American Journal of Chemical Engineering
2017; 5(3-1): 28-41
http://www.sciencepublishinggroup.com/j/ajche
doi: 10.11648/j.ajche.s.2017050301.14
ISSN: 2330-8605 (Print); ISSN: 2330-8613 (Online)

Design, Fabrication and Validation of a Laboratory Flow Loop for Hydrate Studies

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To cite this article:
Odutola T. O., Ajienka J. A., Onyekonwu M. O., Ikiensikimama S. S. Design, Fabrication and Validation of a Laboratory Flow Loop for Hydrate Studies. *American Journal of Chemical Engineering*. Special Issue: Oil Field Chemicals and Petrochemicals. Vol. 5, No. 3-1, 2017, pp. 28-41. doi: 10.11648/j.ajche.s.2017050301.14

Received: March 3, 2017; **Accepted**: March 4, 2017; **Published**: April 27, 2017

Abstract: The peculiar nature of the offshore environment has necessitated the need for the Oil and Gas industry to develop durable subsea technologies and better hydrate inhibitors to prevent hydrate formation and assure flow. This paper discusses the design, fabrication and validation of a laboratory flow loop for hydrate studies. The laboratory loop is a closed loop of 12meters, fabricated using 0.5inch 316 stainless steel pipe enclosed in an insulated 4inch Polyvinylchloride (PVC) pipe. The skid mounted loop was fitted with pumps, temperature gauges, pressure gauges, differential pressure transmitters, a gas mixing vessel, an inhibitor mixing vessel, and a Natural Gas cylinder. Hydrate formed in the loop when natural gas was contacted with water under suitable hydrate forming temperature and pressure conditions and was indicated by an increased loop temperature, an increased differential pressure and a decreased loop pressure. Loop Validation was done by flowing a single phase fluid of water, a single phase fluid of gas and a 2 phase fluid of gas and water in three different experimental runs respectively. Each experimental run lasted 2 hours during which temperatures and pressures around the loop were recorded every minute. Hydrate formation was observed in the experimental run conducted with the two phase fluid (gas and water) and the experimental run conducted with gas alone due to water condensing out of gas during cooling. Hydrate did not form in the experimental run conducted with single phase fluid of water. The laboratory flow loop adequately predicts hydrate formation and has been used in screening and selection of Kinetic Hydrate Inhibitors (KHI).

Keywords: Gas Hydrate Equipment, Flow Assurance, Hydrate Loop Validation

1. Introduction

Sub-sea developments generate flow assurance challenges due to the low seawater temperature and high pressure flow. These conditions of temperature and pressure can lead to hydrate formation in the presence of gas hydrate formers and water. Gas hydrates are known to be a major flow assurance problem for the oil and gas industry. This problem increased as production fields moved to harsher conditions of colder temperatures, greater sea depths and higher water cuts. Blockage of pipelines by hydrates causes flow stoppages and significant financial loss, as well as potential environmental and safety concerns [1].

Hydrate formation requires: the presence of a hydrate former, appropriate temperature and pressure conditions and sufficient amount of water. Other important factors are turbulence and nucleation sites [2]. Hydrates usually form at elevated pressure, but they can also form during gas expansion due to Joule Thomson's effect [3]. When crystals of hydrates form, they come together to form hydrate plugs that can obstruct flow [1]. Some available techniques for hydrate management include: thermal insulation [4], heat treatment [5] pressure control, phase separation [6] and chemical method [7]. Understanding hydrate formation mechanism in gas dominated systems [8], oil dominated systems [9], [10] and water dominated systems [11] is crucial for proper design of hydrate equipment.

Multiphase loops are a simple and reliable approach to approximate hydrate formation conditions. In multiphase flow loops, the usual protocol in obtaining phase equilibria data involves observing the hydrate phase by indirect means, such as an associated pressure decrease or temperature increase in the fluid phase [12]. This can be achieved by conducting a constant volume experiment or a constant

pressure experiment in the loop depending on its design. Multiphase loops can be open or closed loops. Open loops are simple to build and have relatively low cost of fabrication. They are very safe as they usually have few safety barriers. They can operate at atmospheric pressure. On the other hand, closed flow loops have more complex design and fabrication cost is quite high. They are pressurized hence there is safety concern of burst or explosion in these loops. Most hydrate research loops are closed loops. In order to establish well controlled and relevant multiphase flows in a loop, the length/diameter ratio should be L>300D [13].

