IOP Conference Series: Materials Science and Engineering

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To cite this article: N Ridzuan *et al* 2020 *IOP Conf. Ser.: Mater. Sci. Eng.* **736** 022116

IOP Conf. Series: Materials Science and Engineering **736** (2020) 022116 doi:10.1088/1757-899X/736/2/022116

Screening and interaction study of the operating parameter influence the wax formation using design of experiment

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Abstract. Oil and gas industry has been facing the wax deposition issue due to low temperature condition during the transportation of crude oil from offshore to onshore. The most common method to overcome this problem is by injecting polymeric wax inhibitor in the pipeline. The aim of this work is focused on the screening of the factors that affect the wax deposit inhibition using two types of wax inhibitor; which are poly(ethylene-co-vinyl acetate) (EVA) and Petronas-pour point depressant (PPD) through Design Expert software version 7.1.6. The other three factors evaluated include cold finger temperature (5 & 20℃), rotation speed of impeller (100 & 600 rpm) and the duration of experiment (2 & 6 h). Sixteen experiments were run to observe the rate of wax deposition using cold finger apparatus suggested by level of factorial analysis. The results revealed a significant model with R^2 value of 0.989 indicating that 98.9% of the variable response can be explained by the model. From the analysis of variance (ANOVA), factor D, which has the addition of EVA in crude oil was found to be the main factor affecting the wax deposit inhibition followed by duration of the experiment, rotation speed, cold finger temperature and Petronas-PPD factors. Furthermore, the interaction between factor D (EVA) and factor E (Petronas –PPD) shows the greatest influence to reduce wax deposition. The best configurations to minimize the amount of wax deposit were found using standard order No 1 which is at 5℃, 100 rpm and for the duration of 2 h using EVA as wax inhibitor. The amount of wax deposit measured is 0.018g. Hence, it can be concluded that factor D, and interaction between factors D and E need to be focused in controlling the parameters to minimize wax deposition.

1. Introduction

Crude oil or often called as ‘black gold’ is an unrefined petroleum product that is composed of hydrocarbon deposits and organic materials. The colour of the crude oil can vary from black to yellow depending on its hydrocarbon composition and it has a range of viscosity. It is a non-renewable source, which means that it cannot be replaceable naturally and therefore it is a limited source. There are some basic parameters that can affect the wax deposition to occur such as the temperature and the flow rate. Wax consists of long straight-chain molecules that become tangled and form wax deposit when the crude oil temperature is decreases [1]. Waxes are a complex mixture of solid (at ambient temperature) hydrocarbons which consist mainly of paraffin hydrocarbons with a small amount of naphthenic and aromatic hydrocarbons as well as polar compounds [2-4]. This shows that temperature is the most critical factor in the wax crystals formation. The wax appearance temperature (WAT) or the cloud point is the temperature at which the first wax crystal forming in the oil is measured using different methods.

The production of crude oil has been extended into deep-waters with extreme environment that happened at a cold temperature and would cause the wax deposition to occur. This is especially problematic for pipelines in deep-sea environments, as even in relatively warm climates, the water temperature will be on the order of 5℃ [5]. Wax deposition occurring in the oil-gas pipeline system reduces the effective flow area, increases the flow resistance and reduces the transportation ability of pipelines, and even leads to a potential blockage [6-7]. The solubility of paraffin waxes is not only sensitive to temperature variation, but also an integration of physiochemical properties of the crude and other operation factors in production system [7].

The production costs may increase significantly due to the remediation techniques, such as pipeline insulation, pigging, and heating [8]. Magnetic treatment technology, a representative of physical treatment technique, was applied in the oilfields to decrease viscosity and inhibit wax deposition since 1980s [21]. When the transportation of waxy crude oil is performed below the WAT, the usual remediation technique used is regular pigging [9]. The more cooperative method that is effective to determine the interaction between two or more factors on a response is using full factorial design (FFD). This method is considered more than one factor at a time and require only few set of experiments. However, the one factor at time (OFAT) is favoured by non-experts, especially in situations where the data is cheap and abundant. However, this technique is not able to be used for at all possible combinations of factor concurrently. The weakness of this technique is that the interactions between all the factors considered cannot be explored. Thus, it is important to determine the impact of all possible factors towards the wax deposition. In order to optimise a response that is influenced by several independent factor, a new approach to replace OFAT method could be studied [10]. Due to the lack of information of the dominant factors that attribute to the wax formation, the relationships between factors that affects the deposition of wax in oil transportation need to be built. The relationship is expected to be able to predict the uncertainties of wax deposition amount within the different range of experimental conditions and values. 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 Raw materials

