

## Performances of Polyvinyl-Alcohol Inhibitor for Gas Hydrate Formation along Flow Lines

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**ABSTRACT:** The formation of hydrate in gas transporting pipelines is an epidemic as they may form plugs that block flow lines, these hydrate plugs become a plaque to pipelines themselves and to downstream equipment such as valves, blow out preventer etc. due to the high cost of hydrate inhibition using thermodynamics inhibitors, which require large quantities of alcohols or its electrolytes ( up to 40wt%) to thermodynamically disfavor the formation of hydrates, increasing interest is going towards kinetic inhibitors which requires low dosage hydrate inhibitors to delay the onset of nucleation. This study investigated the performances and effectiveness of Polyvinyl-Alcohol as a kinetic hydrate inhibitor at constant volume using Compressed Natural Gas (CNG) and water in a laboratory flow loop at maximum pressure of 10.38bar for 2hours. 0.01wt%, 0.02wt%, 0.03wt%, 0.04wt% and 0.05wt% of Polyvinyl-Alcohol were used respectively. From the analysis, gas hydrate formation was observed at temperature below 15.3C when the process was uninhibited (controlled). When inhibitors were used gas hydrate formation were not seen even at very low temperature, though depending on the concentration of Polyvinyl-Alcohol used. The various temperature at which gas hydrate were formed are 9.7, 8.9, 5.3, 0.3 and 0.1C respectively. The comparative studies shows that the higher the concentration of PVA used the better its effectiveness and performances. With 0.05wt% of Polyvinyl-Alcohol, optimum removal of hydrate crystals was observed. For monitoring and prediction of the requisites temperature to prevent hydrate formation along gas flow lines, a quadratic model was used which proved effective in gas hydrate study as statistical evaluations showed correlation coefficients above 0.99 with insignificant deviation of less than 0.01.

**KEYWORDS:** Gas Hydrate, Flow loop, Polyvinyl Alcohol, Inhibitors, Thermodynamic, Kinetic.

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### I INTRODUCTION

Natural gas hydrate are ice-like solid formed when low molecular weight hydrocarbon (gases) contact water molecules at elevated pressure and reduced temperature (Carroll, 2003). In oil and gas production system or platform, formation and agglomeration of gas hydrate particles is an undesirable process which can restrict the flow regime and flow line and eventually plug pipelines and facilities causing significant downtime and loss in production (Davies et al., 2010 and Mohammadi et al., 2005). Deepwater pipelines or flow lines are particularly prone to hydrate formation and agglomeration due to its cold environment. Hydrate control and prevention is the most critical flow assurance issue in every oil and gas or deep-water project. Gas hydrate are non-stoichiometric crystalline compounds that occur when water molecules attached themselves together through hydrogen bonding and form cavities which can be occupied by gas or volatile liquid molecules (Gbaruko et al., 2005). Hydrate formation requires the presence of a hydrate former (gases) and sufficient amount of water and is favored by high pressure and low temperature (Corrigan et al., 1996). Gas hydrate crystal structures are composed of irregular spherical cavities of hydrogen bonded with water molecules that enclosed the guest molecules. Each water cavity generally contain at least one guest molecule trapped within it by a repulsive force in the crystal structures (Giavirini and Hester, 2011). The formation of gas hydrate involves four different stages which are; gas-liquid mass transfer, nucleation, crystal growth and agglomeration (Englezos et al., 1987, Kelland, 2013). Its formation requires extensive contact between water and one or more of the hydrate formers under suitable condition of pressure and temperature (Yousif and Sloan, 1991, Christiansen and Sloan, 1994). Sloan (1991) described hydrates as a nuisance because they block transmission lines, plug

