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Anti-Wax Nickel based Coating for Stainless Steel Pipeline

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ABSTRACT

This study aims to determine the efficacy of nickel-based coating as wax repellent for stainless steel pipelines. Two variables for nickel-based coating were studied namely nickel sulphate and sodium hypophosphate. Results show that 100% and 50% reduction of wax deposition for coating with sodium hypophosphate and coating with nickel sulphate respectively. These values indicate that nickel based coating can reduce wax deposition on the pipe surface. It also depicts that coating with more sodium hypophosphate serve better anti-wax coating compared to nickel based coating with more nickel sulphate. SEM analysis shows good surface morphology for the coating and displays a microstructure that plays an important role in protecting stainless steel surfaces.

1. Introduction

Deposit wax is referred as Naphtenic hydrocarbons of (C_{18} – C_{36}), which are the main component of crude oil. Over time, this wax will gradually deposits in pipes if the temperature of the crude oil is below its wax appearance temperature [17]. Wax deposition has been one of the significant problems in production and oil transportation in pipelines. It causes the flow area in the pipes becomes smaller with time and eventually incurs blockage that affecting the safety and capacity of the pipelines [5]. Oil productivity will also be affected and incurred considerable economic loss to the company.

Coating is one of the methods to inhibit wax deposition in pipelines. A lot of studies has been carried out to prevent wax from deposited in pipes. For example the use of copper oxide to coat pipeline [1], Zn–Fe and Zn–Ni–Fe alloy coatings for oil pipeline, Bio-inspired composite coating for extreme underwater pipeline [9] and Ti-Ni composite coating [10]. These studies have demonstrated positive results where the coatings show better anti-wax performance.

This study applies conversion coating method to coat the metal surface [13]. Conversion coating is a coating method that is prepared by modifying metal surface using chemical or electrochemical

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methods. Conversion coating can increase surface hardness [11], metal protection [15] and surface colour decoration [3]. This method has shown evident that it exhibits better anti-wax capabilities than carbon steel and heat treated carbon steel in water contained crude oil [17].

Nickel based coating has been widely used to prevent wax deposition on metals due to its reasonable cost [2]. There are several types of nickel based coating such as Ni-P coating [14], Zn–Ni–Fe alloy coatings [12] and Ti-Ni coating [10]. This study uses Ni-P coating to coat metals for several reasons. The reasons include it can be applied to a variety of substrate materials and uniformly on intricate part geometries [6], it has good capabilities in anti-friction, anti-corrosion and electromagnetic characteristics [7] and its uniform deposition and excellent wear and corrosion resistance characteristics [8].

However, Ni-P has drawback where it starts getting oxidized at temperature ≥ 400 °C in the air and ultimately affect the corrosion property of the coating [16]. Thus, Sodium hypophosphite is also introduced in coating because hypophosphite has been mostly used as reducing agents for electroless nickel and also capable of enhancing corrosion resistant of the pipeline [2].

This paper investigates the effect of nickel coating as anti-wax material on stainless steel pipe. This is done by varying nickel sulphate concentrations and sodium hypophosphate concentrations of nickel coating and analyse its morphology.

2. Materials and Methods

2.1 Crude Oil Sample

The crude oil used in this research was obtained from Local Malaysia Oil offshore field. The tested wax appearance temperature (WAT) of sample is 25°C and the water content is 26.81%.

2.2 Stainless Steel Substrate

Stainless steel plates of 3.0 cm x 3.0 cm x 0.4 cm were used as the substrates. The stainless steel substrate was degreased ultrasonically in ethanol for 5 min and was rinsed with deionized water. Then, it was immersed into 5% dilute hydrochloric acid for 1 min and rinsed again with deionized water thoroughly with temperature set at 100°C. This process is to remove contaminating materials before coating.

2.3 Preparation of Nickel-Based Coatings

The nickel-based coating synthesized based on method used by Ping *et al*., [14]. Plating bath solutions are prepared by mixing nickel sulphate powder, sodium hypophosphite powder, sodium citrate powder and sodium acetate powder. Samples for coatings are prepared based on Table 1 and Table 2. The coating method used in this research was dipping followed by heat treatment. Stainless steel substrates were immersed in the solution for an hour and dried before being placed into an oven at a temperature of 150°C for an hour.