Downscaling of petroleum systems to a laboratory environment requires establishing a fixed envelope of operating conditions that allows experimental work to focus on studying various phenomena related to a specific area of research [14]. Flow loops give excellent analysis of hydrate formation as they can model flowing conditions, shut in conditions and restart conditions. The limitation of flow loop equipment is the crushing of hydrate crystal by the pump hence; Experiments involving the use of Anti Agglomerants are difficult to interpret when conducted with flow loop [15]. Some hydrate experiments conducted in existing hydrate flow loops are discussed below.

Peytavy *et al* [16] discussed experiments conducted in a Hydrate loop (Figure 1) developed to properly model pressures experienced deep offshore. The equipment consists of a 1inch 316 stainless steel jacketed pipe of length 35.6m, some pumps and a 90 liters storage tank where the test fluid (water, oil or inhibitors) are stored. This hydrate facility works by circulating gas saturated liquid around the loop. Cooling/heating fluid is circulated around the loop with the aid of a jacketed pump in order to regulate the loop temperature (from 50°C to 0°C) in flowing and shut down conditions. The loop flow rates can vary from 0 to 6 m^3/h, and the liquid velocities from 0 to 3 m/s. The maximum pressure the loop can withstand is 165bara (about 2393 psia). Other components installed on the loop for maximum performance of the equipment are: a mass flow meter for measuring the density and mass flow rate of the test fluid, two differential pressure cells rated 0-20 bar for measuring the differential pressure at the pump and vertical rings of the pipe, a compressor to compress gas to 200bar, a pressure regulated gas make-up system to ensure constant temperature all through the experiment, and a real time computerized data acquisition system. Hydrate indicators used in this equipment are: rise in pressure drop between inlet and outlet of pump and an increased cumulative gas make-up rate. The cumulative gas rate can also be used to evaluate the quantity of hydrates formed.

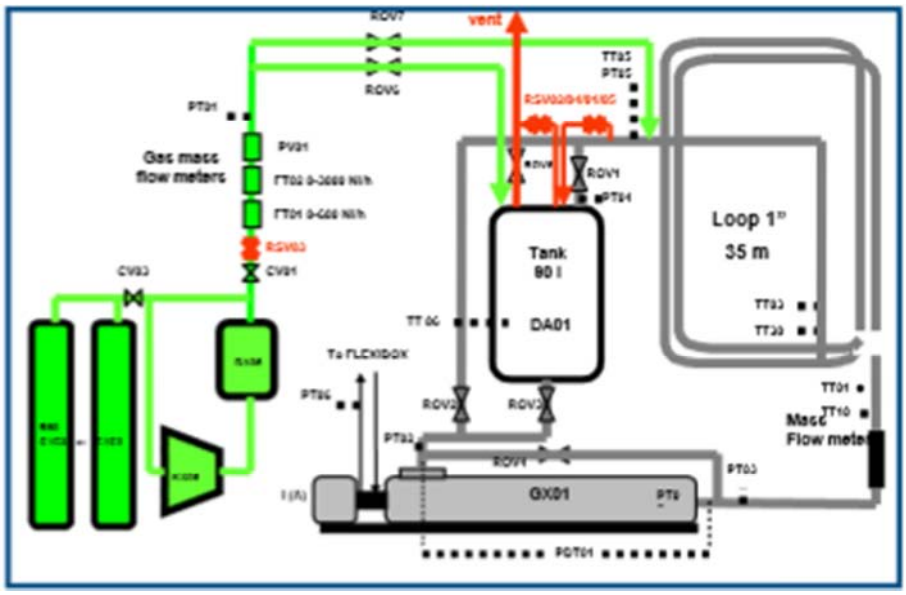

Figure 1. *PFD of the 165 bar hydrate loop [16].*

Mauricio *et. al* [17] investigated natural gas hydrate behaviour in gas pipelines during continuous flow and restart conditions using a hydrate flow loop designed by Commonwealth Scientific and Industrial Research Organisation (CSIRO). The CSIRO hydrate loop Hydrate Flow Loop, (Figure 2) which was designed to closely mimic conditions found at Australian offshore gas producing pipelines, is a 40m long 316 stainless steel flow line of 1inch internal diameter connected to liquid pumps and a gas compressor. The flow loop handles brines, non-corrosive gases, and water. To achieve temperatures between-8 to 30°C in the loop, liquid coolant is generated in a chillier unit and circulated through a 4 inch pipe-in-pipe system. Pressure in the loop can be regulated using high pressure gas cylinders or the natural gas storage system. Resistance Temperature Detector (RTD) sensors and pressure transmitters (PT-1 to PT-6) measure the pressure and temperature at different points in the flow line. Video recordings from four different high pressure sight windows (VW1 to VW4) at different locations in loop are used to inspect fluids inside the loop. A two phase separator tank is used in collecting the mixture of fluids and hydrates transported along the loop and

