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Optimization of Wax Deposition in a Sub-Cooled Pipeline using Response Surface Methodology

Adeyanju Olusiji Ayoade and Oyekunle, Layioye Ola (Chemical and Petroleum Engineering Department, University of Lagos.)

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Abstract

Key factors affecting wax deposition in sub-cooled pipelines (wall (coolant) temperature (A), inlet oil temperature (B), and the percentage of wax inhibitors in the crude (C), and oil flow rate (D)) were experimental studied using the fabricated flow loop rig designed to simulate the flow of relatively higher temperature crude oil in sub-cooled pipeline.

In an effort to investigate the possibility of minimizing the wax deposits volume in a flow of crude oil in sub-cooled pipeline, response surface methodology (RSM) was used to evaluate the individual and interactive effects of four variables affecting the wax deposits process using central composite design (CCD). It was observed that for the crude oil samples the experimental data highly fitted to the predicted data because of the predicted R-squares is in reasonable agreement with the adjusted R-square.

In the laminar-turbulent transition flow regimes (2000 < Reynolds number (Re) > 3000), A, B, C, D,AB, AC, BC, A^2, B^2, and C^2 are significant, as their individual P-value were less than 0.05 by each of the term, while AD, BD, CD and D^2, are insignificant as their P-values were more than 0.05. While in the laminar flow regime (Re < 2000) the results were similar to that observed in the turbulent flow regime except the flow rate term, (D) which was insignificant (with P-values of more than 0.05) due to insignificant effect of shear dispersion and removal in the laminar flow regime.

In applying the response surface methodology central composite design (RSMCCD) in the Minitab-16 software to minimized wax deposit in crude oil flow in sub-cooled pipeline wax deposits were reduced to 64cm^3 in the laminar flow regime and 43cm^3 in the laminar-turbulent transition flow region. The small error percentage between the predicted and actual volume of wax deposit (4.68% in the laminar and 4.55% in the laminar-turbulent transition flow regime: indicated that the software models were valid and accurate in representing the actual experimental values and also in predicting the inhibition of wax deposit within the range studied.

Introduction

Crude oils as a complex mixture consist of saturates (paraffin/wax), aromatics, naphthenes and resin. At reservoir conditions: temperature (70 -150^{o}C) and pressure (50 – 100MPa), these components are dissolved in the crude oil, however at the subsea condition, where the pipelines are harbored, the heavy components mainly the paraffin/wax and asphaltene precipitate out of the crude oil and subsequently dissolved on the pipe wall leading to production and transportation challenges in the pipelines.

Wax accumulation preventive techniques: Thermal, pigging and chemical additions are commonly used in the petroleum industry. Cost incurred by the applications of these preventive techniques can be drastically reduced if the wax deposition processes can be minimized during crude oil production operations (Huang .et al, 2011). A method of using a response surface methodology to minimize the wax deposition process in a flow of single phase crude oil in sub-cooled pipeline is proposed. This technique enables the interactions of the influencing parameters on wax deposition to be evaluated and the objective function to be optimized with a limited number of designed experiments.

In efforts to understand the fundamental variables influencing the wax deposition, studies have been carried out to improve on the knowledge of wax deposition process. Various researchers have simulated wax deposition process in the laboratory; basically the cold finger and the flow loop apparatus have been used. In cold finger experiments, small steel cylinder heat exchangers maintained at lower temperature are immersed in a cylinder containing higher temperature crude oil. The bulk oil and the coolant temperatures are controlled by two different water baths (Paso and Fogler (2004); Jennings and Weispfennig (2005). While most flow loop devices, comprise of the annular pipe with hot crude oil flowing in inner pipe and the coolant liquid flowing in the annulus between the inner pipe and the outer pipe. The flow rates of the crude oils are controlled by regulating the pump, while the oil and coolant temperature are regulated by the two separate water baths (Todi and Deo (2006); Edmonds et al, (2008); Bidmus and Mehrotra (2009); Lashkarbolook et al. (2010); Huang et al. (2011)). Temperature differential between the bulk crude and the cold pipe wall, residence time and flow rate have been identified as the factors influencing wax deposition (Kelechukwu et al, 2010; Towler, 2011). Norland, (2012) used experimental design method to determine the best formulation in inhibiting wax deposit in flow of warm crude oil in sub-cooled pipeline using a group of pour point depressants (PPDs) known as acrylate ester polymers. In this study, Response surface design method was used to design the experiments to be performed to model the wax deposition process, analyze the results, and interpret the interactions between the wax deposition process parameters in flow of single phase crude oil in sub-cooled pipeline.

Materials and Methods

Materials

Crude oil sample whose properties are as tabulated in Table 1, from a subsea fields in the Niger Delta of Nigeria (part of Gulf of Guinea) was used for the study. The crude sample was observed to form stable emulsion with the used wax inhibitors when agitated. The crude oil-inhibitor emulsion was then used for the demulsifier screening. The demulsifiers used for the study: methyl methacrylate, butyl acrylate and acrylic acid were selected on the basis of their solubility in xylene (which is the solvent used in the wax inhibitors preparation).

Table 1: Crude oil properties	
Gravity at 60°F, API°	42.1
Viscosity at 60°F, cp	8.5
Wax Appearance Temp. ,°C	43
Pour point, °C	27
Wax Content %	40

Wax Deposition Experimental Methods

The experimental setup consisted of a flow loop shown in fig. 1. This flow loop was used to perform the wax deposition experiments under the single oil phase conditions.

This flow loop is made of mild steel pipe of length 0.4m with an inside diameter of 0.1cm. The experimental setup has two sections; the test section and the reference section. The crude oil temperature was regulated with a temperature regulator; the oil is pumped through the test and then through reference section after passing through the liquid mass flow-meter along the flow lines. The test section is jacketed with a steel jacket in which cold water pumped from a cooling bath was circulated in the opposite direction to the crude oil flow in the pipeline . The purpose of the test section is to maintain the inner pipe wall at a lower temperature than both the bulk oil temperature and Wax Appearance Temperature (WAT) so as to generate the wax deposit on the inner pipe wall (just like what would be encountered in actual pipelines). The configuration of the reference section is completely identical with the test section. However, contrary to the test section, the inner pipe wall temperature in the reference section is maintained at a higher temperature than the bulk oil temperature to prevent wax deposition by circulating the heated water through the jacket of reference section. Thermocouples (T) were placed both at the inlet and outlet of the test tube and the reference tube to determine the temperatures at each point. Thermocouples were also attached to the cooling water tank and crude oil tank to take temperature reading. The wax deposit in the previous experimental run was ensured to be removed by flowing hot oil for few hours before the commencement of subsequent experimental run.