2.4 Wax Deposition Test

The wax deposition test is conducted in an adapted-designed apparatus [17]. The crude oil was heated up to 80°C and stirred until complete dissolution. Coated and non-coated samples were settled into the specific placeholders which are on the inner wall of the equipment. Crude oil sample was maintained at 80°C for 30 minutes and then temperature was reduced until 25°C.Next, substrates were taken out and weighted.

The anti-wax deposition ratio, R_{aw} was determined by finding the weight difference of stainless steel after experiment over initial weight of stainless steel [12]. The formula is

$$R_{aw} = \left(\frac{W_1 - W_2}{W_1}\right) \times 100 \quad (1)$$

R_{aw} = Anti-wax deposition ratio
W_1 = The stainless steel sample, g,
W_2 = The wax deposition amount on sample, g.

Table 1
Samples mixture prepared for nickel sulphate coating

Sample	Nickel Sulfate Hexahydrate, g/L	Sodium Hypophosphite hydrate, g/L	Sodium Citrate hydrate, g/L	Sodium Acetate hydrate, g/L
1	30	40	50	60
2	10	10	10	10
3	10	10	10	10
4	5	5	5	5

Table 2
Samples prepared for sodium hypophosphate coating

Sample	Nickel Sulfate Hexahydrate, g/L	Sodium Hypophosphite hydrate, g/L	Sodium Citrate hydrate, g/L	Sodium Acetate hydrate, g/L
1	30	30	30	30
2	20	30	40	50
3	10	10	10	10
4	5	5	5	5

2.5 Surface Imaging

The surface morphology of nickel coating was examined via scanning electron microscopy (SEM Oxford Instruments INCA X-act from UK). The magnification used in the SEM Imaging test is 10 µm. Eight samples were tested which, include 4 samples of nickel coated with nickel sulphate and 4 samples of nickel coated with sodium hypophosphate.

3. Result and Discussion

3.1 Wax Deposition

Wax deposition test was carried out to study the effect of nickel based coating on steel pipes. Four different concentrations of nickel sulphate and sodium hypophosphite samples of nickel coating were investigated. Table 3 and Table 4 show the weight of deposited wax on the nickel sulphate and sodium hypophosphite coatings respectively.

Table 3
Wax deposition reduction of nickel coating with varying concentration of nickel sulphate

Concentration of Nickel Sulphate, g/L	W_2 (g)	W_1 (g)	R_{aw}
30	0	0	0
40	0.001	0.002	50
50	0	0	0
60	0.001	0.002	50

Table 4
Wax deposition reduction of nickel coating with varying concentration of sodium hypophosphite

Concentration of Sodium Hypophosphite, g/L	W_2 (g)	W_1 (g)	R_{aw}
20	0.002	0.002	0
30	0.002	0.003	33.33333
40	0.001	0.002	50
50	0	0.001	100

The Anti-wax Deposition Ratio, Raw, was calculated to evaluate the efficiency of nickel coating to reduce wax deposition. Data shows that most of the coatings were able to reduce wax deposition significantly at up to 50 or 100% reduction. Nickel coating with varying sodium hypophosphite shows gradual wax reduction as the concentration increases up to 50g/L. Nickel coating with nickel sulphate concentration on the other hand exhibits consistent wax reduction of 50% for concentration of 40 g/L and 60 g/L.

Furthermore, it is found that nickel coating synthesized from nickel sulphate of 30 g/L and 50g/L concentrations showed no wax deposition reduction (Table 3). This is similar happened to nickel coating with sodium hypophosphite concentration of 20 g/L (Table 4). This may due to coating process is unsuccessful thus, it shows no weight on the stainless steel surface recorded. Nevertheless, the nickel coating did not contribute to any further deposition of wax precipitate during the experiment for both variables.

Referring to the data, it is found that the amount of deposited wax recorded was not significant enough to demonstrate the efficiency of coating as anti-wax coating. This is due to the small size of substrates used during experiment, which lead to an insignificant surface area for wax deposition. All samples for wax deposition test were cut into identical sizes of 3.0 cm x 3.0 cm x 0.4 cm. This result may also be contributed by the short duration of deposition time given during the experiment. It is known that in real oil and gas industry, the transportation of the crude oil through pipeline is a continuous process . Hence, the contact duration between oil and pipe surface for wax deposition is longer. This leads to a large gap between the laboratory and industrial scale value. Nevertheless, the results obtained have shown high potential for anti-wax deposition coating.