subsequently to separate the phases. After the separation, separated liquid is sent into the liquid storage tank while gas leaving separator is compressed before it is injected into the flow line.

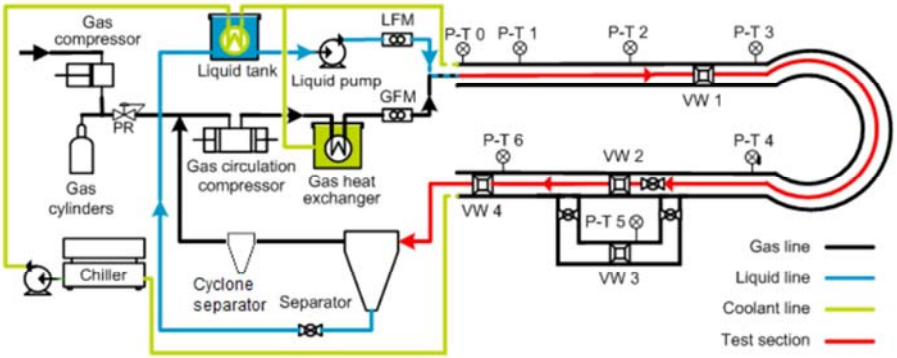

Figure 2. *CSIRO Hydrate Flow Loop Simplified Layout [17].*

Talaghat [18] examined the induction time for gas hydrate formation in presence of Kinetic Hydrate Inhibitors using the Shiraz University of Technology Flow Mini-Loop apparatus. The 12 meters long equipment was prepared using a 316 stainless steel with an inner diameter of 10.6 mm and a pressure rating 10 Mpa (Figure 3). A piston pump injects water or water and inhibitor into the loop and flow is executed by means of a Kracht screw pump with variable rates up to 0.8 m3/hr. Temperature in the loop is regulated by circulating pump fed water/ethylene glycol mixture through a PVC pipe (of 32 mm internal diameter) that covers the 10.6mm 316 stainless steel loop. The loop is monitored by a flow meter, a pressure drop transmitter, three temperature sensors, and two pressure sensors. Experiment carried out in the loop is done under constant pressure (isobaric) process. A regulator and gas make-up system ensures constant pressure all through the experiment.