blow out preventers, jeopardize the foundation of deep water platforms and pipelines, causing tubing and casing collapse and foul heat exchangers, valves and expander. The prevention of gas hydrate formation in oil and gas industry involve the use of chemical inhibitors classified as: Firstly, thermodynamic hydrate inhibitors such as methanol, glycol etc. They are the high dosage hydrate inhibitor which works by altering the thermodynamics of hydrate formation by shifting the hydrate equilibrium to high pressure and low temperature region. Secondly, kinetic hydrate inhibitor are identified as certain water-soluble polymers with both hydrophilic and hydrophobic functional groups which include natural-KHI's found in Antifreeze proteins (AFP's) and synthetic KHI's such as polyvinylpyrrolidone (PVP), polyvinylcaprolactam (PVCap), terpolymer of vinylpyrrolidone (VP), vinyl caprolactam (VCap) etc. This is also known as low dosage hydrate inhibitor (LDHI) which delay the onset of nucleation and growth rate of crystals. And lastly, Anti-Agglomerant (AA) are identified as surfactant with hydrophilic-hydrophobic properties also known as LDHI. They allow the formation of hydrate as small particles, well dispersed and non-adherent and prevent their agglomeration and makes them flow as non-sticky and transportable slurry. Recently, in an experimental study of hydrate dissociation conditions, Richard and Adidharma (2013) investigated the performance of 1-ethyl-3-methylimidazolium chloride (EMIM-CL), novel ionic liquid inhibitors for gas hydrate at low and high ionic liquid concentration in a pressure range of 10-20MPa. Experiment on methane hydrate dissociation condition in the presence of mixed ionic liquid and conventional inhibitors, such as sodium chloride (NaCl) and Monoethylene glycol (MEG), as well as a mixture containing two ionic liquids, EMIM-Cl and EMIM-Br, are also performed to investigate any possible synergistic effects. It was also observed that single component solution of EMIM-Cl demonstrates a progressive increase in inhibition effect at low pressures, it does at higher pressures. Studies in hydrate prevention along gas flow lines have been extensively carried out. The use of various hydrate inhibitors ranging from thermodynamic, kinetic and anti-agglomerant inhibitors have been investigated. However, polyvinyl-alcohol as gas hydrate inhibitor at different concentration has not been extensively investigated which has great potential and performances for gas hydrate prevention in flow lines. Again, while several empirical models such as linear, logarithmic, exponential and multiple linear models have been applied for the study of gas hydrate prevention, quadratic model has not been used. Therefore this study exploited the potential of PVA at low concentrations as gas hydrate prevention inhibitor. Also quadratic model was used to study the gas hydrate prevention and formation temperature. This project work was tailored on the investigation and evaluation of the performances of polyvinyl-alcohol as a kinetic hydrate inhibitors (LDHI) at different concentrations. And also, to ease the monitoring and prevention of hydrate formation along flow line, experimental data was fitted into a quadratic model which proved effective for the prediction of temperature necessary for hydrate prevention and control in gas flow lines.

## II MATERIALS/METHODS

### 2.1 MATERIALS

The materials used for this experimental study include; hydrate inhibitors (polyvinyl-alcohol), compressed natural gas (CNG), water, ice-block for cooling, refrigerator, flow loop pipes, mettler top loading balance, spatula, measuring cylinder and beaker. The polyvinyl-alcohol were purchased from Chris Solong Scientific Company 14 Obi by 20 Ojoto Mile 2 Diobu, Port Harcourt, Rivers State Nigeria, with a purity of at least 97%.

### 2.2 METHOD/PROCEDURES

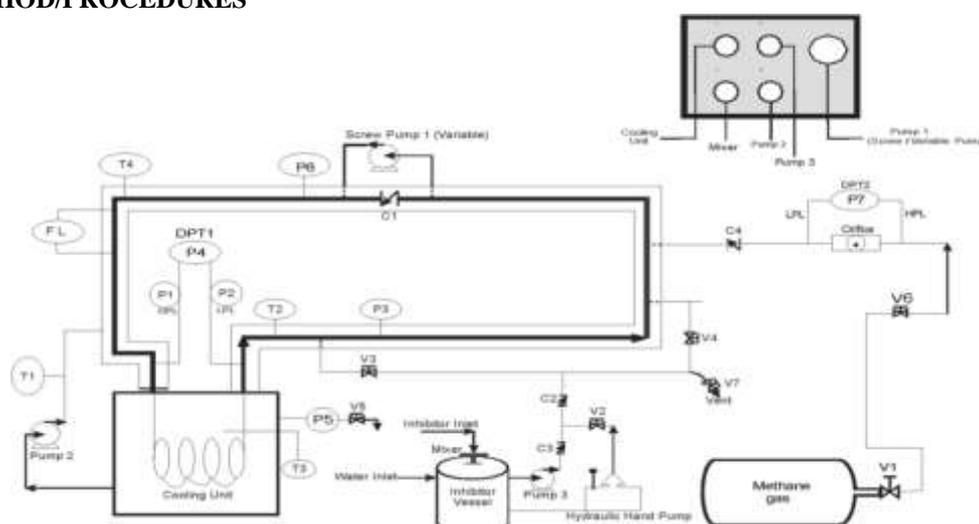


Fig 2.1: Process Flow Diagram of Laboratory Flow Loop

For this study, a mini flow loop apparatus was used. The experiment was carried out in the Advanced Petroleum Research Group Laboratory in the Department of Petroleum Engineering, University of Port Harcourt, Rivers State, Nigeria. The laboratory flow loop in this study is a closed loop of 12m fabricated using 316 stainless steel pipe of 0.5inch internal diameter, enclosed in an insulated 4inch polyvinylchloride (PVC) pipe. The experimental investigation of gas hydrate prevention using inhibitors was performed at same operating condition of the flow loop. After the preliminary preparation, 2609.45g (100%) of water was measured and turned into the inhibitor vessel. Valve 5 and pump 3 were then turned on to build up pressure up to 25Psi and thereafter, valve 5 and pump 3 were turn off. The compressed natural gas (CNG) was injected or pumped into the system by turning on valve 1, valve 3 and the orifice. The valves were turned off after attaining to the maximum operating loop pressure. Pump 2 was turned on to filled the jacket with the cooling water from the refrigerator loaded with ice and kept running until the temperature at which hydrate was formed. At every time interval of 10 minutes for a period of 2 hours, the inlet and outlet (flow loop) pressure, inlet and outlet (flow loop) temperature, cooling water (sub cooled) temperature and sample point (hydrate forming) pressure were recorded. The experiment was repeated by measuring and injecting the concentration of polyvinyl-alcohol (PVA) investigated at 0.26g, 0.52g, 0.78g, 1.05g, 1.31g representing 0.01wt%, 0.02wt%, 0.03%, 0.04wt% and 0.05wt% respectively with 2660ml of water into the inhibitor vessel and allow to run.