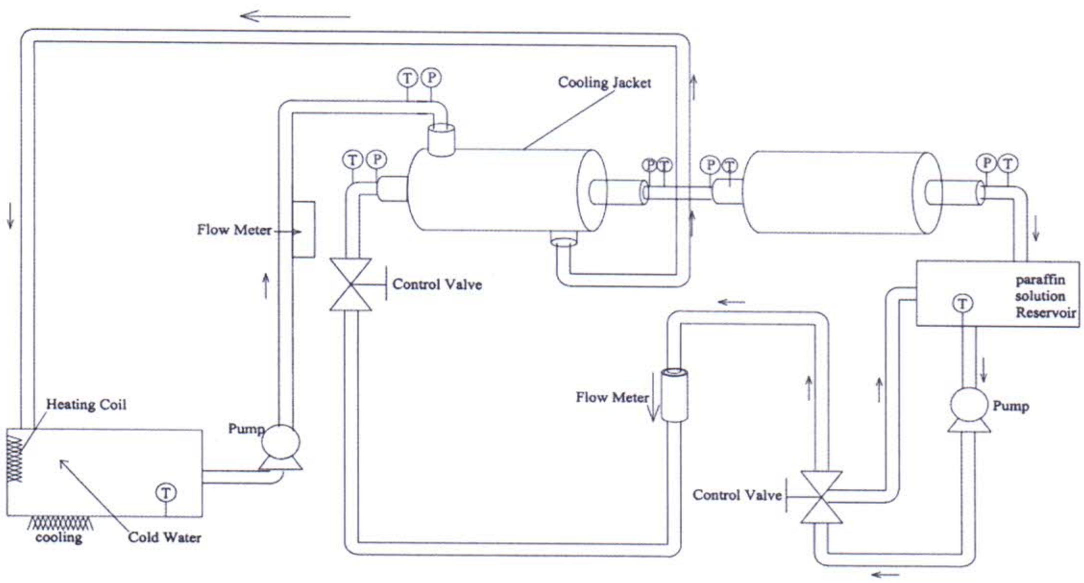

Fig. 1: Schematic diagram of wax deposition test flow loop

Demulsifier Screening Process

The demulsifiers were screened by comparing their effect in inhibiting wax deposits. Fig.2 showed the observed result when equal percentage of each of these demulsifier was mixed with the oil/wax inhibitors mixture in the flow loop Rig.

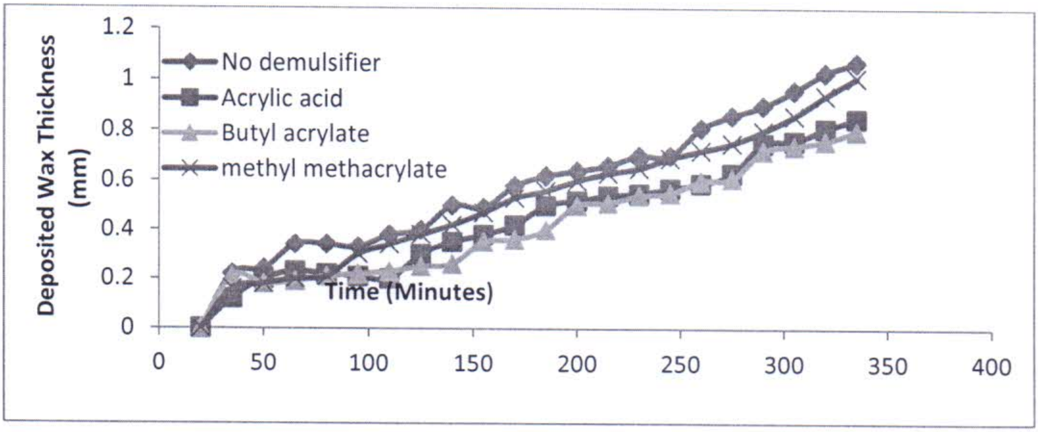

Fig.2: Effect of different demulsifiers on wax deposit.

Optimization of wax deposits inhibition process

The optimal conditions for wax deposit inhibition process during the flow of single phase fluid in sub-cooled pipeline were determined through the use of response surface methodology central composite design using four identified wax deposit inhibition variables: Bulk oil temperature, coolant temperature, flow rate and the fraction of wax inhibitor in the oil.

A total of thirty-one (31) experiments were conducted on each of the seven (7) crude oils, with Twenty-five (25) experiments organized in a factorial design (including sixteen (16) factorial points, eight (8) axial points and 1 centre point) the remaining five (5) experiments involving the repetition of the central point to obtain the good estimate of the experimental error. The response selected for analysis was the volume of deposited wax in cubic-centimeters.

The levels of the variable factors in the experiment are shown in Table 2.

Table 2: Levels of factors selected for the central composite design for wax deposition inhibition						
Factors		Levels				
		-2	-1	0	1	2
Cooling water temperature (K), A	X_1	277	282	287	292	297
Bulk oil temperature (K), B	X_2	321	323	325	327	329
Wax inhibitor percentage (%), C	X_3	1	2	3	4	5
Flow rate (Litres/min), D	X_4	0.3	0.4	0.5	0.6	0.7

The close range of the overall wax inhibition values when the experiments were repeated validate the effectiveness of the proposed wax inhibition evaluation method (Overall wax inhibition). The wax deposit inhibition process was optimized for the crude sample using the response surface methodology central composite design (RSMCCD). The study was of two stages: when the flow rates are in the laminar flow regime (Reynolds number less than 2000 (Re < 2000) and when the flow rates are in the laminar-turbulent transition flow regime (2000 < Re < 3000).

The RSMCCD software was first used to determine the minimum number of variable combinations required to evaluate the optimum condition for wax deposit inhibition for each of the oil. Then, experiments were performed with variable combinations. The volume of wax deposit from the experimental studies were then entered into the software so as to develop empirical models that correlates the volume of wax deposit to the identified variables through first, second term and interaction term.