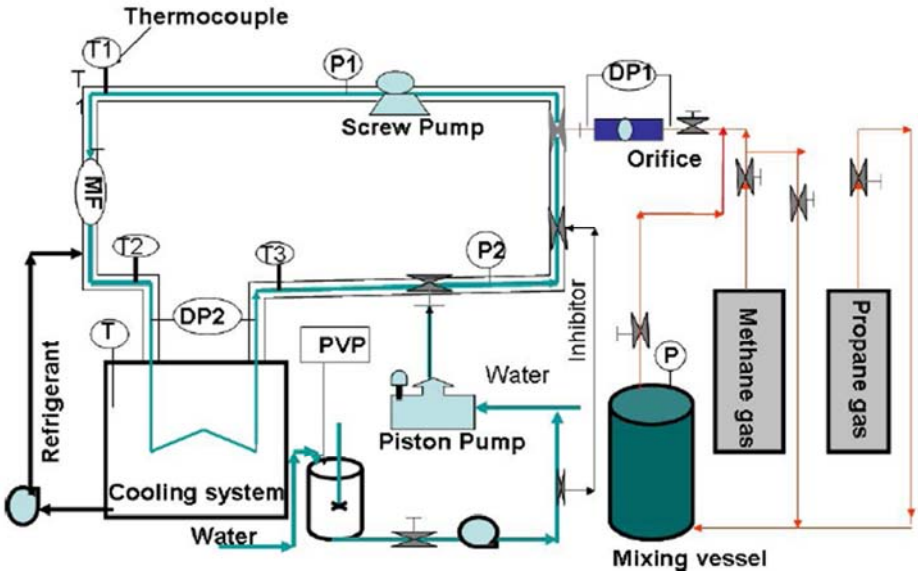

Figure 3. *Shiraz University of Technology Flow Mini-Loop Apparatus [18].*

Sakurai *et al.* [19] discussed experimental studies on a hydrate loop modeled to mimic temperature conditions of the Nankai sea bed. The 20m hydrate loop was designed by Japan Oil, Gas and Metals National Corporation (JOGMEC) and Oil Field Production Technologies (OPT). The flow loop (Figure 4) has two sections. The first section consistsof an approximately 6m optically clear acrylic section with an internal diameter of 8mm which allowed image capture in horizontal and vertical flow lines and at flow stagnation points. The second section is approximately 14m of seamless stainless steel tube, also with internal diameter of 8mm.It contains a variable speed gear pump for calculating flow, flow meters for measuring mass flow rate and density. All flow meters and guages are connected to a data categorization system that records all sensor readings every 6 seconds.Fluid temperature in the flow loop is kept constant by the water bath. Pressure of the flow loop is controlled by a Syringe which regulates water volume of the flow loop. A high speed camera captures the horizontal flow process while two conventional cameras capture the top corners in this experiment.The entrie flow loop is installed inside an insulated and refrigerated cold room.

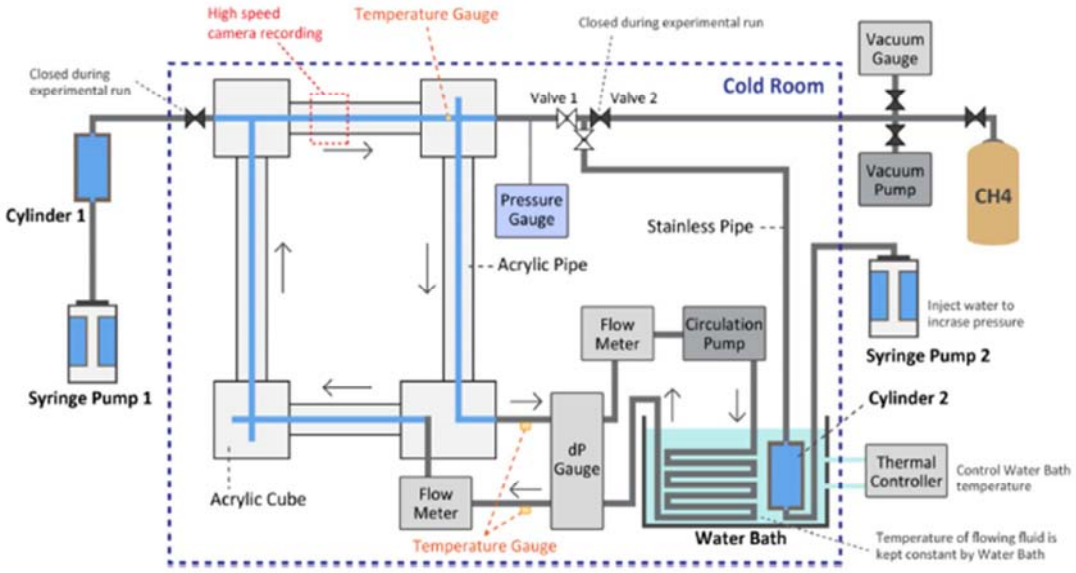

Figure 4. *Schematic of JOGMEC & OPT Flow Loop [19].*

Vijayamohan *et al.* [20] conducted hydrate experiments on partially dispersed systems using the University of Tulsa Flow loop. The flow loop is 2.9 inch in internal diameter pipe and 162 feet long (Figure 5). It is equipped with a commercial twin screw Liestritz pump to circulate the fluids through the loop. The loop is jacketed and temperature is controlled by the coolant fluids (between 80 °F to 32 °F)circulating in the jacket. The pressure in the loop can be controlled by injecting gas through the gas line which can be found around where the pump discharges fluid. Pressure drop across the pump is read out from the differential pressure transducer PDR8 (Figure 5) located across the pump while the differential pressure across the two long straight sections of the loop is measured through PDR6 and PDR5. The flow loop temperature is measured through thermocouples located along the flow loop. A mass flow meter measures the mass of gas added. The loop has four viewports that can be used for direct visual observation. A sample port is located in the bend opposite to the pump for sample collection throughout the experiment. Hydrate formation can be detected in the loop through loop pressure decrease and visual observation of the viewports.