### III RESULTS AND DISCUSSION

#### 3.1 Hydrate Phase Envelope without Inhibitors

The hydrate phase envelope without inhibitor was studied in the laboratory and the result depicted in Figure 3.1. In the Figure, the hydrate risk and hydrate free regions had be indicated as well as theequilibrium point (the point at which the operating pressure and temperature of the flow loop intercepts with those of the hydrate formation curve).

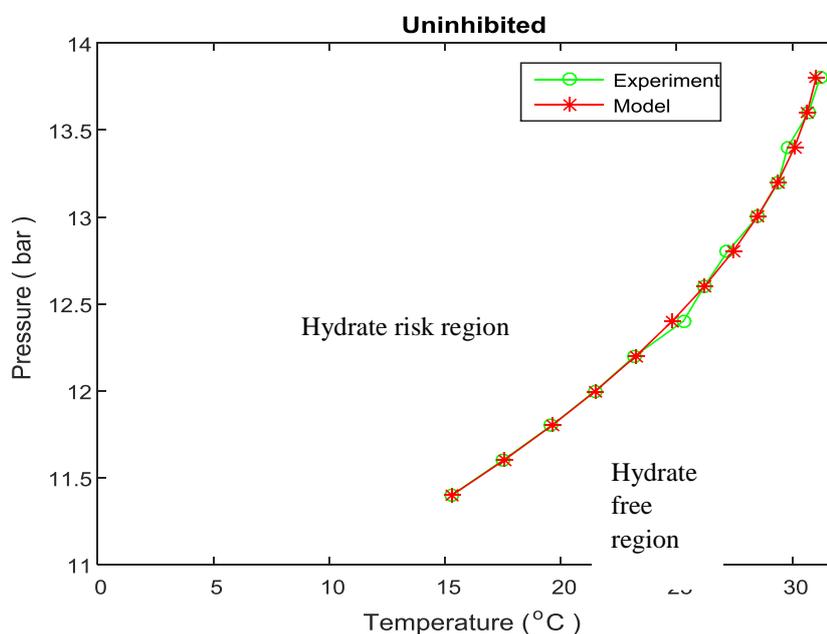


Figure 3.1: Hydrate Formation phase envelopewithout inhibitors

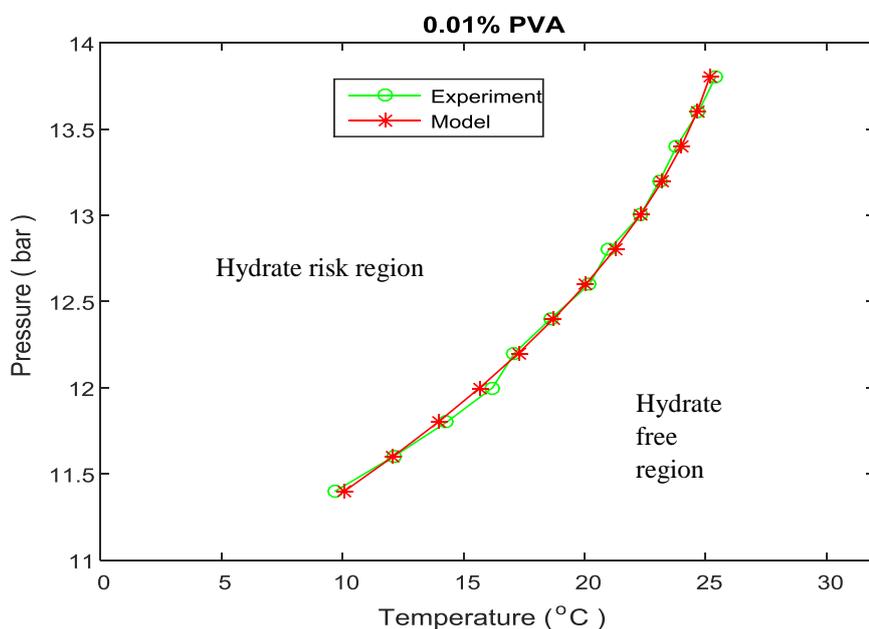
The formation of gas hydrate under high pressure and low temperature in gas pipelines was studied without inhibitor as shown in Figure 3.1. Gas flows into the flow loop at pressure of 13.3 bar and temperature of 34.1 °C and leaves at pressure of 12.1 bar and temperature of 2.8 °C after 2 hours. The hydrate formation curve decreases downwards from right to left. Also, a slight decrease in the loop pressure tremendously decreases the gas hydrate formation temperature. Thus, the influence of the sub cooling temperature of the flow line causes a decrease in the operating pressure and gas hydrate formation temperature from 13.8bar and 31.2 °C to 11.4bar and 15.3 °C. On Figure 4.1, the hydrate risk region, which is by the left of the hydrate formation curve and the hydrate free region, which is by the right of the curve have been indicated. However, when the flow loop is operated at temperatures below 15.3 °C, the system will be exposed to the risk of hydrate especially, without inhibitor.

