FORMULATION OF POLYMERIC INHIBITOR FOR VISCOSITY REDUCTION OF CRUDE OIL

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ABSTRACT. *Generally, waxes and asphaltenes are classified as solid category which involved with deposition of high-molecular-weighted compounds along pipelines which leads to production issues. This study presents the effect of different mixture concentration consisting of copolymer and solvent on crude oil viscosity in order to find a solution for reduction of wax and asphalthene deposition along the surface of pipelines. There were two proportions used which are ethylene-vinyl acetate 25 (EVA 25), methylcyclohexane (MCH) and paraxylene as first proportion and EVA 40, MCH and paraxylene as second proportion. EVA is a polymer that comprises of linear chain of polyethylene fragment and vinyl acetate molecule which has the ability in controlling the size of formed wax crystals. Laboratory experiments were designed by response surface methodology (RSM) specifically using central composite design (CCD) to formulate ratio and analyzed optimum percentage composition of mixture to obtain a good model. The optimum parameters were 10.02% of EVA 25, 10.00% of MCH and 79.98% of paraxylene for first proportion and 10.00% of EVA 40, 45.78% of MCH and 44.22% of paraxylene for second proportion to minimize the viscosity of crude oil.*

KEYWORDS: Crude Oil, Ethylene-vinyl acetate, Methylcyclohexane, Paraxylene, Wax, Response surface methodology (RSM)

INTRODUCTION

Wax defined as hydrocarbon containing 20 to 40 carbon atoms in a chain where those structures consists of few structural types including straight chain, branched chain and cyclic chain (Yao *et al.*, 2016). For asphaltene, it defined as saturated hydrocarbon which consists of carbon, nitrogen, oxygen, hydrogen and sulphur (Ariza-León *et. al.*, 2014). Both of wax and asphaltene involved in stability of crude oil at distributed state. Instability of temperature will caused of coagulation of wax and flocculation of asphaltene during the process of transportation of crude oil along pipelines from oil rig to the shore.

In crude oil, wax molecules exist in liquid phase where the molecules undergo crystallization when it exposed to cold condition (Oh *et. al.*, 2009). Drastic change of solubility of waxes is the main reason for deposition of waxes where this condition cause wax molecules crystallize and become a solid gel, decreasing cross sectional area of pipelines (Bai and Zhang, 2013). Thus, changes of solubility of waxes result in the formation of gel which minimizes the transportation efficiency.

There are several assumptions made because of complex nature of waxy crude oil (Aiyejina *et. al.*, 2011; Anisuzzaman *et. al.*, 2017 a,b; Luthi, 2013; Kralova et al., 2011). Those assumptions are used to highlight the oil properties that affect their flow ability in production, refining and transportation of crude oil. There are lots size of pumps and pipeline system produced by oil industries which been led by those assumptions. During cooling process, waxy crude oil encounter thermal shrinkage where the gas voids appear while the fluid turn into multiphase (Singh *et al.*, 2008). Voids in the gel occur because of decrease in volume of waxy-oil gel during that process (Chala *et al.*, 2015, Chala *et al.*, 2016; Lionetto *et al.*, 2007; Kané *et al.*, 2003). Those conditions caused in reduction of pressure which break the gel along pipelines due to non-uniform gel formation (Wachs *et al.*, 2009; Sakthipriya et al., 2015).

As time goes on, the potential of blockage on the pipelines due to wax deposition will increase during transportation of crude oil along the pipelines from the oil rig to the shore. This problem may occur due to wax coagulation which will convert to wax crystals (Anisuzzaman *et. al.*, 2019; Anisuzzaman *et. al.*, 2018; Pendersen and Rønningsen, 2003). Since the precipitations along the pipelines will result in decreasing space of inner pipelines during transportation of crude oil, it may cause blockage of path. This blockage problem will affect the transportation, production processes and cost for maintenance of the industry (Coto *et al.*, 2014). Hence, in order to prevent or reduce the problem of wax deposition along the pipelines, few modifications of flow properties of the crude oil should be implemented.

