Optimizing Study of Some Factors That Influence the Performance of Valve Trays in Crude Oil Distillation Towers

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Abstract. Valve trays play an important role in the productivity of crude oil distillation towers. The performance efficiency of these valves depends on their erosion-corrosion and fouling corrosion. In this research, NiCrAlY powder with constituents of different ratios of Molybdenum (Mo) was used as a deposited coating layer on the surface of 446 stainless steel. The scanning electron microscope and XRD were used to study the microstructure of the coating layer. Microhardness test was carried out to evaluate coating layer hardness. Erosion – corrosion test was performed to evaluate the performance of the coating layer under an aggressive medium of glass sand and a mixture of sulphuric acid and hydrochloric acid. Contact angle test was carried out to evaluate the contact angle of coated and uncoated samples. Taguchi / ANOVA technique was used to tabulate data and to optimize the results. The results showed that the coating layer containing 4% Mo exhibit high erosion-corrosion resistance and good contact angle, which improving the fouling resistance of the samples under working conditions.

Keywords: Valve trays, erosion-corrosion resistance, fouling corrosion, Mo addition, design of experiments Taguchi/ANOVA technique.

1. Introduction

Valve trays are popular because of their efficiency and low cost. The hole of the tray is covered by moveable valves. The vapour load controls the entrance area for the vapour, and thus the movement of the valve. At low vapour loads, the valves are closed, and the vapour enters the tray through the open hole of the valves. Increasing the vapour load caused a wider opening of the valves. The valves are in some way self-adjusting to the vapour load. This is the main advantage of valve trays(Gmehling, Kleiber, Kolbe, & Rarey, 2019). Erosion- corrosion is an important problem for valves trays because of the crude oil's containment (sand) and naphthenic acid. The valves are exposure to fouling corrosion as a result of the presence of sludge and sulfur, and thus decrease the efficiency of the tower.

Erosion – corrosion of metallic materials describes the process of material degradation in which mechanical wear by solid particles, liquid or the combination of both processes combines with
corrosion induced by dissolution or oxidation of surfaces (Rajahram, Harvey, & Wood, 2009). The naphthenic acid erosion-corrosion (NAEC), and corrosion at high temperatures of naphthenic acid (NAC) is the major problems of corrosion of crude oil refineries. These two types of corrosion have become a prominent feature of transmission lines and valves in which flow velocity at high rates in addition to occurring in the other equipment such as heat exchangers, furnace tubes atmospheric and vacuum columns (Slavcheva, Shone, & Turnbull, 1999). The (NAC) and (NAEC), cause significant economic losses to the crude oil refineries and decrease productivity.

Fouling is the accumulation of unwanted material on solid surface of equipment and structures. Deposits, sludge, scaling, sediments, precipitates, unused residue are related to fouling (Coletti & Hewitt, 2014). Corrosion in the equipment of oil refining cause increases in the fouling rate, and vice versa (Kapusta, Ooms, Buijs, Fort, & Fan, 2001; Litschewski, 1996; Yanes, 2012). Corrosion products such as Iron oxide and sulfides and other typical scale materials can cause fouling. Also, clay or dirt from poor desalting/washing of brine salts and catalyst fines may all cause corrosion. (Speight, 2014).

Taguchi method is a standardized form of experimental design technique. It involves reducing the variation in a process through the robust design of experiments. The overall objective of the method is to produce a high-quality product at a low cost to the manufacturer (Anderson & Whitcomb, 2004)

2. Experimental procedure:

The technique of thermal spraying was used in this research. The automated spraying was used by linking the spray gun to an automated control system, that was locally manufactured. The amount of powder entering the spray gun was controlled using an electronic pulse technology system to ensure, uniform distribution of the deposited metal on the substrate surface, that was fixed on a table moving with a bi coordinate system (X, Y) as shown in figure (1).

![Figure1: Thermal Spray device](image)

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**References**

(Rajahram, Harvey, & Wood, 2009)

(Slavcheva, Shone, & Turnbull, 1999)

(Coletti & Hewitt, 2014)

(Kapusta, Ooms, Buijs, Fort, & Fan, 2001)

(Litschewski, 1996)

(Yanes, 2012)

(Speight, 2014)

(Anderson & Whitcomb, 2004)
2.1 Sample and powder materials:

(AISI 446) stainless steel was used in this research with dimensions 22mm in diameter and 6mm in thickness. Sample pretreatment (degreasing and scale removing) were performed according to standard specifications. The roughening of the samples was carried out using sand shoot blast technique to obtain a rough surface (N12) according to (DIN) standard. NiCrAlY powder material was used with particle size range (25-53) micron. Molybdenum powder with particle size (less than 53 microns) was added with ratios (2,4, and 6%).

