Evaluation on the factors influencing the deposition of wax using full factorial design

N Ridzuan¹, A A Azhar¹ and P Subramanie¹
¹Faculty of Chemical and Natural Resources Engineering, Universiti Malaysia Pahang, Lebuhraya Tun Razak, 26300 Kuantan, Pahang, Malaysia
*E-mail: norida@ump.edu.my

Abstract. The purpose of this study is to determine the rate of wax deposition of Malaysian crude oil by evaluating the operational factors using full factorial design (FFD) approach (Design Expert version 7.1.6). Four factors were studied, which are rotation speed (A), cold finger temperature (B), experimental duration (C) and type of wax inhibitor (D), Poly(ethylene-co-vinyl acetate) (EVA) and xylene were used as a wax inhibitor. The individual effects of each factor and its interaction effects towards the response variable were studied. The result demonstrated that fewer wax deposit was obtained (0.0055 g) when optimal conditions of 2 h duration with 600 rpm rotation speed at 15°C cold finger temperature and EVA as an inhibitor were used. From the analysis of variance (ANOVA), factor B was found to be the main factor in affecting the wax deposit formation. However, the interaction between factors A and D shows the highest influence to reduce wax deposition. Furthermore, the model is designated with high determination coefficient ($R^2 = 0.8221$), which explains 82.21% of the variability in the response. Hence, it can be concluded that factor B, and interaction between factors A and D need to be focused in controlling the parameters to minimize wax deposition.

1. Introduction
As the conventional oil reserves decline, the production of unconventional oil, such as waxy crude oil and heavy crude oil, increases. In fact, approximately 20% of the global oil reserves are of waxy crude oil, while heavy crude oil constitute about half of the recoverable oil reserves [1]. The production and transportation of these oil reserves in the deep water region (with the ambient seawater of extremely low temperature) through the pipelines lead to various types of flow assurance challenges and wax deposition which is one of the most significant problems in the industry [2]. Under the conditions of temperature, pressure, and crude oil composition occurring in underground reservoir, the paraffin is in suspension (colloidal) or solution in the crude. The temperature, pressure, and the amount of dissolved gas in oil are reduced when the reservoir is penetrated (as the oil flows through the pipeline [3]. However, certain studies claimed that the reduced temperature and gas liberation during the production are responsible for the reduction of wax solubility in the crude oil (Akinyemi et al., 2018). As the crude oil flows through the pipeline to the surface, reduced temperature and pressure reduce the solubility of paraffinic hydrocarbons (or known as waxes).

Wax formation from paraffin deposition of crude oil constitutes a major and serious problem in both onshore and offshore production facilities [4]. This paraffin deposition will cause flow assurance problem for oil and gas production. During the transportation of crude oil, at temperature below wax appearance temperatures (WAT), the crude oil will be solidified and form wax crystals [5]. The wax will grow and form a crystalline net that will trap all molecules of liquid hydrocarbon, causing oil flow
blockage in pipeline [6]. According to Telford [7], the design of experiments is a structured and organized way of conducting and analyzing controlled test to evaluate factors that are affecting a response variable. Full factorial design (FFD) is a more operative method to determine the impact of two or more factors on a response compared to one-factor-at-a-time (OFAT) experiments which contrast only one factor at a time while keeping others fixed [8-9]. Therefore, the objective of this research is to identify the factor that will influence the wax formation the most and to investigate the interaction between the factor analysis.

2. Materials and method

2.1. Materials
Crude oil sample was supplied by PETRONAS Penapisan Terengganu, Kertih, Malaysia. The chemical wax inhibitors used were poly(ethylene-co-vinyl acetate) (EVA) and xylene which were obtained from Sigma-Aldrich.