Therefore, the objective of this study was to formulate a mixture of ethylene-vinyl acetate (EVA), MCH and paraxylene at different ratios and tested for flow properties improvement of crude oil viscosity. This study was conducted using EVA with two concentrations consisting of EVA 25 and EVA 40. Meanwhile, MCH was used as a solvent for the polymer and paraxylene as the asphaltene dispersant or inhibitor. Besides, this study was also conducted using response surface methodology (RSM) specifically central composite design (CCD) type to optimize percentage composition of the sample in order to enhance flow properties. According to previous research, RSM was used to study the effect of shear rates on improvement flow properties of crude oil.

MATERIALS AND METHODS

Materials and chemicals

The crude oil used in this study was Malaysian crude oil, specifically taken from Sabah platform. Malaysian crude oil generally has very little amount of wax but high content of asphaltene (Ridzuan *et al.*, 2016; Anisuzzaman *et al.*, 2017). In order to study the effect of concentration of polymer on viscosity of crude oil, one copolymer being selected which is EVA with two concentrations consisting of EVA 25 and EVA 40.

Preparation of chemicals and crude oil

The crude oil was heated in an oven at 90°C overnight to melt any primitively formed wax crystals and asphaltene agglomerates. The spindle of viscometer, measuring cylinder and pipette was heated first for about 60°C to avoid the precipitation of wax at the point of contact between the hot crude oil and cold apparatus. Prior to mixing of inhibitor, EVA, MCH and paraxylene were heated in a water bath at 50°C (Anisuzzaman *et. al.*, 2017 a,b).

Preparation of inhibitor

In the preparation of the inhibitor, the reaction was conducted at 90°C. The individual chemicals, EVA, MCH and paraxylene were measured separately of its respective volume and weightage in accordance to manipulate percentage composition. The total volume of inhibitor used is 0.1 mL. Care was taken to replace the micropipette tube for the both chemicals to avoid contamination. Polymer was added with MCH and paraxylene and heated at 90°C to melt the polymer. When the inhibitor has completely melted, the crude oil was poured into the measuring cylinder. The sample was shaken for about 30 seconds to allow the mixture of the inhibitor and crude oil. Then, the samples were placed in the oven for 15 min to allow the reaction to take place.

Experimental procedure

The rheological measurements were carried out using viscometer (Brookfield Programmable Viscometer DV-II + PRO) at fixed temperature which is 25.6^{o}C. The temperature of 25.6^{o}C was chosen to stimulate the real average temperature of the seabed. The amount of each chemical had been fixed to 0.3 g of total weight respectively. The crude oil was removed from the oven and allowed to decrease about 5^{o}C from initial temperature before measurement of viscosity using viscometer. Meanwhile, the rotational speed of spindle of viscometer also had been fixed to 100 rpm (Anisuzzaman *et. al.,* 2017 a,b).

Response surface methodology (RSM) modeling

In this work, CCD of RSM was used for data analysis (Bono *et. al.,* 2014, Torgut et al., 2017). The implementation of CCD was started with design of experiment, followed by data analysis. This method was used instead of one-to-one factor in order to reduce the number of experiments. A three level, three-factor CCD has been employed in order to obtain optimum composition for the reduction viscosity of crude oil. The factors or the independent variables studied were the percentage of EVA, percentage of MCH and percentage of paraxylene and the response was viscosity.

RESULTS AND DISCUSSION

In this study, total of experimental runs required suggested by CCD was 16 for each set. Weight and volume of each chemical was calculated in accordance to ratio given in order to find viscosity of each sample. The data obtained from CCD is shown in Table 1.

