all parameters is illustrated in figure 1(b). The excellent corrosion resistance of Alloy 1(65.4%Ni and 24.4%Cr) in aqueous environments is well-justified to be due to the ability of these metallic alloys to contain the passive film in numerous aqueous solutions. This passive layer prevents corrosion by containing a barrier inhibiting diffusion of the corrosive medium into the metal thus decreasing the dissolution. However, in the existence of aggressive ions such as chloride ion (Cl⁻), and sulphate ion (SO₄²⁻), which are often existed in many industrial environments, the passive oxide layer is susceptible to localized corrosion attack resulting of pits formation which would ultimately accelerate the failure of components in service [19–22]. The general observation on these results is an evident increase in cumulative weight loss with exposure time for all the samples. The reason for the constant difference in cumulative weight loss from the start until the end could be the difference in composition and structure generated by the different addition amounts of Ni and Cr. The

**Figure 2.** Factorial plots: (a) normal Probability plot for the weight loss, (mg) and (b) interaction effect plot for the mean values of weight loss, (mg)
role of Ni and Cr is the same which enhances the passive surface film formation, in the existence of oxygen. These passive films impede the corrosion process and extended protection through the formation of the protective oxide. Increased weight loss with immersion time is due to the interaction that occurs between the acid and the metal surface in solution, which tends to destroy the metal surface gradually with time thereby increasing the weight loss of the metal. Weight loss in constant concentration of the corroding increased with an interaction effect plot for the mean values of weight loss, which obtained by DOE. A mathematical model for the variation of weight loss, (mg) and corrosion rate (mg cm$^{-2}$ h$^{-1}$) was built to relate them with alloying elements content, pH, and testing temperatures and time. Both models represent more than 80% of the variation within the measured weight loss, which represents the reliability of the models. Percent Cr eq. the content was most significant for increasing the corrosion resistance. As expected, testing increasing temperature and time increases the values of weight loss, (mg). The statistical model developed had predictability that explains about 80% of the variation within the measured weight loss, (mg) and corrosion rate (mg cm$^{-2}$ h$^{-1}$) values.

The estimated regression coefficients in the model (1) for weight loss, (mg) show that the main and interaction effects have a noteworthy influence on weight loss, (mg). The p—values are below the accepted value of 0.05, table 3. Also, the p-value for 2-way and 3-way interactions are below the accepted value of 0.05. Similar model is obtained for corrosion rate (mg cm$^{-2}$ h$^{-1}$) (model 2) in table. Within the variation range of the variables studied, these models may be used to predict the weight loss, (mg) or corrosion rate (mg cm$^{-2}$ h$^{-1}$) of the four alloys in the current study. The R-Sq and adjusted R-Sq (adj) values for the weight loss, (mg) regression models are 84.53% and 82.5%, respectively. A similar regression model for the corrosion rate (mg cm$^{-2}$ h$^{-1}$) is obtained for Fe and Ni-based alloys but not presented. The R-Sq and adjusted R-Sq (adj) values for the corrosion rate (mg cm$^{-2}$ h$^{-1}$) regression model are 84.95% and 82.97%, respectively. Mathematical models (Model 1 and 2 in table table 3 and ) are developed to acquire an understanding of the effect of the studied variables and their interactions on the weight loss, (mg) and corrosion rate (mg cm$^{-2}$ h$^{-1}$) of Ni-Cr-Mo alloys.

The coefficient of determination (R 2 and adjusted R 2) show high levels (above 80%) for model 1 and model 2, which proves the adequacy of the models developed. Concerning adjusted R 2, weight loss, (mg) model 1 explains 82.5% of the variation in the data (table 3), while corrosion rate (mg cm$^{-2}$ h$^{-1}$) model 2 explains 82.97% of the variation in the data (table 4). Estimated coefficients for weight loss, (mg) and corrosion rate (mg