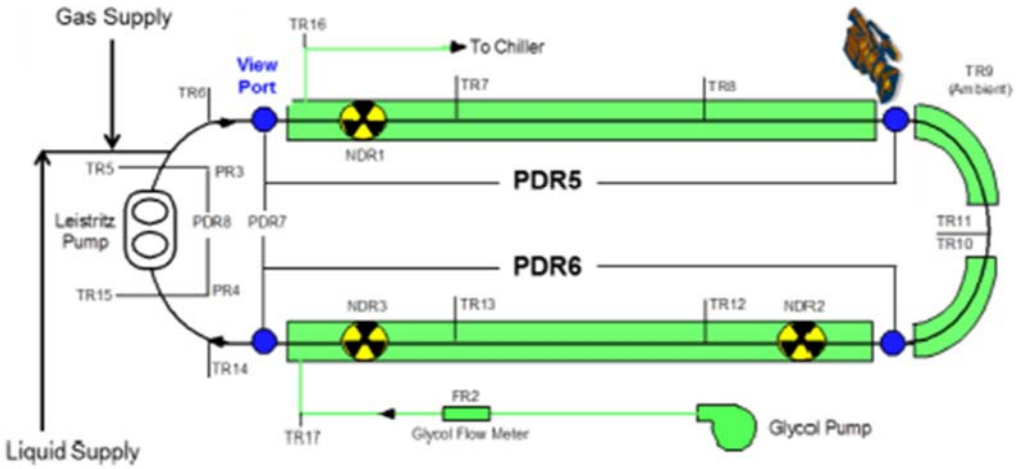

Figure 5. *Schematic of University of Tulsa flow loop [20].*

Lv *et al.* [21] conducted hydrate experiments in China University of Petroleum flow loop. The facility (Figure 6) is a 30m pipe in pipe system with stainless steel of internal diameter 2.54cm covered with another pipe with internal diameter 5.08cm.The annulus between these pipes is filled with a blend of water and glycol used to cool the equipment to temperatures as low as -20°C. Pressure is maintained at a desired value during hydrate formation using a gas make-up tank. Two sight glasses are also installed in the test sections to visually monitor the hydrate formation process. It is equipped with a flow meter for measuring flow rate and liquid mixture density; a Gamma ray densitometer for measuring mean density of the multiphase fluid and a Focused Beam Reflectance Measurements (FBRM) probe for

measuring the evolution of bubbles, droplets, and solid particles. Thermo couples were installed at various points along the loop to monitor loop temperature and Linear pressure drop along the loop is monitored by differential pressure sensors along the loop.