The raw crude oil was supplied by Petronas Refinery from Kerteh Terengganu, Malaysia. The EVA polymer which contains 25% vinyl acetate was purchased from Sigma Aldrich, whereby Petronas-PPD was supplied by Petronas. Table 1 shows the physical properties of the crude oil sample. The raw material and viscosity analysis procedure used in this study were identical with those reported in previous paper, where their detailed description can be found [11].

Table 1. Physical properties of crude oil.

Physical Properties	Value
Viscosity mPa.s at 35℃	4.67
WAT, ℃	11
Pour Point, ℃	28

2.2 Experimental set-up for factorial analysis

Five factors were used for factorial design analysis; which are cold finger temperature (5 & 20℃), rotation speed of impeller (100 & 600 rpm) and duration of experiment (2 & 6 h), including two types of polymer wax inhibitor which are EVA and Petronas-PPD. The design of experiment was performed where all the factors were randomised. From the factorial analysis, the most influential factor for wax inhibition can be determined; and the interactions between factors and how it affects the amount of wax deposit can be evaluated. Table 2 shows the lower and higher values represented by the actual values.

Table 2. Values for lower (−) and higher (+) levels of the factors investigated in factorial design.

Factor	Code	(−) Low Level Value	(+) High Level Value
Rotation speed (rpm)	A	100	600
Cold finger temperature (°C)	B	5	20
Duration of experiment (h)	C	2	6
Poly(ethylene-co-vinyl acetate) (EVA)	D	NO	YES
Petronas-PPD	E	NO	YES

2.3. Cold finger analysis

Figure 1 shows the cold finger apparatus set-up to study the rate of wax deposition. 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. A 300 mL of crude oil were measured and transferred into the stainless-steel jar for the rate of deposition analysis. The 16 experiments were run based on the different setting designed by design expert simulator. A total of 5 mL amount of wax inhibitor was injected in the cold finger. At the end of the experiment, the wax deposit was scrapped and being weighed for further analysis. The experiment was repeated for three time for accuracy.

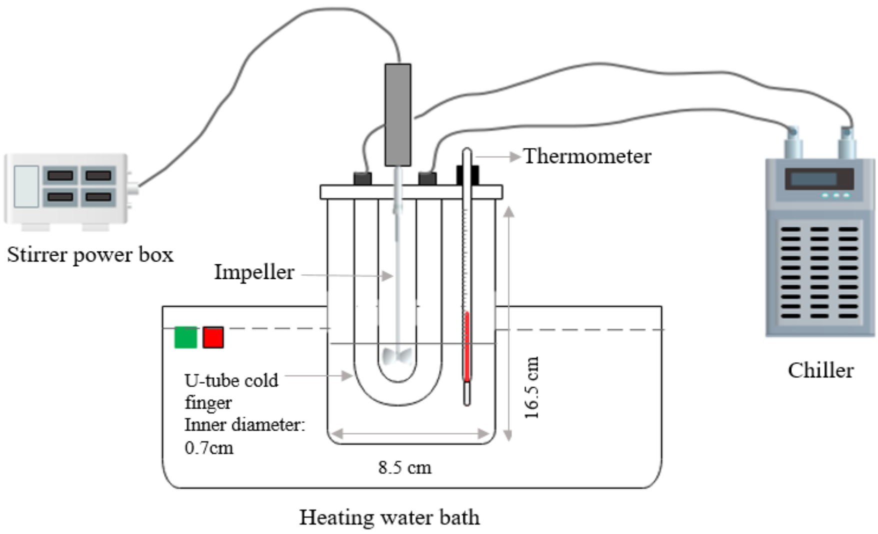

Figure 1. Cold finger apparatus set-up.

2.4 Design of experiment

Table 3 shows a total of 16 run and each row represented the specified condition for each run. The experiments sequence was sorted in standard order to prevent bias from the system of uncontrolled factors. The statistical data were analysed using the software called Design Expert 7.1.6. Equation 1 was used to calculated the paraffin inhibition efficiency (%PIE):

$$\%PIE = \frac{w_f - w_t}{w_f} \times 100\% \quad (1)$$

where w_f is the total wax deposit been weight without the addition of wax inhibitor (in grams) and w_t is the total wax deposit been weight with the addition of wax inhibitor (both in gram)

Table 3. Screening design according to standard order.