Table 1: Percentage ratio from central composite design (CCD)

Run	Factor 1 A: (MCH) (%)	Factor 2 B: (EVA) (%)	Factor 3 C: Paraxylene (%)
1	19.7858	51.4160	28.7985
2	79.9914	10.0000	10.0085
3	10.0000	42.5300	47.4700
4	45.7767	10.0000	44.2232
5	10.0000	10.0170	79.9827
6	23.1321	20.0060	56.8621
7	10.0000	42.5300	47.4700
8	31.1315	58.8680	10.0000
9	33.2855	33.3800	33.3342
10	49.1917	40.8080	10.0000
11	45.7767	10.0000	44.2232
12	10.3279	70.0000	19.6720

13	79.9914	10.0000	10.0085
14	58.2104	19.0331	22.7565
15	10.3279	70.0000	19.6720
16	10.0000	10.0172	79.9827

There were few properties observed to design good model for this study. The properties including design summary, model development, ANOVA, model graphs and analysis summary. For ANOVA, analysis included variance, fit statistics and coefficient of model of chemical proportion.

Model development

A quadratic model was used to express the responses as a function of independent variables. The test of statistical significance was performed on the total error criteria, with a confidence level of 95%. The significant terms in the model were found using analysis of variance (ANOVA) for each response. The adequacy of the model was confirmed using R^2 and the adjusted R^2 values. The numerical optimization techniques of the software were used for simultaneous optimization of the multiple responses. Under this optimization, the desired goals for each variable and response were chosen within a range.

The variables and responses of viscosity obtained from experiments are listed in Tables 2 and 3. The experimental data were used to calculate the coefficients of the quadratic equation. The ANOVA of each case and the diagnostic plot was presented. Based on ANOVA analysis, RSM stated that the data entry was valid since most of the requirements to get good model were achieved. For variance, the p-value should be less than 0.005 while Lack of Fit value should be greater than 0.1. For fit statistics, value of R^2 should not be less than 0.75 and adjusted R^2 should have similarly high value to R^2. In addition, adjusted R^2 and predicted R^2 should be within 0.2.

Table 2: Data entry in RSM for proportion of EVA 25, MCH and paraxylene

Run	Factor 1 A: (MCH) (%)	Factor 2 B: (EVA) (%)	Factor 3 C: Paraxylene (%)	Response 1 Viscosity
1	19.7858	51.4160	28.7985	12
2	79.9914	10.0000	10.0085	6
3	10.0000	42.5300	47.4700	12
4	45.7767	10.0000	44.2232	6
5	10.0000	10.0170	79.9827	6
6	23.1321	20.0060	56.8621	12
7	10.0000	42.5300	47.4700	12
8	31.1315	58.8680	10.0000	12
9	33.2855	33.3800	33.3342	12
10	49.1917	40.8080	10.0000	6
11	45.7767	10.0000	44.2232	6
12	10.3279	70.0000	19.6720	12
13	79.9914	10.0000	10.0085	6
14	58.2104	19.0331	22.7565	6
15	10.3279	70.0000	19.6720	12
16	10.0000	10.0172	79.9827	12

Table 3: Data entry in RSM for proportion of EVA 40, MCH and paraxylene

Run	Factor 1 A: (MCH) (%)	Factor 2 B: (EVA) (%)	Factor 3 C: Paraxylene (%)	Response 1 Viscosity
1	19.7858	51.4160	28.7985	18
2	79.9914	10.0000	10.0085	18
3	10.0000	42.5300	47.4700	6
4	45.7767	10.0000	44.2232	12
5	10.0000	10.0170	79.9827	6
6	23.1321	20.0060	56.8621	12
7	10.0000	42.5300	47.4700	6
8	31.1315	58.8680	10.0000	12
9	33.2855	33.3800	33.3342	6
10	49.1917	40.8080	10.0000	12
11	45.7767	10.0000	44.2232	6
12	10.3279	70.0000	19.6720	12
13	79.9914	10.0000	10.0085	6
14	58.2104	19.0331	22.7565	6
15	10.3279	70.0000	19.6720	12
16	10.0000	10.0172	79.9827	6

Statistical analysis of the design model

Table 4 shows the analysis of variance of proportion MCH, EVA 25 and paraxylene. On the other hand, Table 5 shows the analysis of variance of proportion MCH, EVA 40 and paraxylene.