Wax deposits inhibition optimization at the laminar flow regime (Re < 2000)

Constraining the four wax deposition process variables within the following values: Oil bulk temperature (321 - 329 K), coolant/pipeline temperature (277 - 298 K), oil flow rate (0.2 - 0.7 liter/min (Re < 2000)) and the percentage of wax inhibitors added to the oil (1 - 5%). The effects of the identified variables and their interactions on wax deposition were studied.

6.2: Wax deposits inhibition optimization at the laminar-turbulent transition flow regime (2000 < Re < 3000)

The variables were restrained within the following values: Oil bulk temperature (321 - 329 K), coolant/pipeline temperature (277 - 298 K), oil flow rate (1.1 - 1.5 liter/min) (2000 < Re < 3000)) and the percentage of wax inhibitors added to the oil (1 - 5%). The experiment were conducted as designed usinge Minitab 16 software experimental designs, with sixteen (16) factorial points, eight (8) axial points and 1 centre point) the remaining five (5) experiments involving the replication of the central point to obtain the good estimate of the experimental error. The response selected for analysis was the volume of deposited wax in cubic-centimeters.

Statistical analysis

Each response of the wax deposit inhibition process was used to develop an empirical model that correlates the volume of wax deposit to the wax deposit variables studied through first, second order and interaction terms according to the following second order polynomial equation.

$$V = \alpha_0 + \alpha_1 A + \alpha_2 B + \alpha_3 C + \alpha_4 D + \alpha_{11} A^2 + \alpha_{22} B^2 + \alpha_{33} C^2 + \alpha_{44} D^2 + \alpha_{12} AB + \alpha_{13} AC + \alpha_{14} AD + \alpha_{23} BC + \alpha_{24} BD + \alpha_{34} CD \quad 1$$

Where V represents the predicted response (volume of deposited wax), A is the cooling water temperature in Kelvin, B is the bulk oil temperature in Kelvin, C is the percentage of wax inhibitor, WI (group of AEP/xylene/butyl acrylate mixture) in the crude oil sample, and D the oil/WI flow rate: α_{12}, α_{13}, α_{14},, α_{23}, α_{24} and α_{34} are interaction coefficients; and α_{11}, α_{22}, α_{33} and α_{44} are quadratic coefficients. The results and the second-order polynomial were analyzed using analysis of variance (ANOVA) in Minitab 16 software. The model terms were selected or rejected based on the probability (P) value with 95% confidence level. The quality of the fit of the polynomial model equation was expressed by the coefficient of determination (R^2), adjusted R^2 and 'adequate precision'. The respective contour plots were obtained based on the effect of the level of the two factors. The 'point optimization' process was employed to optimize the level of each factor for maximum response.

ANOVA and Model fitting (Laminar flow regime (Re < 2000))

The ANOVA results of the equations for the crude oil sample in the laminar flow region (Re < 2000) when the flow rates are between 0.3 to 0.7 liters per minute based on the statistical analysis, the models was highly significant, with very low probability values ($P<0.05$). The significance meant that there were only small chances that incorrect predictions could occur because of experimental error or noise factors. Decomposing the models sum of squares into several sources, significant terms were verified based on individual P-values of 0.05. The significant variables and interactions for the oil showed that, A, B, C, AB, AC, BC, A^2, B^2,and C^2, are significant judging by their individual P-value less than 0.05, while D, AD, BD, CD and D^2, are insignificant as their P-values are more than 0.05. This confirms the assumptions in most of the available wax deposition models that the shear dispersion and removal effects which depend on flow rate are inconsequential in the laminar flow regime. The lack-of-fit test F-tests were used to test the model's (regression equations) adequacies. The lack-of-fit results were not statistically significant as the P-value of 0.106 was returned.

Results and discussion

The Wax deposits model (regression equations) as given in equ. 1 for the oil samples was optimized by settling the partial derivatives of the equations to zero with respect to the corresponding variables.

The results of the optimum values for each variable and the resulting minimum response (wax deposit volume) for the crude oil sample are as shown in Table 3.

Table 3: Wax optimization results (laminar flow)

Coolant Temp. (K)	Bulk Oil Temp. (K)	Perc. of WI (%)	Flow Rate (Liter/min)	Opt. wax dep. vol. software (cm^3)	Wax dep. vol. (Exp.), cm^3
258	307	2.7	0.65	67	64

Experimental verification of the predicted optimum conditions gave an actual volume of wax deposit of $64cm^3$ as against wax deposit volume of 67 cm^3 from the software. The small error (4.68%) between the predicted and actual volume of wax deposit: indicated that the software models were valid and accurate in representing the actual experimental values and also in predicting the inhibition of wax deposit within the range studied.

ANOVA and Model fitting (laminar-turbulent transition flow regime (2000 < Re < 3000))

Similarly, the ANOVA results (P-values) of the models equations for the crude oil sample in the laminar-turbulent transition flow region (2000 < Re < 3000) when the flow rates are between 1.1 to 1.5 liters per minute showed the models to be highly significant, with very low probability values ($P < 0.05$). Similar decomposing the models sum of squares into several sources, and verifying the significant terms based on individual P-values of 0.05. The determination significance of the variables and interactions for the crude oil sample are shown in Table 4 .

Table 4: P-vales of the variables/interactions	
Term	
Constant	0.012
A: Coolant temp.	0.026
B: Oil temp.	0.015
C: W.I. Percent	0.014
D: Flow rate	0.012
A^2: Coolant temp.* Coolant temp.	0.025
B^2: Oil temp. * Oil temp.	0.025
C^2: W.I. Percent * W.I. Percent	0.035
D^2: Flow rate * Flow rate	0.036
AB: Coolant temp. * Oil temp.	0.021
AC: Coolant temp. * W.I. Percent	0.025
AD: Coolant temp. * Flow rate	0.538
BC: Oil temp. * W.I. percent	0.011
BD: Oil temp. * Flow rate	0.444
CD: W.I. percent * Flow rate	0.877
Lack of fit	0.131

The results from this study showed that, A, B, C, D,AB, AC, BC, A^2, B^2, and C^2 are significant judging by their individual P-value less than 0.05, while AD, BD, CD and D^2, are insignificant as their P-values are more than 0.05. Hence the flow rate term (D) that was shown to be insignificant during the laminar flow regimes, was now significant (P-value < 0.05) in the turbulent flow regime, this confirms the importance of shear dispersion and removal in the laminar-turbulent transition flow regime, when the flow rate is high (2000 < Re < 3000)

Optimization of the Wax deposits models (regression equations) for the crude oil sample was achieved by settling the partial derivatives of the equations to zero with respect to the corresponding variables.