2.2 Thermal spray parameters:

In this work, thermal spray coating was performed by using an oxyacetylene thermal spray gun. The work sample was fixed in front of a spray gun on a flat table. The sample fixture comprised a ball screw with a stepper motor which makes the upward and downward movements possible. The distance between the spray gun and the sample was 100 mm (Fakhri, Al-Gaban, & Yousif, 2019) and other parameters that related to the thermal spray process are illustrated in table (1).

### Table 1: thermal spray parameters

| Oxygen pressure (bar) | Acetylene pressure (bar) | Powder feeding (g/min) | Traverse velocity (mm/min) |
|-----------------------|--------------------------|------------------------|---------------------------|
| 5                     | 0.8                      | 10                     | 10                        |

2.3 Erosion corrosion test:

To evaluate the performance of the coating layer, erosion-corrosion test was carried out by using the erosion-corrosion system as shown in figure (2).
The system consists of polystyrene tank with two-zones separated by a filter, one of them filled with (a mixture of the hydrochloric acid solution and sulphuric acid solution) and the other filled with (250) gram glass sand with particle size (50 to 150) micron. The concentration of the hydrochloric acid and sulphuric acid were (5, 10, and 15 M).

Taguchi/ ANOVA technique was used to tabulate data and to optimize the results. Four parameters with three levels for each parameter were studied as illustrated in the table (2).

Table 2: Factors and their levels

| Factor          | Level 1 | Level 2 | Level 3 |
|-----------------|---------|---------|---------|
| Mo %            | 2       | 4       | 6       |
| H₂SO₄ (M)       | 5       | 10      | 15      |
| HCl (M)         | 5       | 10      | 15      |
| Exposure time (hr.) | 12     | 24      | 36      |

3. Results and discussion:

Taguchi orthogonal array was used to determine the design of the experiment. A total of nine experiments were executed, and the results are shown in table (3).

Table 3: Taguchi orthogonal array results

| Mo% | HCl conc. (M) | H₂SO₄ conc. (M) | Expos. time (hr.) | Trial 1 | Trial 2 | Trial 3 | Mean | S/N ratio |
|-----|--------------|-----------------|-------------------|---------|---------|---------|------|----------|
| 1   | 2            | 5               | 5                 | 12      | 0.126   | 0.119   | 0.113| 0.119333 | 18.467   |
| 2   | 2            | 10              | 10                | 24      | 0.139   | 0.140   | 0.139| 0.139333 | 17.119    |
| 3   | 2            | 15              | 15                | 36      | 0.144   | 0.143   | 0.144| 0.143667 | 16.8530   |
| 4   | 4            | 5               | 5                 | 12      | 0.133   | 0.132   | 0.133| 0.132333 | 17.5666   |
| 5   | 4            | 10              | 10                | 24      | 0.132   | 0.134   | 0.131| 0.132333 | 17.5666   |
| 6   | 4            | 15              | 15                | 36      | 0.219   | 0.221   | 0.223| 0.221000 | 13.1121   |
| 7   | 6            | 5               | 5                 | 12      | 0.236   | 0.231   | 0.233| 0.233333 | 12.6404   |
| 8   | 6            | 10              | 10                | 24      | 0.236   | 0.237   | 0.236| 0.235333 | 12.5663   |
| 9   | 6            | 15              | 15                | 36      | 0.223   | 0.237   | 0.236| 0.235333 | 12.5663   |

Where Trial 1, 2, and 3 are weight loss (g) due to erosion-corrosion action.

To determine which factor has a strong significant effect on minimizing weight loss, the response table for means was created. From which it is shown that the percentage addition of molybdenum has a high delta value (0.0967) with rank 1. While hydrochloric acid concentration, sulphuric acid concentration and exposure time have a weak significant effect on minimizing weight loss, as shown in table (4).
Table 4: response table for means

| Level | Mo%   | HCl (M) | H2So4 (M) | Exposure time (hr.) |
|-------|-------|---------|-----------|---------------------|
| 1     | 0.1341| 0.1576  | 0.1617    | 0.1632              |
| 2     | 0.1332| 0.1690  | 0.1690    | 0.1642              |
| 3     | 0.2299| 0.1704  | 0.1666    | 0.1698              |
| Delta | 0.0967| 0.0129  | 0.0073    | 0.0066              |
| Rank  | 1     | 2       | 3         | 4                   |

Data listed in table (4) are used to plot the main effects plot of the means. From which it has been shown that the lowest value for weight loss can be achieved at (4% Mo), (5 M HCl), and (5 M H2SO4), as shown in figure (3).

![Main Effects Plot for Means](image)

**Figure 3: Main Effect Plot for Means**

In addition, examining figure (3), indicates that the values of the weight loss of the factors HCl concentration, H2SO4 concentration and exposure time, are somewhat close, which indicates the weakening of these factors’ effect on the average weight loss.