Standard Order	A : Cold Finger temperature (°C)	B : Rotation speed (rpm)	C : Duration (hr)	D : EVA	E : Petronas-PPD
1	5.00	100.00	2.00	Yes	No
2	20.00	100.00	2.00	Yes	Yes
3	5.00	600.00	2.00	Yes	Yes
4	20.00	600.00	2.00	Yes	No
5	5.00	100.00	6.00	Yes	Yes
6	20.00	100.00	6.00	Yes	No
7	5.00	600.00	6.00	Yes	No
8	20.00	600.00	6.00	Yes	Yes
9	5.00	100.00	2.00	No	Yes
10	20.00	100.00	2.00	No	No
11	5.00	600.00	2.00	No	No
12	20.00	600.00	2.00	No	Yes
13	5.00	100.00	6.00	No	No
14	20.00	100.00	6.00	No	Yes
15	5.00	600.00	6.00	No	Yes
16	20.00	600.00	6.00	No	No

3. Results and discussion

3.1. Analysis of variance (ANOVA)

Table 4 shows the total mass of wax collected with the combination of five factors at different operating conditions according to standard order sequence. Table 5 shows the results for ANOVA where the significant terms correspond to the codes A, B, C, D and E. This result reveals that factor D gave the most significant effect to the expression of wax deposit ($P < 0.0500$) as compared to other factors as it also yields a high percentage of contribution. From the responses, the mean value of wax deposited was 0.20 g with 0.045 standard deviation. The significance of the model equation was evaluated using the F test ANOVA. From table 5, the results revealed a significant model with R^2 value of 0.989 indicating that 98.9% of the variable response can be explained by the model. Whereas, for the coefficient of determination, which is the adjusted R^2 was calculated and determined as 0.9448. This value indicates a good agreement between the predicted and the observed values of wax deposit formation.

Factor D shows the highest percentage of contributor value, with a percentage of 41.12%, in affecting the amount of wax deposit followed by duration of the experiment, rotation speed, the differential temperature (ΔT) was the main factor affecting the rate of wax deposition. Wax deposit formed due to the decrease in the dissolving ability of components in the crude oil and an increase in the precipitating ability [12]. Therefore, it is tend to increase the precipitation potential of wax molecule on the cold surface and strengthen the driving force of the wax molecule diffusion from the bulk oil to the cold surface. According to Lim et al [12], the experimental duration is proportional to the increase of wax deposit on the cold surface. Once the long chain n-paraffin molecules continuously diffuse from the crude sample to the gelation layer, the wax ageing will keep continuous occur.

Table 4. Screening design according to standard order.

Standard order	Weight of Wax (g)	Standard order	Weight of Wax (g)
1	0.018	9	0.031
2	0.022	10	0.246
3	0.039	11	0.345
4	0.023	12	0.147
5	0.138	13	0.572
6	0.043	14	0.249
7	0.054	15	0.504
8	0.327	16	0.467

Table 5. Analysis of variance (ANOVA).

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	% contribution
Model	0.54	12	0.045	22.40	0.0132	
A-cold finger	1.9E-03	1	1E-003	0.97	0.3966	0.36
B-rotation speed	0.022	1	0.022	10.70	0.0467	3.94
C-duration	0.14	1	0.14	68.32	0.0037	25.13
D-EVA	0.22	1	0.22	111.78	0.0018	41.12
E-Petronas-PPD	6.0E-03	1	6E-003	3.00	0.1815	1.11
AD	0.016	1	0.016	8.05	0.0658	2.60
AE	3.6E-03	1	3E-003	1.83	0.2686	0.67
BD	1.2E-03	1	1E-003	0.64	0.4837	0.23
BE	0.020	1	0.020	9.99	0.0509	3.67
CD	0.020	1	0.020	9.85	0.0518	3.62
CE	0.014	1	0.014	7.01	0.0772	2.94
DE	0.074	1	0.074	36.70	0.0090	13.50
Residue	6.0E-03	3	2E-003			
Cor Total	0.55	15				