Experimental verification of the predicted optimum conditions gave an actual volume of wax deposit of 41cm^3 as against wax deposit volume of 46 cm^3 from the software (Table 24).

Table 5: Wax optimization results (laminar-turbulent transition flow)					
Coolant Temp. (K)	Bulk Oil Temp. (K)	Perc. of WI (%)	Flow Rate (Liter/min)	Opt. wax dep. vol. software (cm^3)	Wax dep. vol. (Exp.), cm^3
262	310	3.2	1.25	46	44

The small error (4.55%) between the predicted and actual volume of wax deposit indicated that the software models were valid and accurate in representing the actual experimental values

Response surface plots (Laminar flow regime)

In the laminar flow regime (Re < 2000), the surface plots from the experimental data analyses are as shown in figs.3 – 8.

The 3D response surfaces and two dimensional contour lines were based on the individual crude oil's model with two variables kept constant at their coded zero levels, while varying the other two variables within the experimental range. The figures show the interactive effect of two of the variables: Coolant temperature, oil temperature, Wax inhibitors percentage and the flow rates on each other keeping the other two variables at their coded zero level.

Figs. 3 show the interactive effect of coolant temperature and oil temperature on each other keeping the other two variables; Wax inhibitor percentage and flow rate at their zero level of 3% and 0.5 liter respectively the results show that the volume of deposited wax decreases to the 96 cm^3 as the coolant and oil temperature increases then begins to increase again to a wax volume of 312 cm^3. This is due to higher temperature difference between the two temperatures leading to more pronounce effect of molecular diffusion of wax particles toward the pipe's wall.

Also though the effect of coolant temperature on wax deposition was insignificant at low bulk oil temperature the effect wax more prominent as the bulk oil temperature increases, this is due to the higher rate of molecular diffusion between the bulk oil and the pipe wall at higher radial temperature gradient leading to more wax deposit on the pipe wall.

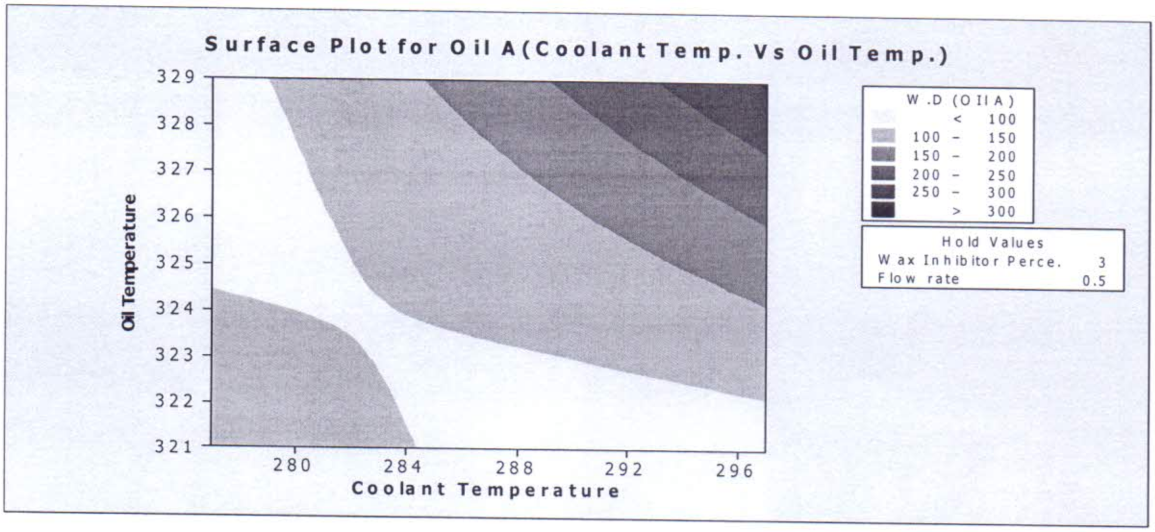

Fig. 3: Contour graph for Crude oil sample (Coolant Temp. Vs. Bulk Oil Temp.)

Figs 4 shows the interactive effect of coolant temperature and wax inhibitor percentage on each other keeping the other two variables; oil temperature and flow rate at their zero level of 325K and 0.5 liters respectively the results show that the volume of deposited wax decreases to the 87 cm^3 as the coolant and wax inhibitor percentage increases then begins to increase again up to a wax volume of 264 cm^3 as the increase in the two variable continues. This shows that there exist optimum values of wax inhibitor percentage and coolant temperature that gives a minimum volume of wax deposit and increasing or decreasing the values of these variables beyond their optimum value will result in increase in wax deposit volume.

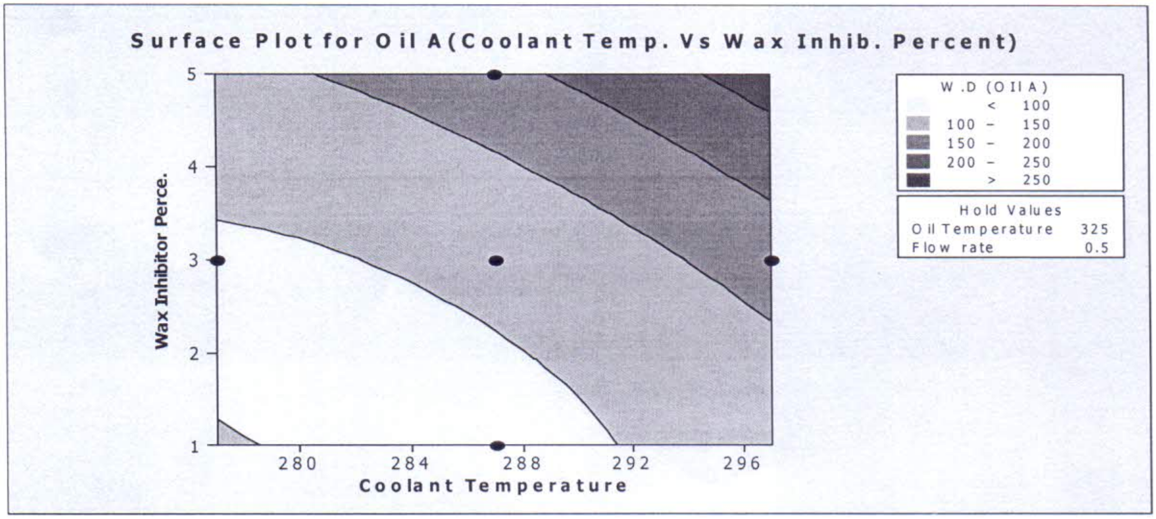

Fig. 4: Contour graph for Crude oil sample (Coolant Temp. Vs. Wax Inh. %)