2.2. Cold finger experimental procedure
To conduct the experiment, the crude oil was first conditioned above the WAT to remove the crude oil thermal history and to obtain a homogeneous mixture where all wax particles were dissolved [10]. The temperature of heating water bath and chilling water were set at 50°C and 5°C, respectively [11] A 300 mL of crude oil sample was prepared in a vessel and was placed inside the water bath. Then, 10 mL of chemical inhibitor was injected into the vessel. After 2 hours, the wax deposit on the cold finger was scrapped off from the cold finger and weighed. The experiment was repeated for three times to obtain a precise data. The experiment was conducted according to the standard order sequence designed by the Design Expert as shown in Table 1.

| Standard Order | A  | B  | C  | D     | Wax Deposit(g) |
|----------------|----|----|----|-------|----------------|
| 1              | 200| 5  | 2  | EVA   | 0.1996         |
| 2              | 600| 5  | 2  | EVA   | 0.7579         |
| 3              | 200| 15 | 2  | EVA   | 0.1016         |
| 4              | 600| 15 | 2  | EVA   | 0.0055         |
| 5              | 200| 5  | 4  | EVA   | 0.0824         |
| 6              | 600| 5  | 4  | EVA   | 0.9996         |
| 7              | 200| 15 | 4  | EVA   | 0.0665         |
| 8              | 600| 15 | 4  | EVA   | 0.1310         |
| 9              | 200| 5  | 2  | Xylene| 0.6676         |
| 10             | 600| 5  | 2  | Xylene| 0.2168         |
| 11             | 200| 15 | 2  | Xylene| 0.1501         |
| 12             | 600| 15 | 2  | Xylene| 0.0738         |
| 13             | 200| 5  | 4  | Xylene| 0.7077         |
| 14             | 600| 5  | 4  | Xylene| 0.7537         |
| 15             | 200| 15 | 4  | Xylene| 0.014          |
| 16             | 600| 15 | 4  | Xylene| 0.1614         |

A = speed of rotation, rpm; B= cold finger temperature, °C; C = experimental duration, hr; D = type of wax inhibitor
2.3. Design of experiment

The FFD method was used in this study to screen the best factors that contribute to the wax deposition and identify the interaction between factors. The four factors used in this experiment were: rotation speed, (A) (200–600 rpm), cold finger temperature, (B) (5–15°C), experimental duration, (C) (2–4 hr) and type of inhibitor (D) (EVA and xylene). Wax deposit will be the response variable. The statistical analysis of data was conducted using Design-Expert 7.1.6. The data analysis was conducted on each factor based on the result in order to analyze the effect of each factor and interactions between factors on how it affects the amount of wax deposit.

Table 1 presents a total of 16 experiments where each row of the matrix represents one run of experiment at a different condition for each run along and the experiments were repeated thrice for each run in order to obtain precise data. The experiments were run in random sequence in order to reduce the systemic bias of uncontrolled factors [12].

3. Results and discussion

3.1 Analysis of variance (ANOVA)

ANOVA was used to analyze the results of the experiment and to determine the variation due to each factor. From the ANOVA analysis, the mean value is 0.32 with a standard deviation of 0.20. From the F-test of ANOVA result as shown in Table 2, values of "Prob > F" less than 0.05 indicate that model terms are significant. Factor B is a significant model term. Whereas, the other factors are insignificant. This is presumable as a result from experimental error which can be described as “noise” to the system [13]. The model p-value of 0.0409 implies that the model is significant. Furthermore, the model is designated with high determination coefficient ($R^2 = 0.8221$), which explains 82.21% of the variability in the response. The predictive equations are based on the fitted model for the response with all the factors in coded form (-1,1) expressed in Equation 1 and Equation 2 for EVA and xylene, respectively:

\[
\text{Wax Deposit} = + 0.21 + (3.84 \times 10^{-4})A + (4.50 \times 10^{-4})B - 0.026C - (6.45 \times 10^{-5})AB + (3.88 \times 10^{-4})AC - (8.25 \times 10^{-3})BC
\]  

(1)

\[
\text{Wax Deposit} = + 0.707 - (7.26 \times 10^{-4})A + (4.50 \times 10^{-4})B - 0.026C - (6.45 \times 10^{-5})AB + (3.88 \times 10^{-4})AC - (8.25 \times 10^{-3})BC
\]  

(2)

3.2 Main factors analysis on the amount of wax deposit

Figure 1 shows the main factors graph of FFD with four screening factors at two levels to determine the individual effect of the operating factor on the amount of wax deposit.