| Term                              | Coefficient | P-Value |
|-----------------------------------|-------------|---------|
| Constant                          | 654.721     | 0.000   |
| % Ni eq.                          | −6.522 33   | 0.000   |
| %Cr eq.                           | −32.1763    | 0.000   |
| Temperature, (°C)                 | −16.7219    | 0.000   |
| Time, (min)                       | −3.671 35   | 0.000   |
| pH                               | 13.5383     | 0.287   |
| % Ni eq. %Cr eq.                  | 0.405 682   | 0.000   |
| % Ni eq. Temperature, (°C)        | 0.166 774   | 0.000   |
| % Ni eq. Time, (min)              | 0.036 6997  | 0.000   |
| % Ni eq. pH                       | −0.137 150  | 0.035   |
| %Cr eq. Temperature, (°C)         | 0.816 093   | 0.000   |
| %Cr eq. Time, (min)               | 0.177 147   | 0.000   |
| Temperature, (°C) pH              | −0.035 3877 | 0.000   |
| Time, (min) pH                    | −0.001 565 00 | 0.000 |
| % Ni eq. %Cr eq. Temperature, (°C) | −0.010 2688 | 0.000   |
| % Ni eq. %Cr eq. Time, (min)      | −0.002 210 10 | 0.000 |
| % Ni eq. Temperature, (°C)        | −4.85929E-05 | 0.000 |
| Time, (min)                       |              |         |
| % Ni eq. Temperature, (°C) pH     | 0.000 437 848 | 0.001   |
| % Ni eq. Time, (min) pH           | 0.000 140 598 | 0.000   |
| %Cr eq. Temperature, (°C) pH      | 0.000 694 467 | 0.021   |
| Temperature, (°C) Time, (min) pH  | −2.81753E-04 | 0.000   |

Table 3. Mathematical model (1) for weight loss, (mg): estimated coefficients for weight loss, (mg) using data in uncoded units.
cm$^{-2}$ h$^{-1}$). Models using data in uncoded units are listed in table 3. The model analysis summary is illustrated in table 3. The adjusted -squared implies that the model has good predictability.

Response optimization for weight loss, (mg) and corrosion rate (mg cm$^{-2}$ h$^{-1}$) of Fe and Ni-based alloys and their global solution can be estimated. The model showed that the optimum process variables were 17.73% Ni eq., 22.05% Cr eq., 30°C, 59.25–76.64 min and 1.01 pH to achieve a minimum weight loss, (mg) and corrosion rate (mg cm$^{-2}$ h$^{-1}$) of 1–3 mg and 0.1–0.15 mg cm$^{-2}$ h$^{-1}$, respectively. Alloy 4 (61%Fe -17%Ni-17.7%Cr-1.3%Mn-1.55Mn-0.75%Nb) is the best solution for such optimization. To achieve a weight loss, (mg) and Corrosion Rate (mg cm$^{-2}$ h$^{-1}$) of 10–11 mg and 3 mg cm$^{-2}$ h$^{-1}$, the optimum process variables were 90.95%–91.54% Ni eq., 0.00%Cr eq., 60°C, 65.78–61.16 min and 1.01 pH. Alloy 1 (65.4% Ni, 9.87%Mo and 24.4%Cr) show the best corrosion resistance at low temperature and time for different corrosive media. The best solution is the modified version of Alloy 2 for such optimization. It is possible to determine the conditions necessary to achieve optimal corrosion resistance. The results of this study will provide a large input to existing data, in particular, in the selection of conditions necessary to achieve optimum corrosion resistance of such alloys, by determining the process control parameters necessary.