Also, figs 5 shows the interactive effect of coolant temperature and flow rate on each other keeping the other two variables; Wax inhibitor percentage and oil temperature at their zero level of 3% and 325K respectively, though the effect of flow rate on wax deposit is insignificant (P-value > 0.05) the results show that the volume of deposited wax increases to the 122 cm^3 as the flow rate increase from 0.3 to 0.7 liter/min at different coolant temperatures. Indicating that the effect of shear dispersion (when increase in flow rate allow more wax particles to be sheared toward the pipe due to radial velocity gradient created in the fluid flow) is not negligible in the laminar flow region.

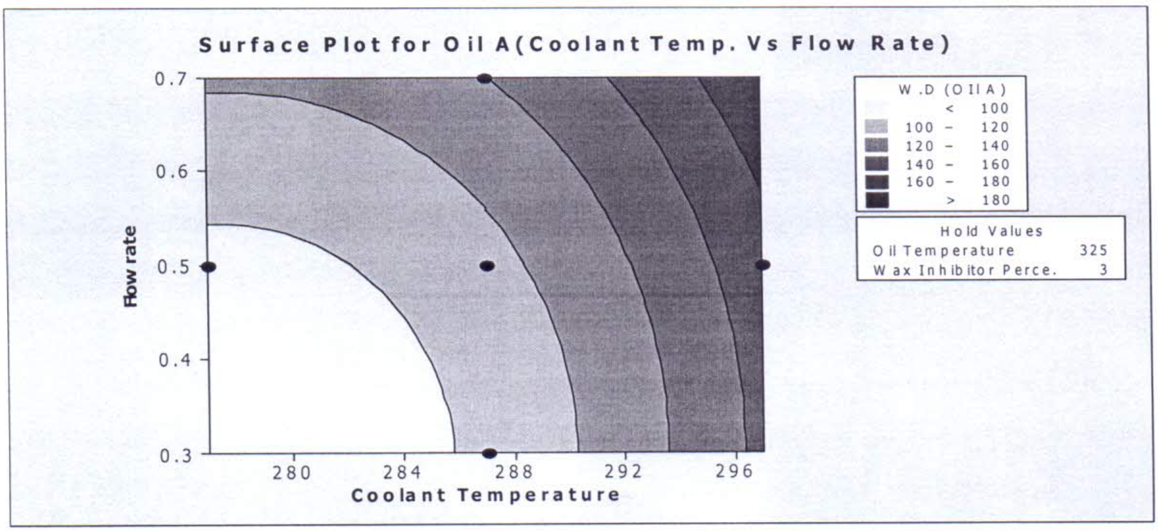

Fig. 5: Contour graph for Crude oil sample (Coolant Temp. Vs. Flow rate)

Figs 6 shows the interactive effect of Wax inhibitor percentage and oil temperature on each other keeping the other two variables; coolant and flow rate at their zero level of 287K and 0.5

liters respectively the results show that the volume of deposited wax decreases to the 87 cm^3 as the Wax inhibitor percentage and oil temperature increases then begins to increase again up to a wax volume of 268 cm^3. This is due to higher temperature difference between the two temperatures leading to more pronounce effect of shear diffusion of wax particles toward the pipe's wall. This effect overwhelmed the wax deposit reducing capability of the wax inhibitors. Simultaneous variation of the wax inhibition percentage between 1 – 5% and the bulk oil temperature shows that the wax deposit reduces from 137cm^3 to about 87cm^3 before increases to about 273cm^3, confirming that the optimum percentage of wax inhibitor percentage occur for minimum wax deposition.

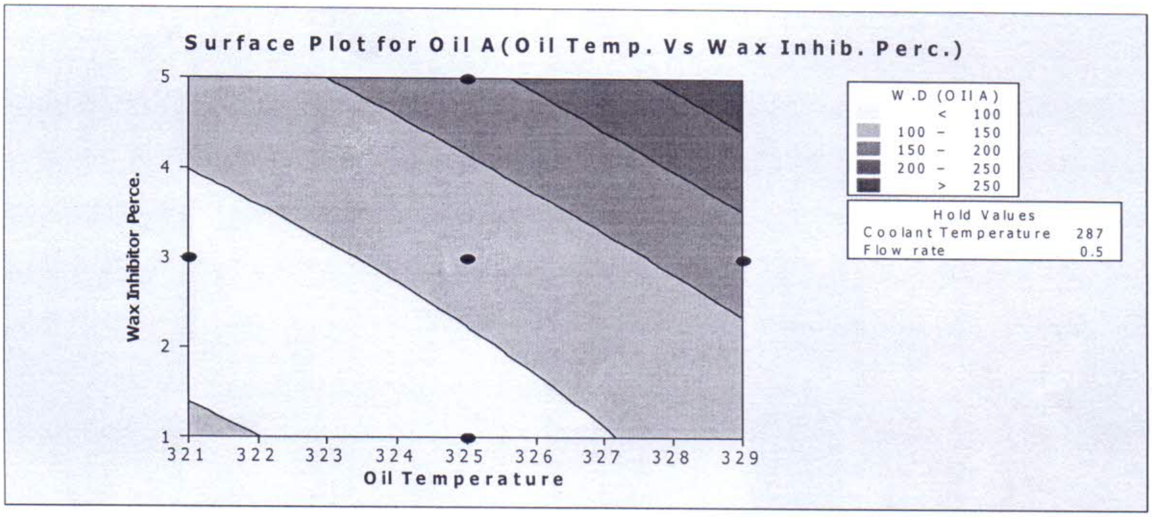

Fig 6: Contour graph for Crude oil sample (Bulk Oil Temp. Vs. Wax Inh. %)