### 3.2 Performance of Kinetic Hydrate Inhibitor

Similarly Table 3.1 shows the recorded loop pressures (LP) and the loop temperatures at which gas hydrate were formed under the influence of varying concentrations of kinetic hydrate inhibitors (KHI). At the end of the experimental analysis data were recorded at each run for the varying concentrations of polyvinyl alcohol (PVA). The effluent volume were collected and measured as 1050ml, 1100ml, 1180ml, 1280ml and 1300ml for 0.01% PVA, 0.02% PVA, 0.03% PVA, 0.04% PVA and 0.05% PVA respectively. Though the effect of volume of hydrate was not studied but the volume of effluent recorded at the end of every experimental run increases as the concentration of Polyvinyl-alcohol increases. This phenomenon shows that the higher the volume of effluent recorded the better the performances and effectiveness of inhibitor used.

**Table 3.1: Influence of Kinetic Hydrate Inhibitor (KHI) on Hydrate Formation**

Time (min)	OT (°C)	Loop Temperature of PVA (°C)					Sub cooling (°C)	OP (bar)	LP (bar)
		0.01%	0.02%	0.03%	0.04%	0.05%			
0.0	34.1	25.4	24.6	20.5	16.5	14.5	28.0	13.3	13.8
10.0	25.3	24.7	23.3	19.2	15.4	13.3	15.0	13.2	13.6
20.0	17.8	23.8	22.6	18.3	14.6	12.6	8.5	13.1	13.4
30.0	15.1	23.1	21.7	17.1	13.5	11.5	3.0	13.0	13.2
40.0	12.4	22.3	20.4	16.5	12.4	10.4	2.5	12.9	13.0
50.0	11.2	21.0	19.2	15.6	11.3	9.2	0.5	12.8	12.8
60.0	10.5	20.2	18.5	14.8	10.8	8.3	0.0	12.7	12.6
70.0	8.6	18.6	17.8	13.7	9.7	7.1	0.5	12.6	12.4
80.0	7.9	17.1	16.2	12.3	8.3	6.2	-1.0	12.5	12.2
90.0	6.3	16.2	15.3	11.5	7.5	5.1	-1.5	12.4	12.0
100.0	5.2	14.3	13.4	9.6	4.8	3.4	-2.0	12.3	11.8
110.0	3.9	12.1	11.3	7.5	2.4	1.3	-3.0	12.2	11.6
120.0	2.8	9.7	8.9	5.3	0.3	0.1	-3.5	12.1	11.4



**Figure 3.2: Hydrate Formation Curve for Flow Line Inhibited with 0.01% PVA**

Polyvinyl alcohol (PVA) has proven economical and effective in gas hydrate prevention in flow lines. The use of varying concentrations of THI and KHI for the study of gas hydrate prevention enables the best viable concentration of inhibitor to be determined. Thus, Odutola *et al.*, (2016) had applied several concentrations inhibitors for gas hydrate prevention in flow lines. Figure 3.2 shows the prevention of gas hydrate along the flow loop over the period of the experiment using 0.01 wt% PVA inhibitor. At the same operating conditions of the flow loop, the hydrate formation curve decreases from right to left, and the gas hydrate formation temperature reduces from 25.4 °C to 9.7 °C, while for the model, it reduces from 25.2 °C to 10.1 °C. Again, with 0.01 wt% PVA inhibitor, the hydrate risk region was shifted to the left as against when the flow line was used without inhibitor thereby, reducing the deposition of hydrate crystals, which increases the loop pressure.