Table 4: Analysis of variance of proportion MCH, EVA 25 and paraxylene

Source	Sum of squares	df	Mean square	F-value	p-value	
Model	372.62	3	124.21	39.45	<0.0001	significant
A-MCH	12.58	1	12.58	4.00	0.0629	
B-EVA 25	245.58	1	245.58	77.99	<0.0001	
C-paraxylene	118.68	1	118.68	37.69	<0.0001	
Residual	50.38	16	3.15			
Lack of fit	32.38	8	4.05	1.80	0.2120	not significant
Pure error	18.00	8	2.25			
Cor total	423.00	19				

Table 5: Analysis of variance of proportion MCH, EVA 40 and paraxylene

Source	Sum of squares	df	Mean square	F-value	p-value	
Model	366.90	3	122.30	9.05	0.0010	significant
A-MCH	162.34	1	162.34	12.01	0.0032	
B-EVA 40	215.41	1	15.93	15.93	0.0011	
C-paraxylene	20.62	1	1.53	1.53	0.2346	
Residual	216.30	16				
Lack of fit	126.30	8	1.40	1.40	0.3215	not significant
Pure error	90.00	8				
Cor total	583.20	19				

Based on analysis in table 4, model F-value of 39.45 implies the model is significant. For p-value, the model is significant because the value is less than 0.005. In first proportion, EVA 25 and paraxylene are significant model terms. Meanwhile, MCH is not significant since the value is greater than 0.1. For lack of fit, F-value, the good model is a non-significant. From the result, the lack of fit, F-value is 1.80 which the value implied not significant relative to pure error. For second proportion analysis, model F-value of 9.05 implies the model is significant (Table 5). For p-value, the model is significant because the value is less than 0.005. In this experiment, MCH and EVA 40 are significant model terms. Meanwhile, paraxylene is not significant since the value is greater than 0.1. For lack of fit, F-value, the good model is a non-significant. From the result, the lack of fit, F-value is 1.40 which the value implied not significant relative to pure error.

Tables 6 and 7 showed the fit statistics for proportion MCH, EVA 25 and paraxylene and MCH, EVA 40 and paraxylene, respectively.

Table 6: Fit statistics for proportion MCH, EVA 25 and paraxylene

Standard deviation	1.77	R^2	0.8809
Mean	7.50	Adjusted R^2	0.8586
Coefficient of variation (%)	23.66	Predicted R^2	0.8046
		Adequate precision	16.2558

Table 7: Fit statistics for proportion MCH, EVA 40 and paraxylene

Standard deviation	3.68	R^2	0.6291
Mean	7.80	Adjusted R^2	0.5596
Coefficient of variation (%)	47.14	Predicted R^2	0.4242
		Adequate precision	7.5073

Based on both fit statistics above, the first proportion showed that R^2 value of 0.8809, indicating that the model can explain 88.09% of the data variation and only 11.91% of total variations were not explained by the model. R^2 value should not be less than 0.75 (Le Man *et al.*, 2010). According to Koocheki *et al.* (2009), the model can be a good one if value of adjusted R^2 is a similarly high value. Furthermore, adjusted R^2 and predicted R^2 should be within 20% to be in reasonable agreement (Rai *et al.*, 2016). For first proportion, adjusted R^2 value is 0.8586 while predicted R^2 value is 0.8046. Therefore, the model is highly significant because the experimental and predicted values of monomer conversion are in a good agreement. For second proportion, R^2 value was 0.6291. This model can explain 62.91% of the data variation and only 37.09% of total variations were not explained. In this experiment, adjusted R^2 value is 0.6291 while predicted R^2 value is 0.4242. The model is highly significant too because the experimental and predicted values of monomer conversion are in a good agreement.