The predictive equations are based on the fitted model for the response with all the factors in coded form (–1,1) expressed in Equation 2:

$$Wax\ deposit = +0.20 - 0.011A + 0.037B + 0.093C + 0.12D + 0.019E - 0.032AD - 0.015AE + 8.938E - 003BD - 0.035BE + 0.035CD - 0.030CE + 0.068DE \quad (2)$$

3.2. Interactions between factors on wax deposit

Multiple factors interact in affecting the results was identified when the result shows one factor is depending to the second factor. Figure 2 shows the interaction between factor of the experiment duration, C and EVA wax inhibitor, D. The amount of wax deposit without the addition of EVA was increased once the duration of the experiment is extended until at 6 h. The less slope was observed for the experiment without the addition of EVA. The CD interaction is much better with the addition of EVA in reducing the wax formation. This interaction possibly occurs because both the factors contributed the most to wax deposition compared to other factors. The duration factor contributes to the growth of wax as the experiments last much longer towards the ageing effect.

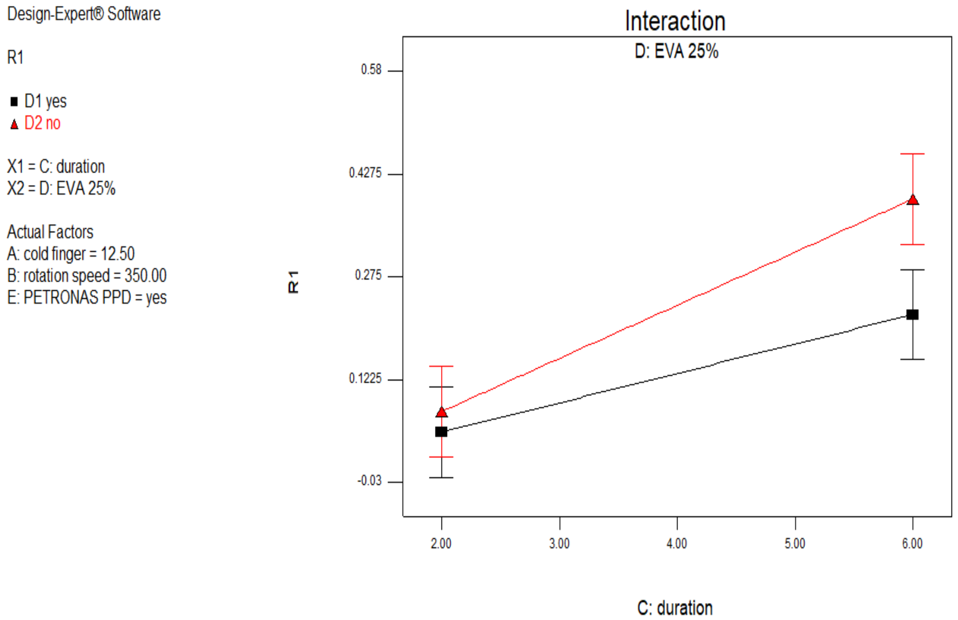

Figure 2. Interaction graphs between factors of duration experiment (C) and EVA (D) on wax deposit.

Figure 3 shows the interaction graphs between factors of duration, C with Petronas-PPD, E which contributes 2.94%. From this graph, it shows that with the presence of Petronas-PPD, the amount of wax deposit increases at 6 hr. Whereas, without its present, the slope become less steeper indicating that the wax formation is reduced. Therefore, this shows that the addition of Petronas-PPD is not efficient in reducing the amount of wax.