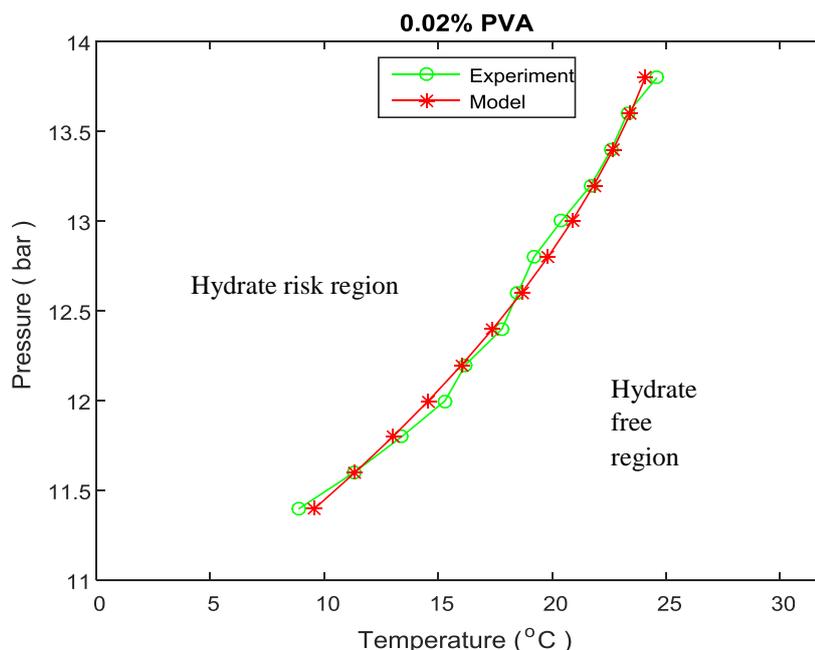


Figure 3.3: Hydrate Formation Curve for Flow Line Inhibited with 0.02% PVA

Figure 3.3 shows prevention of gas hydrate along the flow loop over the period of the experiment using 0.02 wt% PVA inhibitor. At the same operating conditions of the flow loop, the hydrate formation curve decreases from right to left, and the gas hydrate formation temperature decreases from 24.6 °C to 8.9 °C, while for the model, it decreases from 24.1 °C to 9.6 °C. Again, with 0.02 wt% PVA inhibitor, the hydrate risk region was shifted to the left as against when the flow line was used without inhibitor thereby, reducing the deposition of hydrate crystals that increases the loop pressure.

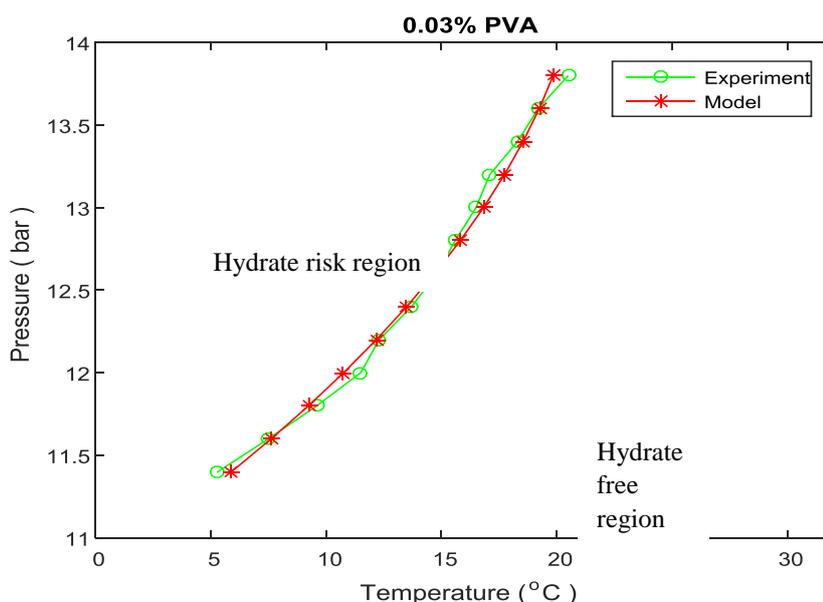


Figure 3.4: Hydrate Formation Curve for Flow Line Inhibited with 0.03% PVA

Figure 3.4 shows prevention of gas hydrate along the flow loop over the period of the experiment using 0.03 wt% PVA inhibitor. Again, at the same operating conditions of the flow loop, the hydrate formation curve decreases further from right to left, while the gas hydrate formation temperature decreases from 20.5 °C to 5.3 °C, and for the model, it decreases from 19.8 °C to 5.9 °C. Again, with 0.03 wt% PVA inhibitor, the hydrate risk region was shifted to the left as against when the flow line was used without inhibitor thereby, reducing the deposition of hydrate crystals that increases the loop pressure.