Tables 8 and 9 show the coefficient of model of proportion MCH, EVA 25 and paraxylene and MCH, EVA 40 and paraxylene, respectively

Table 8: Coefficient of model of proportion MCH, EVA 25 and paraxylene

Coefficient of model	Equation
Actual	Viscosity = -3.553 ×10-6 + 0.0312A +0.1517B +0.0993C
Coded	Viscosity = 14.14+ 1.60A +7.58B + 4.96C

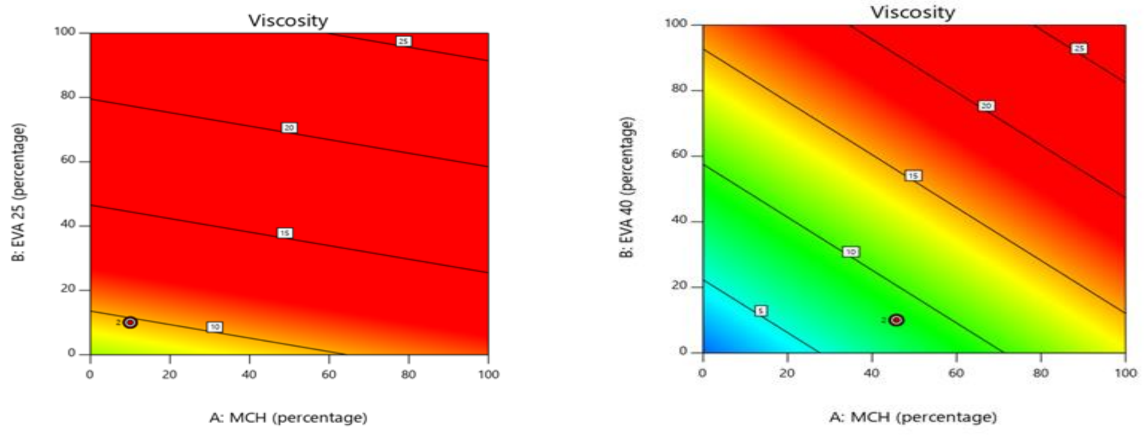
Table 9: Coefficient of model of proportion MCH, EVA 40 and paraxylene

Coefficient of model	Equation
Actual	Viscosity = -9.88×10^{-6}+ 0.1146A +0.1421+0.0414C
Coded	Viscosity = 14.90+ 5.73 +7.10B + 2.07C

The coded equation used to identify the relative impact of the factors by comparing the factor coefficients. Meanwhile, actual equation make predictions about the response for given levels of each factor. The positive signs in the models signify synergetic effects of factor while the negative sign indicates antagonistic effect.

Model graphs

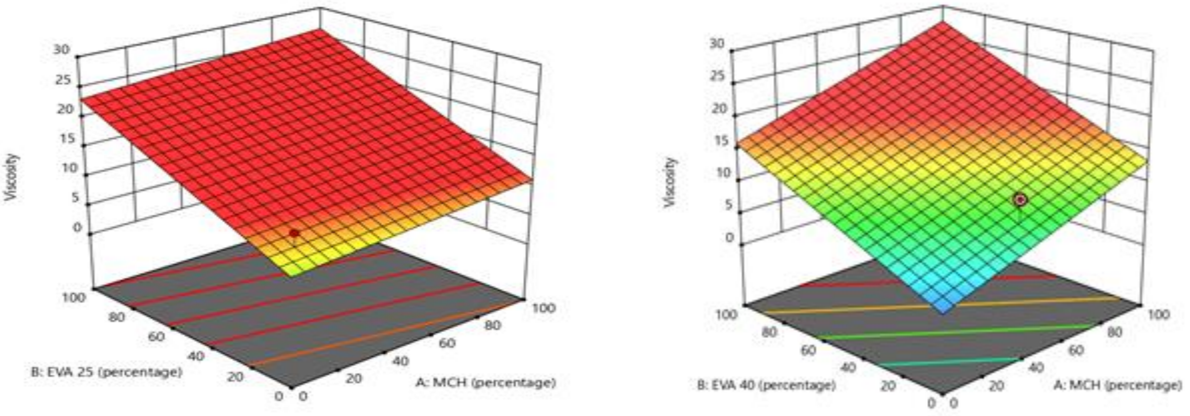
Figure 1 (a, b) shows the contour plot of viscosity (%) vs EVA 25 (%), MCH (%) and EVA 40 (%), MCH (%), respectively. The contour plot depicted that the optimum ratio was determined by mixture of high percentage of paraxylene, low percentage of EVA 25 and low percentage of MCH. Based on Figure 1 (b), contour plot of second proportion, the optimum ratio was determined by mixture of medium percentage of paraxylene, low percentage of EVA 40 and medium percentage of MCH.