Figs 7 shows the interactive effect of flow rate and oil temperature on each other keeping the other two variables; coolant temperature and Wax inhibitor percentage at their zero level of 287K and 3% respectively, though insignificant (P-value > 0.05) the results show that the volume of deposited wax increases from 72 cm^3 as the flow rate and oil temperature increases up to a wax volume of 296 cm^3. This is due to dual effect of higher temperature difference between the coolant and oil temperatures and the shear dispersion effect leading to more pronounce effect of shear diffusion/dispersion of wax particles toward the pipe's wall. The effect of flow rate on wax deposition was also confirmed to be insignificant in the laminar flow regime (0.3 – 0.7 liter/min)

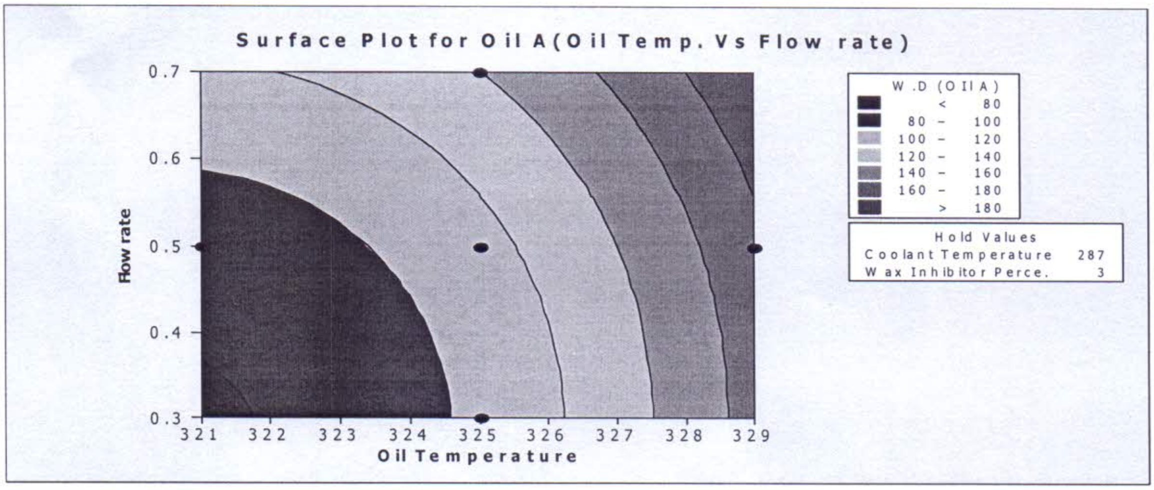

Fig. 7: Contour graph for Crude oil sample (Bulk Oil Temp. Vs. Flow Rate)

Figs 8 shows, though insignificant (P-value >0.05) the interactive effect of Wax inhibitor percentage and flow rate on each other keeping the other two variables; coolant and oil temperature at their zero level of 287K and 325K respectively the results show that the volume of deposited wax increases from 95 cm^3 as the flow rate and Wax inhibitor percentage increases up to a wax volume of 296 cm^3. This is due to overwhelming effect of shear dispersion at this temperature range. Also the effect of flow rate was observed to be insignificant in this laminar flow regime.

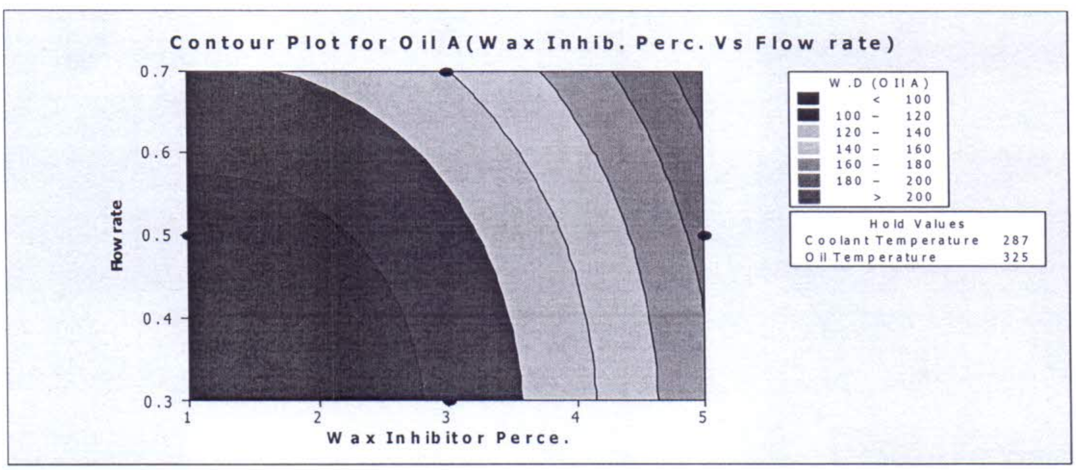

Fig 8: Contour graph for Crude oil sample (Wax Inh. % Vs. Flow Rate)

In these experiments in laminar flow regime (Re < 2000) the interactions between the variables for other oil samples (Oil sample B to G) were observed to be similar to that of oil A. While the interaction AB, AC and BC were observed to be significant with P-value of less than 0.05, the interaction between the flow rate and each of the other factors AD, BD, and CD were insignificant as their P-value were above 0.05.

Response surface plots (laminar-turbulent transition flow regime)

In the pseudo turbulent flow regime (2000 < Re < 3000) the interactions between the bulk oil temperature and the coolant temperature, coolant temperature and percentage of wax inhibitors in the crude oil, and the crude oil bulk temperature and percentage of wax inhibitor in the crude oil for crude oil sample A were similar to what were observed in the laminar flow regime, except the flow rate term, D that is significant in addition to the A, B, C, AB, AC, BC, A^2, B^2 and C^2 terms as their P-value are lower than 0.05 ($P < 0.05$), while the AD, BD, and CD terms are insignificant judging by their P-value of above 0.05 ($P > 0.05$).