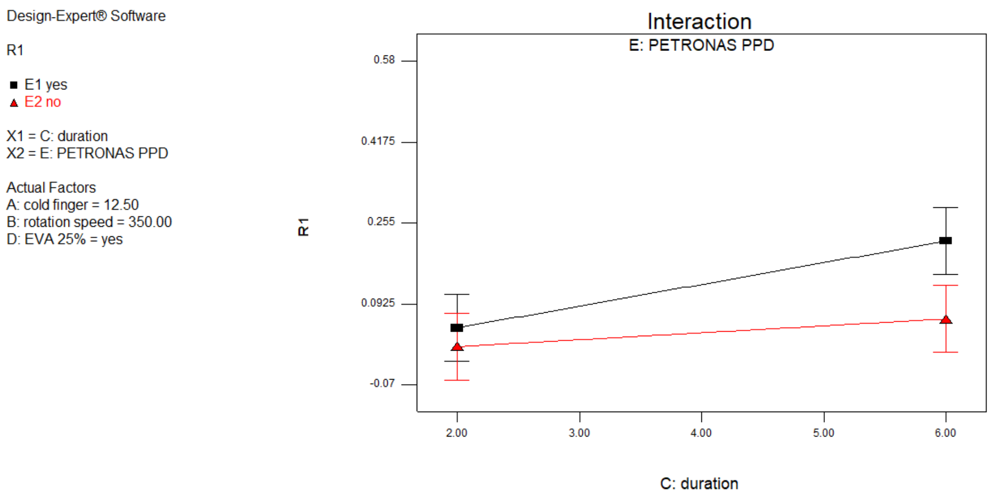

Figure 3. Interaction graphs between factors of duration experiment (C) and Petronas- PPD (E) on wax deposit.

Moreover, there was a cross between the plot of the two-line factors was found in figure 4. The CE interaction exhibited the highest interaction effect, whereby it contributed 13.5% influence on wax deposition. This indicates that by combining both polymer wax inhibitors can improve the wax deposit reduction. The slope for the addition of Petronas-PPD was found more steeper compare to the absence of inhibitors. The combination of both polymers can successfully improve the ability to reduce the wax formation. It is believed that this is due to the functional group in the polymer.

The mechanism of combining the EVA and Petronas-PPD polymer on reducing wax deposition is not fully apprehended but it is proposed that this combination may act as nucleation site for wax crystals to precipitate producing bigger and more compact wax flocs affecting the wax crystal and oil interface area. Reduction in wax crystal/oil interface area suppresses the interaction between wax crystals thus preventing the agglomeration of wax crystals [13-14]. Vinyl acetate (VA) in EVA has two active oxygen atoms that play an important role in affecting and reducing the crystallisation process.

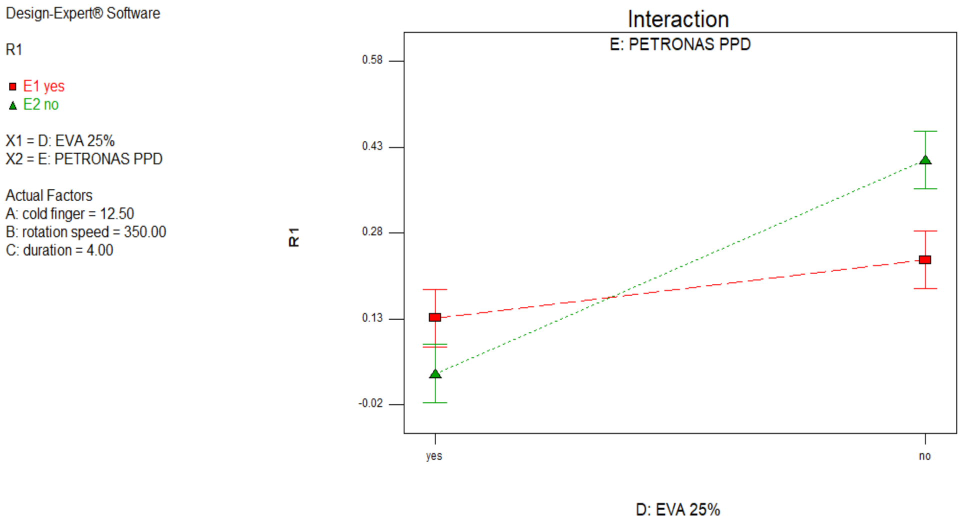

Figure 4. Interaction graph between factors EVA (D) and Petronas- PPD (E) on wax deposit.

4. Conclusions

EVA was found to have the highest contribution of inhibition efficiency of wax deposit as compared to Petronas-PPD. The most effective minimization of the amount of wax formation which is 0.018 g was found to be at the operating parameters of 5℃, 100 rpm, and for a duration of 2 h using EVA. While the DE interaction shows the highest percentage of contribution, which is 13.5%.

Acknowledgments

The authors acknowledge the financial support from Universiti Malaysia Pahang (RDU170352) and the Ministry of Education (MOE), Malaysia, through the Fundamental Research Grant Scheme (RDU 1901060 with reference number FRGS/1/2018/TK02/UMP/03/1) for having this research project to be successfully conducted. We also thank to Petronas Penapisan Terengganu, Malaysia for providing the crude oil samples for the research project.

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