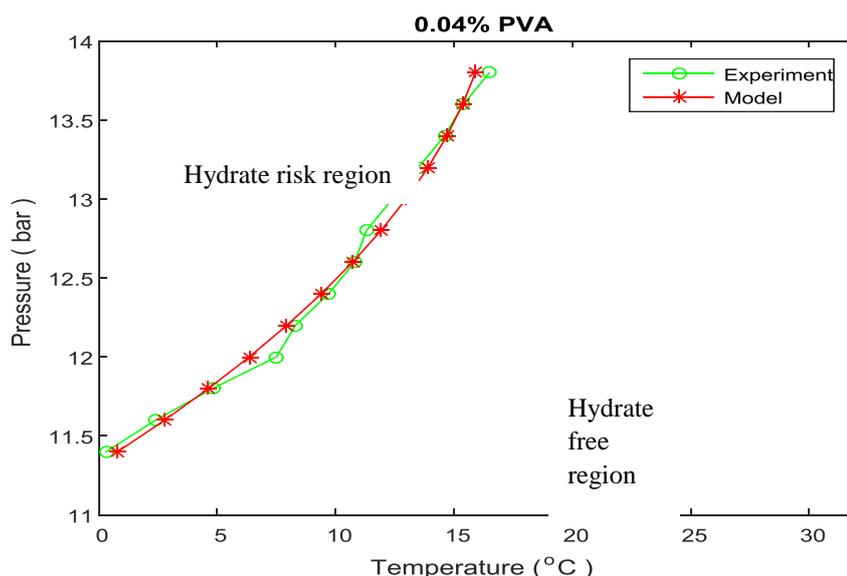


Figure 3.5: Hydrate Formation Curve for Flow Line Inhibited with 0.04% PVA

The prevention of gas hydrate along the flow loop over the period of the experiment using 0.04 wt% PVA inhibitor is shown in Figure 3.5. At the same operating conditions of the flow loop, the hydrate formation curve decreases further from right to left, while the gas hydrate formation temperature decreases from 16.5 °C to 0.3 °C, and for the model, it decreases from 15.9 °C to 0.8 °C. However, with 0.04 wt% PVA inhibitor, the hydrate risk region was shifted to the left as against when the flow line was used without inhibitor thereby, reducing the deposition of hydrate crystals that increases the loop pressure.

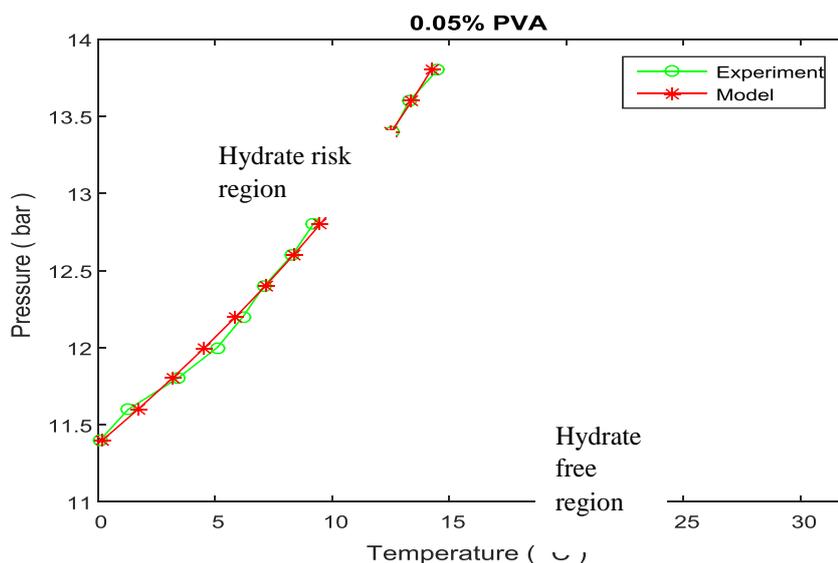


Figure 3.6: Hydrate Formation Curve for Flow Line Inhibited with 0.05% PVA

Finally, the prevention of gas hydrate along the flow loop over the period of the experiment using 0.05wt% PVA inhibitor is shown in Figure 3.6. Again, at the same operating conditions of the flow loop, the hydrate formation curve decreases from right to left, while the gas hydrate formation temperature decreases from 14.5 °C to 0.1 °C, and for the model, it decreases from 14.2 °C to 0.2 °C. Therefore, using 0.05 wt% PVA inhibitor, the hydrate risk region was shifted further to the left as against when the flow line was used without inhibitor thereby, reducing the deposition of hydrate crystals, which increases the loop pressure.

### 3.3 Comparison of Various Concentrations of Kinetic Hydrate Inhibitors

The performances of the various concentrations of kinetic hydrate inhibitors were compared. The performances of the inhibitors were indicated by the shift away of the hydrate formation curves with inhibitors

from the hydrate formation curve without inhibitor. The further the shift of hydrate formation curves away from that without inhibitor, the better the performance.