3.1 Analysis of variance ANOVA:

Interaction plot is usually used in conjunction with an analysis of variance.

The effect of two parameters on the weight loss was investigated, as shown in figure (4). From which it shown that Molybdenum at a range of 4% in combination with HCl concentration, H2SO4 concentration, and exposure time with all their ranges has a significant influence on minimizing the weight loss.
The interaction plot also shows the effect of HCl concentration and H$_2$SO$_4$ concentration on weight loss, it can be observed that the weight loss increases when the concentration of both acids at range (5 M). When the coating layer exposed to HCl and H$_2$SO$_4$ acids, the coated layer start to dissolve and release ions. Then the beginning stage of the formation of a molybdenum-rich oxide stable film (MoO$_3$) starts, can acts as an effective barrier against the diffusion of species through the film and, therefore, decrease the dissolution rate. The effect of HCl concentration in combination with exposure time is the same as the effect of H$_2$SO$_4$ in combination with exposure time on weight loss.

Based on the main effect plot for means and interaction plot, characterization and microstructure analysis were performed for samples with (2%, 4% and 6% Mo) using the scanning electron microscope. Figure (5) shows scanning electron microscope images for samples with 2, 4, and 6% molybdenum. From which it shown that a sample with 2% Mo has a coarse structure, while the sample with 4% molybdenum has a more smooth and homogeneous structure which make it more resistant to erosion-corrosion. As for sample with 6% Mo, a crack can be notice in its structure along the grain boundary, making it weak to resist erosion-corrosion, because the electrolyte penetrates through these defects to reach the substrate. This gives the impression that the percentage of molybdenum addition plays an important role in increasing the performance efficiency of the coating layer in corrosion resistance and that the 6% addition ratio leads to initiate cracks in the coating layer structure. These cracks will enable the electrolyte solution to penetrate to the metal substrate.
Figure 5: Scanning Electron Microscope images for testing samples
The XRD pattern of the coating layer shown $\beta$-Ni(NiAl) and $\gamma$-Ni(NiCr) phases, which behaves as aluminium reservoir and MoO$_3$ which can act as a protective barrier against the diffusion of species that cause corrosion through the film and, therefore, decrease the corrosion rate.

![XRD pattern of the surface of the coating layer]

**Figure 6**: XRD pattern of the surface of the coating layer

### 3.2 Confirmation experiments:

The above experimental analysis, main effect plot for means show that the maximum adhesion force can be obtained with the following conditions presented in Table 5.

**Table 5**: optimal design conditions

| Factor       | Best level |
|--------------|------------|
| Mo (%)       | Level 2    |
| HCl (M)      | Level 1    |
| H$_2$So$_4$ (M) | Level 1   |
| Exposure time (hr.) | Level 1    |

Three experiments of erosion-corrosion (weight loss) were performed under the optimal conditions in order to validate the optimization, from which it shown that the results of confirmation experiments and the predicted weight loss by optimization procedure are considered to be valid based upon the results of Taguchi experiments and within the 95% confidence interval.

### 3.3 Contact angle

After the execution of the confirmation, the contact angle was inspected after and before the coating process. The results show that the surface of the uncoated sample was hydrophilic and the coating changes the nature of the surface to hydrophobic. This means that the samples will have higher resistance to fouling corrosion Figure(6) represents a Photograph of water droplet of Stainless Steel (AISI 446) substrate with and without coating.
a. the contact angle of uncoated samples

b. the contact angle of the coated sample

Figure 7: Photograph of water droplet of Stainless Steel (AISI 446) Substrate with and Without Coating

4. Conclusion

In this work, NiCrAlY with different ratio of Mo (2, 4, and 6%) were applied on (AISI 446) stainless steel by using a flame spray technique. Taguchi / ANOVA technique was utilized in this research to optimize the results of erosion-corrosion and tabulate the data. The parameters investigated were Molybdenum percentage, HCl concentration, H₂SO₄ concentration, and exposure time. From the results of Taguchi, it is observed that the percentage of Mo has a strong and significant effect on minimizing weight loss, which has high delta value (0.0967) with rank 1. The other factors (HCl concentration, H₂SO₄ concentration, and exposure time) have an approximately close and weak effect on weight loss with delta value 0.0129, 0.0073, and 0.0066 respectively. The main effect plot for means showed that the low weight loss was achieved at level 2 for Mo% and level 1 for HCl concentration, H₂SO₄ concentration, and exposure time. SEM image showed that coating layer with 4% Mo makes the structure smooth and homogeneous, which as a result make it more resistant to
erosion-corrosion. Contact angle test showed that the surface of uncoated samples have contact angle 46.859, and 47.772, while the contact angle for the surface of the coated samples were 80.603, 110.612, 110.979, and 102.481, which makes the coated samples more resistance to fouling corrosion.

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