3.2 Surface Characterization

SEM analysis was conducted in order to study the effect of surface morphology to anti-wax deposition of the coating surface. Substrates were heated at 150°C for an hour and cut into 1 cm x 1 cm square shapes before investigation with SEM analysis. Figure 1 shows the surface image of non-coated steel surface. The surface is coarse with some obvious scratches on it which may due to mechanical polishing.

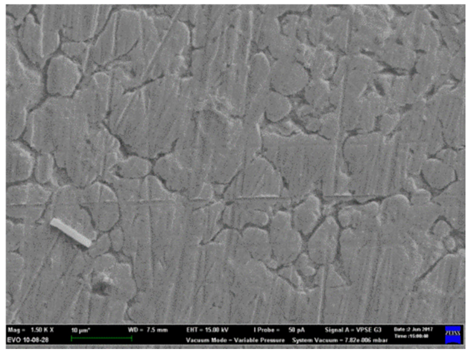

Fig. 1. SEM image of non-coated surface

Figure 2(a) till 2(d) show the SEM images of nickel coated substrate for varies concentration of nickel sulphate from 30g/L till 60 g/L. It is observed that Figure 2(c) and Figure 2(d) illustrates significant distinct surface morphology compared to Figure 2(a) and 2(b), where it shows significant cracks on the surface. Figure 2(d) however has some fine granules on the surface in addition to the crack. Crack surface may due to post plating heat treatment [14]. Figure 2(a) shows similar surface morphology like the non-coated steel while Figure 2(b) shows the surface is spread with small granules. Small granules may indicate wax deposition.

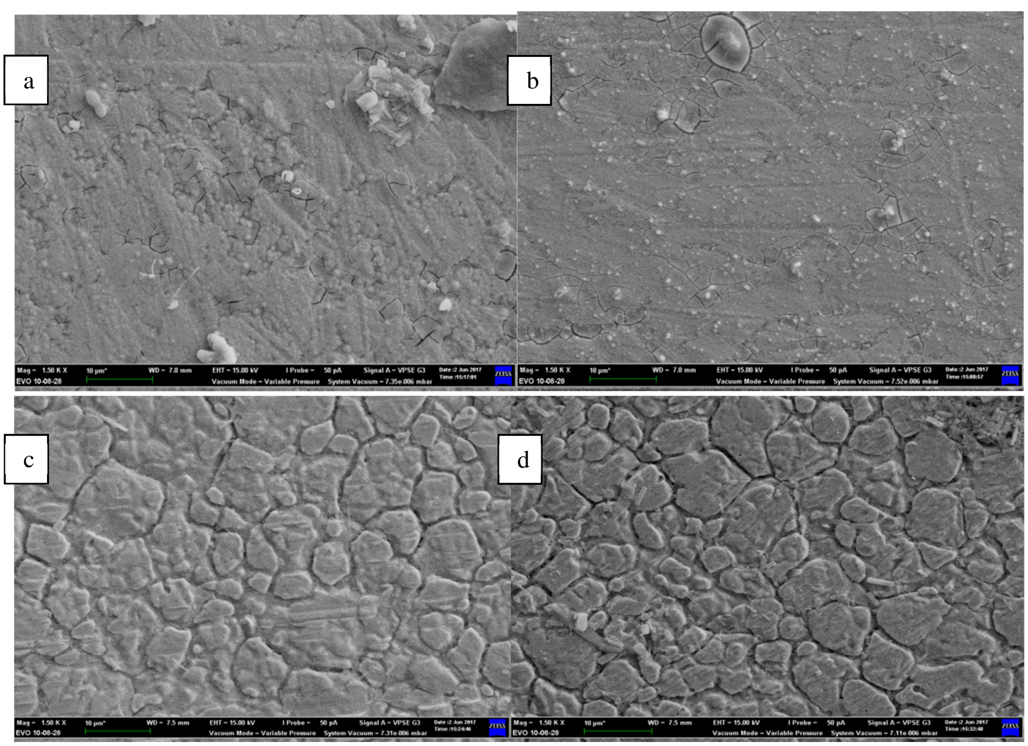

Fig. 2. SEM Images for nickel manipulated stainless steel for 30 g/L (a), 40 g/L (b), 50 g/L (c), 60 g/L (d) of nickel sulphate concentration at 10µm.