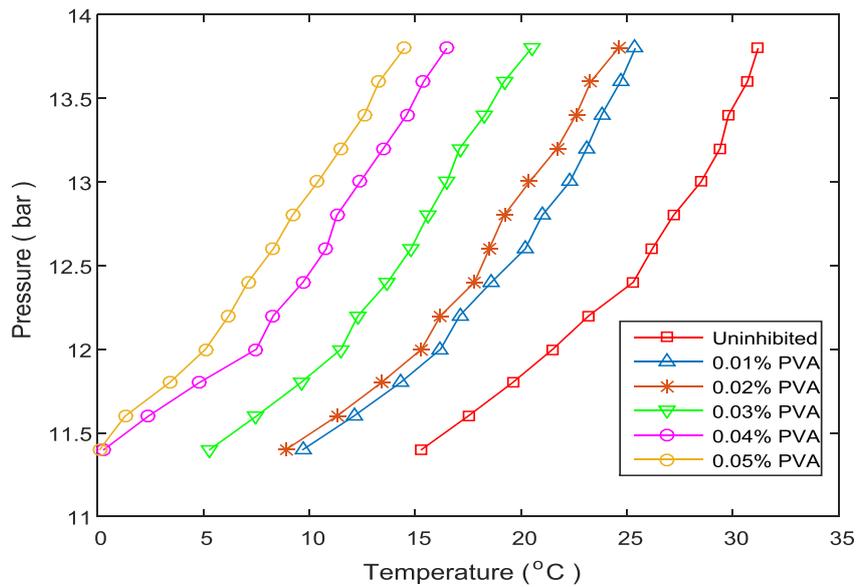


Figure 3.7: Experimental Performances of Kinetic Hydrate Inhibitors

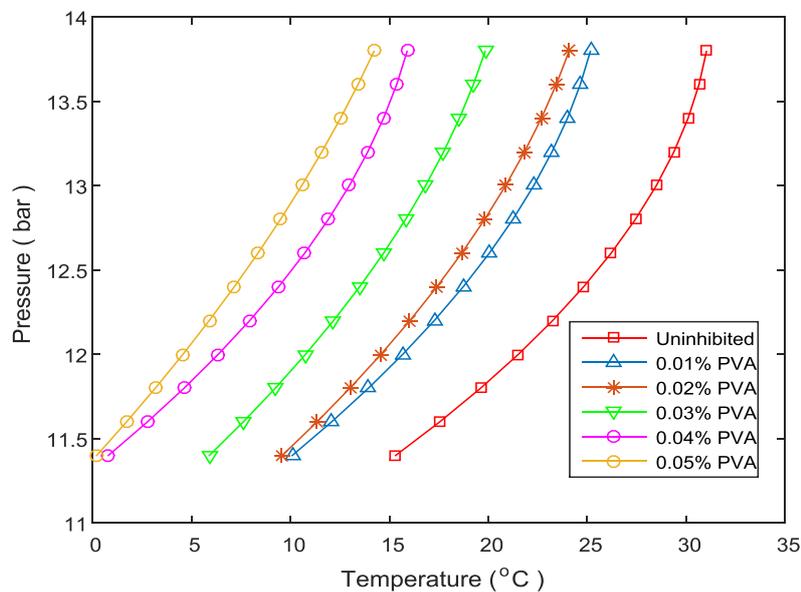


Figure 3.8: Model Performances of Kinetic Hydrate Inhibitors

Similarly, the performance of kinetic hydrate inhibitors for the prevention of gas hydrate formation in the flow line obtained from the experiment was compared as shown in Figure 3.7. Unlike thermodynamic hydrate inhibitor, different concentrations: 0.01, 0.02, 0.03, 0.04 and 0.05 %wt of polyvinyl alcohol were investigated at the same operating conditions of the flow loop. The results showed that when the concentration of PVA inhibitor is increased, gas hydrates were not observed at even very low temperature in the flow loop, which was indicated in Figure 3.7 by the shifting of the curves away from the curve uninhibited. Thus, 0.05% by weight of PVA prevented the formation of hydrate more than the rest of lower concentrations in the flow line. This therefore, agrees with the works of Kakavand and Asachi, (2015) and Odutola *et al.*, (2016). Although, the performance of the 0.01% wt PVA was low, all the concentrations investigated showed the capacity for preventing gas hydrate formation in gas flow line, since the hydrate risk region was shifted to the left as against when the flow loop was operated without inhibitor.

Again, the performances of the different concentrations of PVA inhibitors obtained from the model were also compared as shown in Figure 3.8. Just like the results obtained from the experiment, increase in PVA

concentration increases the effectiveness of PVA inhibitor. Again, results obtained from the empirical model eliminated the fluctuations observed in the experimental results. The fluctuation in experimental results was as a result of errors due to parallel readings, which was corrected by application of the quadratic model.

### 3.4 MODEL FORMULATION

To study any process in real life, a mathematical representation of the process can be applied, which is economically viable. The mathematical model can be formulated through the knowledge of the process parameters. In the flow loop, variation of pressure alters the temperature of the flow loop and hence, resulting in the formation of hydrate especially, when the pressure is very high at low temperature. Hence, it becomes very imperative to study the temperature at which hydrate could form as the pressure of the system is changing due to the length of the flow line. Previously, the dependency of gas hydrate prevention temperature on loop pressure had been studied using logarithmic model by Iyowu (2010); linear model by Lee *et al.* (2001) and Samimi (2012); and polynomial and exponential models by Odutola *et al.* (2016). In this study, quadratic model was used to study the hydrate prevention temperature as a function of the loop pressure, represented by equation (3.1).

$$T = aP^2 + bP + C \quad (3.1)$$

where:

$T$  = Loop temperature (°C)

$P$  = Loop Pressure (bar)

$a, b$  and  $C$  = Constant coefficients

The temperature was solved by fitting the constants and the loop pressures obtained from the experiment into equation (3.1).