The surface and contour plots for the above interactions for crude oil sample-A are as shown in figs. 9 – 14:

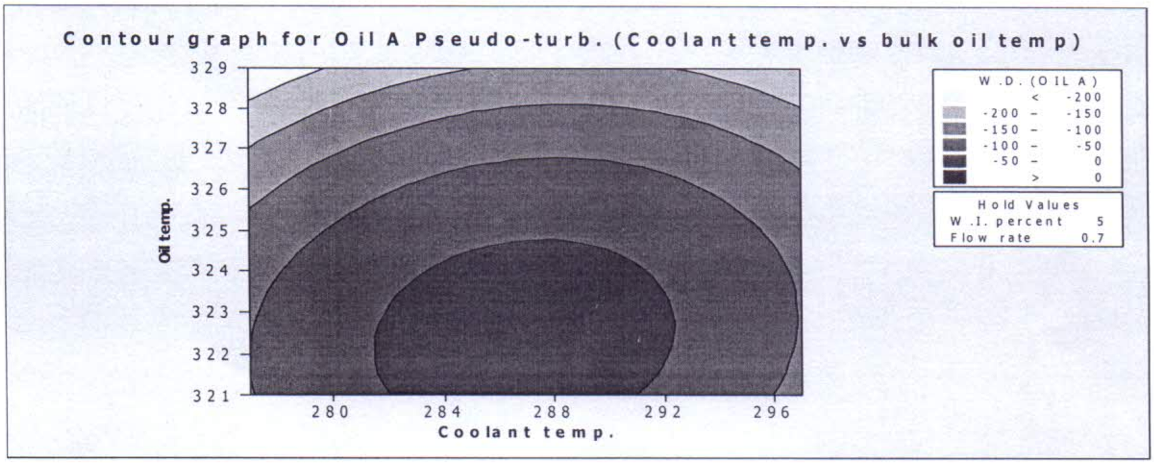

Fig. 9: Contour graph for Oil sample, Pseudo-Turbulent Flow (Coolant temp. vs Bulk Oil temp.)

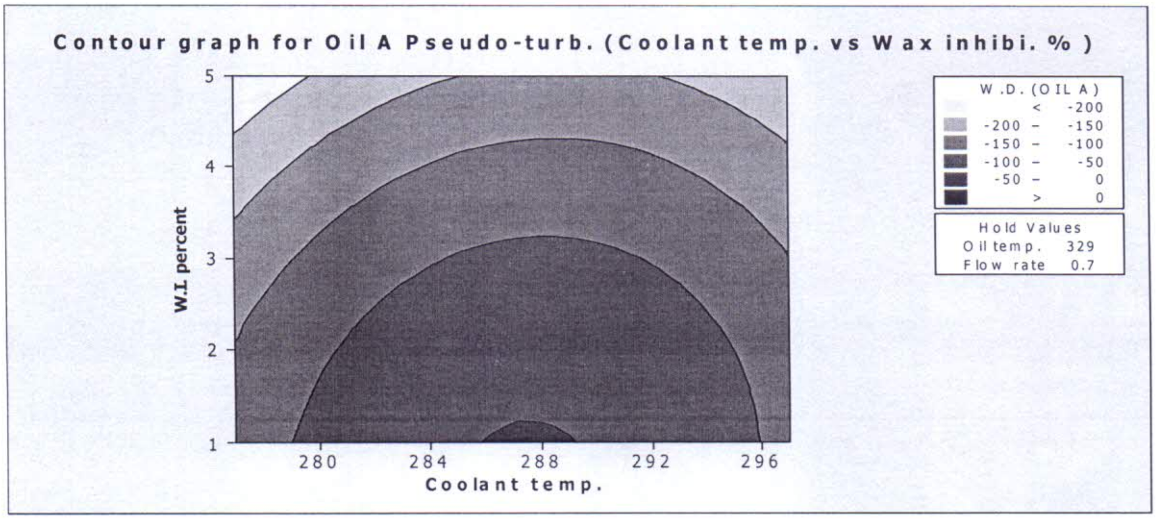

Fig. 10: Contour graph for Oil sample Pseudo-Turbulent Flow (Coolant temp. vs Wax. Inh. %)

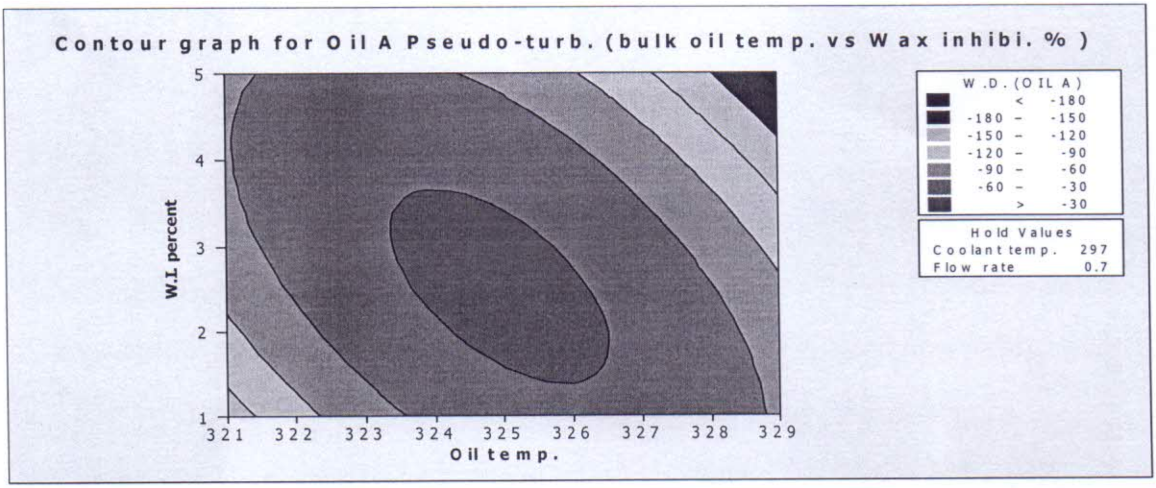

Fig. 11: Contour graph for Oil sample, Pseudo-Turbulent Flow (Bulk Oil temp. vs Wax Inh. %)