Figure 3(a) till 3(d) on the other hand, show SEM images of nickel coated substrate for varies concentration of sodium hypophosphate from 30g/L till 50 g/L. It is observed that Figure 3(c) and Figure 3(d) display distinct images compared to Figure 3(a) and 3(b). The surface is relatively similar to non-coated surface image in Figure 3(a) with some small fine granules on it. Figure 3(b) shows some small granules with rod-like microstructure on the surface. As for Figure 3(c), the surface shows light cracks and some fine nodular structure spreads. Figure 2(d) on the other hand, illustrates obvious nodular structure, formed by typical cauliflower-like shapes surface. This shape is relatively similar to studies conducted by Ping *et al.*, [14].

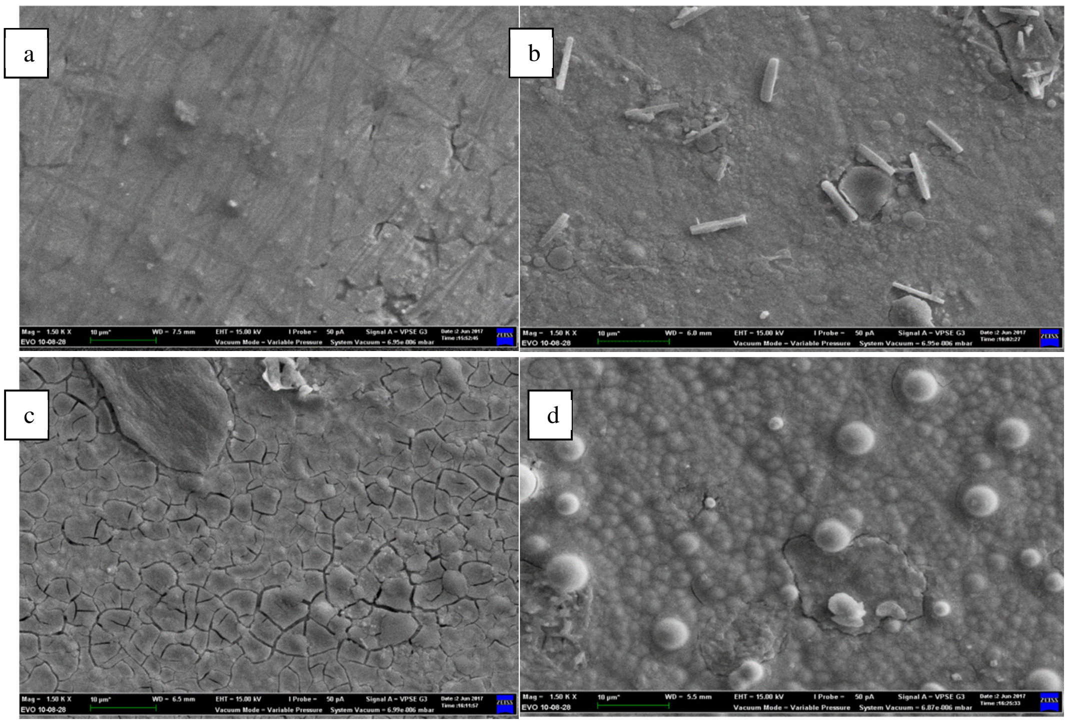

Fig. 3. SEM Images for nickel manipulated stainless steel for 20 g/L (a), 30 g/L (b), 40 g/L (c), 50 g/L (d) of sodium hypophosphite concentration at 10µm.

Nevertheless, the current study only focused on the efficiency of nickel coating to reduce deposition of wax onto pipeline surface. Pipeline coating via dipping and heating may not be durable for industrial application. Hence, further research on the coating method to provide long lasting functional coating is suggested.

4. Conclusion

Nickel-based coating with varies nickel sulphate concentration and sodium hypophosphite concentration were studied to analyse its anti-wax performance. Nickel coating with sodium hypophosphate concentration of 50% exhibits the highest R_{aw} which is 100%. Nickel coating with nickel sulphate concentration of 40% to 60% shows R_{aw} of 50%. This indicates that 50 g/L concentration of sodium hypophosphate demonstrates better nickel coating compared to nickel sulphate to reduce wax deposition in crude oil pipeline. Surface imaging especially for nickel coating with sodium hypophosphate also showing good surface morphology which has potential to enhance anti-wax performance on steel surface.

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