### 3.5 Determination of Constant Coefficients

The constant coefficients in the model was obtained by fitting experimental data into POLYMATH tool for the different PVA concentration including the hydrate phase envelope without inhibitors. To validate the model, analysis of variance (ANOVA) was used via Microsoft Excel, by comparing the experimental and model values of the temperature. The deviation between the model and experiment indicates the level of agreement between experimental and model values.

**Table 3.2: Analysis of variance for Hydrate Phase Envelope without inhibitors**

ANOVA						
Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	0.000137	1	0.000137	5.05E-06	0.998226	4.259677
Within Groups	653.3875	24	27.22448			
Total	653.3876	25				

**Table 3.3: Analysis of variance for 0.01% PVA**

ANOVA						
Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	0.00036	1	0.00036	1.5E-05	0.99698	4.25968
Within Groups	593.649	24	24.7354			
Total	593.649	25				

**Table 3.4: Analysis of variance for 0.02% PVA**

ANOVA						
Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	0.00045	1	0.00045	2.0E-05	0.99647	4.25968
Within Groups	544.471	24	22.6863			
Total	544.472	25				

**Table 3.5: Analysis of variance for 0.03% PVA**

ANOVA						
Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	1.1E-06	1	1.1E-06	5.2E-08	0.99982	4.25968
Within Groups	504.628	24	21.0262			
Total	504.628	25				

**Table 3.6: Analysis of variance for 0.04% PVA**

ANOVA						
Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	0.00062	1	0.00062	2.5E-05	0.99605	4.25968
Within Groups	597.767	24	24.907			
Total	597.768	25				

**Table 3.7: Analysis of variance for 0.05% PVA**

ANOVA						
Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	0.0005	1	0.0005	2.4E-05	0.99616	4.25968
Within Groups	502.803	24	20.9501			
Total	502.804	25				

The summary of the deviations for the respective inhibitors as well as the uninhibited flow loop, with the P-value at 95 % confidence level are presented in Table 3.8.

**Table 3.8: Deviation between Experiment and Predicted Hydrate Formation Temperature along the Flow Loop.**

Sample	P – Values	Deviation
Uninhibited	0.998226	0.0018
0.01% PVA	0.99698	0.0030
0.02% PVA	0.99647	0.0035
0.03% PVA	0.99982	0.0002
0.04% PVA	0.99605	0.0040
0.05% PVA	0.99616	0.0038

Table 3.8 shows the deviation between experiment and model results for temperature at which hydrate could form in the flow loop. The *P-values* showed that there is no significant difference between the experiment and model results for temperature at which hydrate could form in the flow loop with deviations relatively insignificant. Therefore, the model is valid and can be used to predict the temperature at which gas hydrate can be experienced or prevented along gas flow lines.

#### IV CONCLUSION

The transportation of compressed natural gas (CNG) via pipelines especially, in subsea is always saturated with water, causing the formation of hydrates and thus, subsequent impairment of the flow lines. However, methods are available to mitigate this hydrate formation along flow line of gas transport.

Inhibiting the flow line with kinetic hydrate inhibitors will prevent, or drastically reduce the formation of gas hydrate. Therefore, this study investigated the effectiveness and performances of kinetic hydrate inhibitors in preventing gas hydrate formation along gas pipelines. 0.01 wt% to 0.05 wt% of Polyvinyl Alcohol as gas hydrate inhibitors were experimentally investigated for a period of 2 hours.

The flow loop was operated at initial pressure of 13.8 bar, but when the refrigerator was switched on, the temperature of the flow line reduces, thereby attracting the formation of gas hydrate in the system.

The analysis showed that when the flow loop was operated without the use of inhibitor, crystals which is an indication of hydrate formation was observed at temperature below 15.3°C. However, when inhibitors were introduced into the flow line, the temperature at which crystals were observed reduces from 15.3°C to lower temperatures. Thus, lowest temperature (0.2 °C) at which crystals was observed occurred when the flow line was inhibited with 0.05 wt% PVA.

The observation of gas hydrate at low temperatures indicates that kinetic hydrate inhibitors are effective in preventing gas hydrate formation along the flow line. However, the kinetic hydrate inhibitors prove to be effective for gas hydrate mitigation and prevention. Also, since the flow line surrounding temperature influences the formation of hydrate, the hydrate forming temperatures of the investigated inhibitors obtained from the experiment were fitted into quadratic model. The results obtained from the model agreed with the experiment. Finally, increase in concentration of PVA increases the capacity and performances of gas hydrate prevention in the flow loop. Also, this study showed that quadratic model is effective for the prediction of temperature necessary to prevent or mitigate hydrate formation in gas flow lines with very high correlation coefficient.

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