(a) (b)

Figure 1: Contour plot of viscosity (%) vs (a) EVA 25 (%), MCH (%) (b) EVA 40 (%), MCH (%)

Figure 2 (a,b) shows 3D graphs for optimization EVA 25, MCH and paraxylene and EVA 40, MCH and paraxylene, respectively. Figure 2 (a) depicted the 3D graphs for first proportion based on result of contour plot. For optimization, CCD selected experimental run 16 as the optimum ratio to obtain minimum viscosity of crude oil which is 6.0 cP. Figure 2 (b) depicted the 3D graphs for first proportion based on result of contour plot. For optimization, CCD selected experimental run 4 as the optimum ratio to obtain minimum viscosity of crude oil which is also 6.0 cP.



(a) (b)

Figure 2: 3D graphs for optimization (a) EVA 25, MCH and paraxylene (b) EVA 40, MCH and paraxylene

Based on Figures 1 and 2, for first proportion, contour plots and 3D graphs depicted that high percentage of paraxylene with low percentage of EVA and low percentage of MCH lead to low viscosity value. Meanwhile for second proportion, lowest viscosity value can be obtained with medium percentage of paraxylene with low percentage of EVA and medium percentage of MCH. This can be observed through the design point location in four figures above where it located far away from red area which signify high viscosity. For optimization, CCD selected experimental run 16 in first proportion consist of 10.02% of EVA 25, 10.00% of MCH and 79.98% of paraxylene as the optimum ratio for reduction of crude oil viscosity. The viscosity obtained was 6.0 cP.

Meanwhile, optimum ratio for second proportion was 10.00% of EVA 40, 45.78% of MCH and 44.22% of paraxylene which in experimental run 4. The viscosity obtained was also same with first proportion which was 6.0 cP. Both of these values had desirability value of 1 which signified the most desirable predicted responses on the dependent variables.

CONCLUSIONS

CCD in RSM had provided valid ratio for chemical proportion consists of EVA 25 and EVA 40, MCH and paraxylene. The validity has been tested during analysis in RSM since the model obtained was significant in both proportions. Every ratio formulated by CCD has been tested in order to find optimum ratio for viscosity reduction of crude oil. Based on analysis in RSM, the optimum viscosity value which is 6.0 cP which can be obtained from proportion of high percentage of paraxylene, low percentage of EVA and low percentage of MCH for first proportion. For second proportion, the optimum viscosity value was also same with first proportion which was 6.0 cP. The value can be obtained from proportion of medium percentage of paraxylene with low percentage of EVA and medium percentage of MCH. The optimum parameters were 10.02% of EVA 25, 10.00% of MCH and 79.98% of paraxylene for first proportion and 10.00% of EVA 40, 45.78% of MCH and 44.22% of paraxylene for second proportion to minimize the viscosity of crude oil. The obtained quadratic regression model is very adequate based on ANOVA test. For comparison between experimental optimization and RSM optimization, the data obtained depicted that both optimization has value of 6.0 cP as the optimum value for viscosity reduction of crude oil. In conclusion, in this study RSM specifically CCD type was used to optimize percentage composition of the sample in order to enhance flow properties and transportation of crude oil.

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