3.2. Microstructural observation

SEM/EDS investigations were carried out to see the morphology/elemental analysis of the components on the surfaces of the Fe-base and Ni-base alloys after their exposure for long immersion time in acid test solution. Figure 3 displays their SEM micrographs (a, c, e, and g) after their immersion in 1M HCl solutions for 96 h and the corresponding EDS profile analysis (b, d, f, and h) marked in their SEM images (red arrow), respectively. Here, the very low percentages of Fe and Ni compared to their values in the alloy before their exposure to the acid solution is because of the thick layer formation of corrosion products that protecting and hiding its surface. The presence of oxygen also indicates that the corrosion product layer may contain some oxides such as FeO and Fe$_2$O$_3$ for the Fe and NiO Cr$_2$O$_3$ and MoO$_2$, for Ni which may give some protection to the alloy surface [24]. The additional Ni may have developed the formation of NiO to achieve a minimum weight loss, (mg) and Corrosion Rate (mg cm$^{-2}$ h$^{-1}$) of 10–11 mg and 3 mg cm$^{-2}$ h$^{-1}$, respectively. Alloy 4 (61%Fe -17%Ni-17.7%Cr-1.3%Mn-1.55Mn-0.75%Nb) is the best solution for such optimization. To achieve a weight loss, (mg) and Corrosion Rate (mg cm$^{-2}$ h$^{-1}$) of 10–11 mg and 3 mg cm$^{-2}$ h$^{-1}$, the optimum process variables were 90.95%–91.54% Ni eq., 0.00%Cr eq., 60°C, 65.78–61.16 min and 1.01 pH. Alloy 1 (65.4% Ni, 9.87%Mo and 24.4%Cr) show the best corrosion resistance at low temperature and time for different corrosive media. The best solution is the modified version of Alloy 2 for such optimization. It is possible to determine the conditions necessary to achieve optimal corrosion resistance. The results of this study will provide a large input to existing data, in particular, in the selection of conditions necessary to achieve optimum corrosion resistance of such alloys, by determining the process control parameters necessary.

| Term                        | Coefficient | P-Value |
|-----------------------------|-------------|---------|
| Constant                    | 47.8638     | 0.003   |
| % Ni eq.                    | -0.480 393  | 0.001   |
| %Cr eq.                     | -2.364 98   | 0.009   |
| Temperature, (°C)           | -0.576 647  | 0.368   |
| pH                          | 0.013 80    | 0.88    |
| % Ni eq.%Cr eq.             | 0.029 7176  | 0.042   |
| Temperature, (°C) * pH      | -0.008 436 45 | 0.000   |
| % Ni eq.*Temperature, (°C) * pH | -8.67521E-05 | 0.002   |
| %Cr eq.*Temperature, (°C) * pH | 0.000 293 596 | 0.000   |
pH and a thin surface oxide film develops [27]. The corrosion products layer might be cover the whole alloy surface and decreases the aggressiveness attack of the acid molecules on it and confirms the data obtained by the weight-loss method, i.e. the formation of Fe oxide films gives the surface some protection against corrosion.

4. Conclusions

Based on the results of weight loss measurements conclusions are drawn in this study,
1. Mathematical and regression models for calculation of weight loss, (mg) and corrosion rate (mg cm$^{-2}$ h$^{-1}$) in terms of alloying element and different processing parameters are developed to acquire an understanding of the effects of these parameters and their interactions on the weight loss, (mg) and corrosion resistance of Fe and Ni-based alloys.

2. The Ni-based alloy which contains Ni-Cr-Mo is resistant to H$_2$SO$_4$ and KOH in a moderately broad range of concentrations and temperatures, whereas pure Ni is generally unsuitable for hydrochloric acid service.

3. Weight loss increases with an increase in immersion time in acids and base environments. The corrosion rate increases with an increase in temperature. The corrosion rate in different environment follows the order HCl > H$_2$SO$_4$ > KOH.

4. The effect of temperature at a constant acid concentration as in the case of HCl solutions has a strong influence on the corrosion rate of pure Ni, however, the corrosion rate of Ni-based alloy is almost unaffected by the temperature (low activation energy).

5. The Ni-Cr-Mo alloys constitute the best choice, because they are tolerant of residuals, although they are temperature limited at the higher acid concentrations.

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Author contributions

This paper was a shared work between Dr Ghalia A Gaber, Dr Lamiaa Z Mohamed and Dr Mahmoud M Tash. Dr Gh Gaber and Dr L. Mohamed planned and designed the experimental works, while Dr Tash performed the ANOVA and DOE. For the performed tests, Dr Gh Gaber and Dr L Mohamed made the microstructure analysis and corrosion tests while D Tash made the ANOVA and DOE analysis. All authors shared in analyzing the data and wrote the paper in its final form.

Conflicts of interest

The authors declare no conflict of interest.

ORCID iDs

Ghalia A Gaber https://orcid.org/0000-0002-1146-376X
Lamiaa Z Mohamed https://orcid.org/0000-0003-0731-753X
Mahmoud M Tash https://orcid.org/0000-0003-3119-0030

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