![Figure 1 Main factors graph of (a) rotation speed, A; (b) cold finger temperature, B; (c) experimental duration, C; and (d) type of wax inhibitor, D, on wax deposit](image-url)
3.2.1  Effect of rotation speed of impeller

By referring to Table 2, the effect of rotation speed of impeller shows the second highest contributor to wax deposit with the percentage of contribution being 4.67%. Based on Fig. 2(a), the total wax deposit amount increased at the highest stirring speed. Stirring process influenced both the shear stress field and the rate of heat transfer at the cold finger surface [14]. At a higher shear force, it causes an increase in the internal energy that supports the crystallization of paraffin wax in crude oil [15]. Thus, wax deposit increase when stirring speed is increased.

![Figure 2 Wax deposit at different rotation speed (a) 200 rpm and (b) 600 rpm](image)

| Source            | Sum of Squares | DF | Mean Square | F value | p-value Prob > F | % Contribution |
|-------------------|----------------|----|-------------|---------|-----------------|----------------|
| Model             | 1.36           | 8  | 0.17        | 4.04    | 0.0409          |                |
| A - Rotation      | 0.077          | 1  | 0.077       | 1.84    | 0.2173          | 4.67           |
| B - Temperature   | 0.85           | 1  | 0.850       | 20.21   | 0.0028          | 51.36          |
| C - Duration      | 0.035          | 1  | 0.035       | 0.82    | 0.3941          | 2.09           |
| D - Inhibitor     | 0.010          | 1  | 0.010       | 0.24    | 0.6393          | 0.61           |
| AB                | 0.066          | 1  | 0.066       | 1.59    | 0.2483          | 4.03           |
| AC                | 0.096          | 1  | 0.096       | 2.29    | 0.1737          | 5.83           |
| AD                | 0.20           | 1  | 0.20        | 4.71    | 0.0665          | 11.97          |
| BC                | 0.027          | 1  | 0.027       | 0.65    | 0.4470          | 1.65           |
| Residual          | 0.29           | 7  | 0.042       |         |                 |                |
| Cor Total         | 1.65           | 15 |             |         |                 |                |

3.2.2  Effect of cold finger temperature

Cold finger temperature factor shows the highest percentage of contributor value, with a percentage of 51.36%, in affecting the amount of wax deposit amount. Figure 1(b) shows that high wax deposit amount was obtained at a low temperature because of the tendency of wax molecules to form wax crystal below the WAT [16]. To perform the effect of temperature to the wax deposition rate, the cold...
finger temperature was adjusted below the WAT value [14]. At low temperatures, the wax molecules stick to each other easily and precipitate onto the cold finger surface [17].

The comparison of physical appearance of wax deposit at different temperatures are shown in Figure 3(a) and Figure 3(b). From the figures, at 5°C, the wax deposit was observed to be thick and hard, depositing all over the surface of the cold finger. Whereas at 15°C, there was very small amount of wax on the cold finger.

3.2.3. Effect of experimental duration
The experimental duration with an influence percentage of 0.61% indicates the least important factor on wax. Figure 1(c) shows a slight increase in the amount of deposit over time. As wax deposition happens at temperature lower than WAT, the growth of crystal wax increases over the time [18].

3.2.4. Effect of type of wax inhibitor
The type of wax inhibitor gives 2.09% of percentage contributor to the wax deposition rate. This factor has a slight effect to the wax deposit formation. From Figure 1(d), it was further observed that EVA performed better than xylene as a wax deposition inhibitor. Based on the study by Machado and co worker [19], EVA has a long polymeric chain in a polar group, where it acts as wax crystal modifiers to create a barrier to the formation of crystal wax network and thus hinders the wax deposition. Even though xylene has a polar group as EVA, the molecule interaction of xylene with wax molecule is weaker than the EVA as xylene has less carbon molecular weight which leads to lesser intermolecular forces with wax [20].