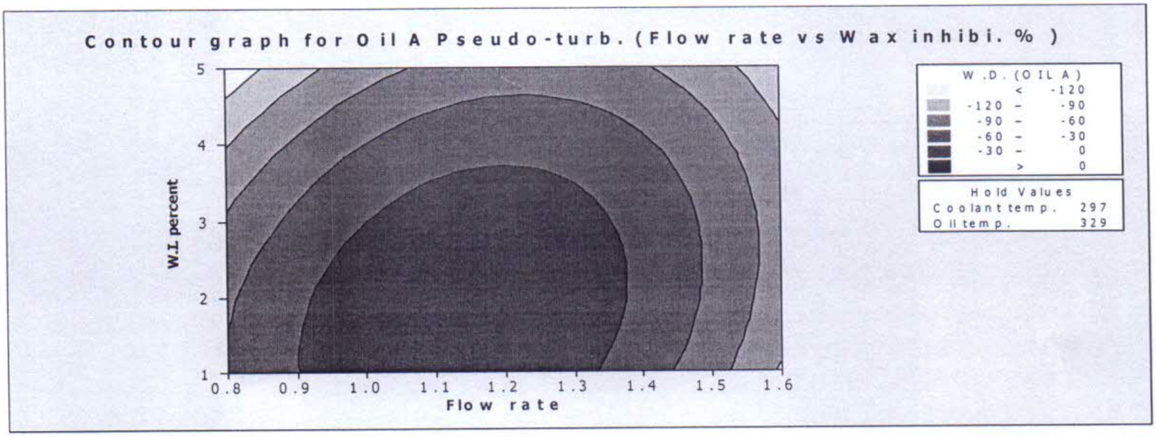

Fig. 12: Contour graph for Oil sample, Pseudo-Turbulent Flow (Flow Rate vs Wax Inh. %)

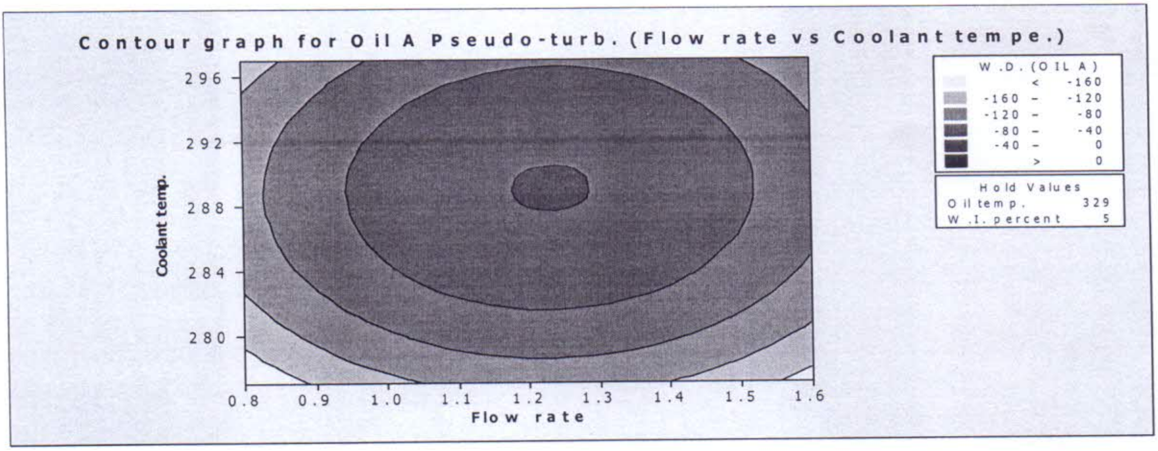

Fig. 13: Contour graph for Oil sample, Pseudo-Turbulent Flow (Flow rate Vs Coolant temp.)

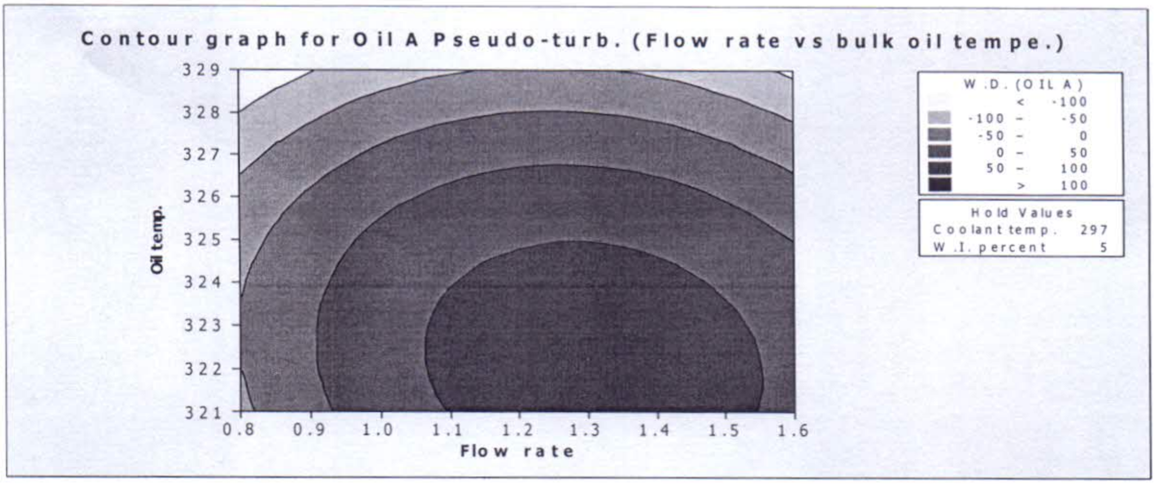

Fig. 14: Contour graph for Oil A, Pseudo-Turbulent Flow (Flow Rate Vs Bulk Oil temp.)

Conclusion

(a) In applying the response surface methodology central composite design (RSMCCD) in the Minitab-16 software to minimized wax deposit in crude oil flow in sub-cooled pipeline wax deposits were reduced to $43cm^3$ in the laminar flow regime and $64cm^3$ in the laminar-turbulent transition flow region.

(b) The shear removal effect in wax deposition observed to be relevant in the turbulent flow regime was shown to have no effect in the laminar flow region as the flow rate term (D), its square D^2 and interactions with other variables, AD, BD and CD were insignificants due to their returned P-value above 0.05.

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