3.3. Interaction factors analysis on the amount of wax deposit
Figure 4 shows the interaction between two factors on the amount of wax deposit for the combination of each factor. Figure 4(a) shows the interaction graph between rotation speed (A) and cold finger temperature (B). The AB interaction contributes 4.3% impact on wax deposition. Factor A shows less wax deposit amount when factor B is at 15°C. By introducing low stirring rate, there is lesser energy for wax to form crystal energy and at a temperature above than the WAT, the interaction at that condition causing lesser wax deposit amount attached on the cold finger [10].

Figure 4(b) shows the interaction graph of wax deposit between rotation speed (A) and experimental duration (C). The AC interaction gives 5.83% influence on wax deposition, which is slightly higher than the AB interaction. In order to obtain lesser amount of wax deposit, low rotation speed should be set for 4 hours' duration. Since factor C gives less impact factor to the wax deposition, it shows not much difference to the amount of wax deposit at a lower rotation speed for a duration of either 2 or 4 hours.
Figure 4(c) shows the interaction graph on wax deposit between rotation speed (A) and the type of inhibitor (D). AD interaction effect has the highest percentage of contribution, which is at 11.97%, towards wax deposition. For a better wax inhibition, EVA should be used at low rotation speed. This operating condition should prohibit wax network from forming at low speed with EVA as the inhibitor since EVA is a good wax crystal modifier and hence, reducing wax deposition in crude oil [21]. However, when xylene was used as inhibitor, the amount of wax can be reduced if the rotation speed was increased from 200 rpm to 400 rpm.

Figure 4(d) shows the interaction graph on wax deposit between cold finger temperature (B) and experimental duration (C). BC interaction exhibited the least interaction effect, whereby it contributed only 1.65% influence on wax deposition. When cold finger temperature is above WAT, there will be lesser amount of wax deposit at a shorter duration. As stated by Jennings and Weispfennig [14], wax deposition will only occur at below WAT and when the duration is longer, a high amount of wax will be produced.

![Interaction graphs between factors on wax deposit: (a) rotation speed, A and cold finger temperature, B; (b) rotation speed, A and experimental duration, C; (c) rotation speed, A and type of wax inhibitor, D; (d) cold finger temperature, B and experimental duration, C, on wax deposit](image)

3.4. Model validation
Model validation was conducted in order to test the reliability and consistency of the model [22]. This can be done by conducting the experiment by varying any of operating factors then compared with the predicted value calculated from the model.

Table 3 shows the model validation data. The data obtained from experiments were then compared with the predicted ones. The predicted values for both experiments were calculated using Equation 1 and Equation 2 for EVA and xylene, respectively. From the table, both experiment 1 and 2 show that the residual value were very close and the difference is small. It means that the predictive equations are reliable to use for further assumption.

| Experiment | Wax Inhibitor | Wax Deposit (g) | Residue |
|------------|---------------|----------------|---------|
|            | Actual        | Predicted      |         |
| 1          | EVA           | 0.8741         | 0.8373  | 0.037   |
| 2          | Xylene        | 0.2826         | 0.2035  | 0.079   |

Experimental condition:
Rotation speed:400 rpm, duration of experiment:2 hour, cold finger temperature:10°C,
4. Conclusion
Lesser wax deposit was obtained (0.0055 g) when optimal conditions of 2 h duration with 600 rpm rotation speed at 15°C cold finger temperature with EVA as wax inhibitor were used. Based on the analysis of variance (ANOVA), the model is designated with high determination coefficient ($R^2 = 0.8221$). Cold finger temperature (B) is identified as the most influencing factor of wax deposition. The combination factor of A and D shows the best interaction effect and has the highest percentage of contribution towards wax deposition. Equation 1 and Equation 2 generated from FFD experiment is useful to researcher to predict wax deposit for future research.

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