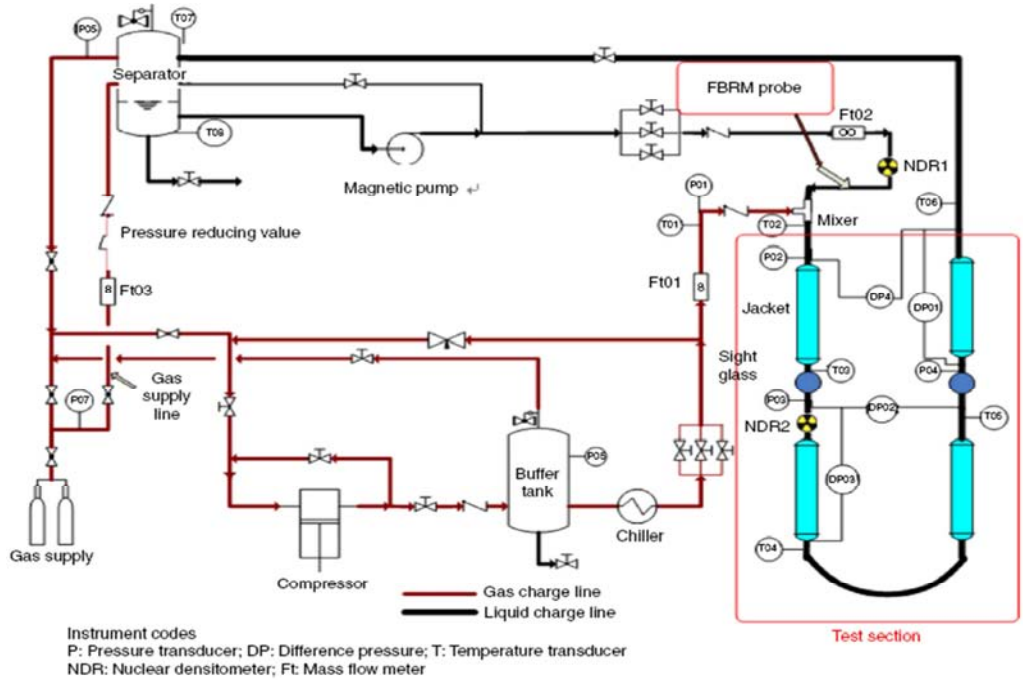

Figure 6. *Schematics of China University of Petroleum Flow Loop [21].*

Sinquin *et al.* [22] discussed multiphase studies carried out on The Lyre Flow Loop (Figure 7). The loop is 140m long with 2inch internal diameter. The 2 inches flow line is double jacketed so as to permit flow of a calorific fluid in counter current pattern. The line is also insulated to reduce energy losses. Thermodynamic conditions allowed in the Lyre Flow loop are: from 0°C up to 50°C for the temperature and pressures from 1 up to 100 bar (1450 psi). Liquid circulation is allowed by a positive displacement pump (flow rate up to $20m^3/h$) and gas by a membrane compressor (flow rate up to $2000Nm^3/h$). The loop can be used to experimentally study the dispersing of hydrate particles in the liquid phase by Low Dosage Additives (LDA).It has a gas make up tank with capacity of $1.2m^3$ which is used in maintaining constant pressure in the loop during hydrate formation experiment. A computer aided supervision system enabled efficient data collection and data sampling at a frequency of 0.25 hertz.

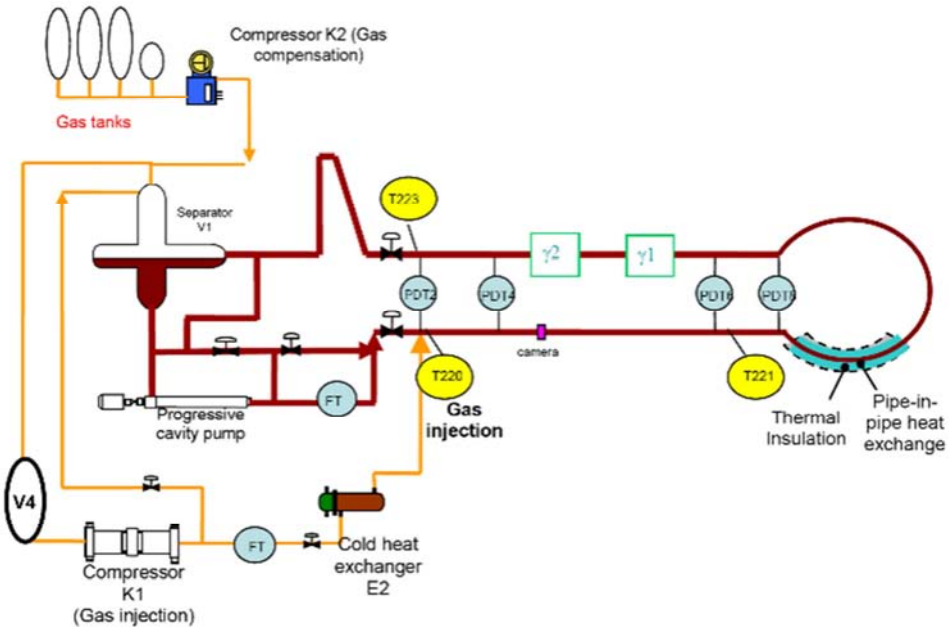

Figure 7. *Schematic of the lyre loop main sensors [22].*

This paper discusses a recently fabricated Hydrate Flow loop (a macroscopic apparatus) and its use in studying hydrate formation and inhibition.

2. Laboratory Flow Loop Design

The design of this laboratory flow loop is a variant of the design by Talaghat [18]. This laboratory flow loop is designed to operate as a constant volume batch process unlike Talaghat design which operates constant pressure experiment.

In the conceptual design, the Laboratory Hydrate loop (Figure 8) is a 12 meters closed loop made with 316 stainless steel tubing of internal diameter of 0.5 inch which can withstand pressures up to 3500psia.

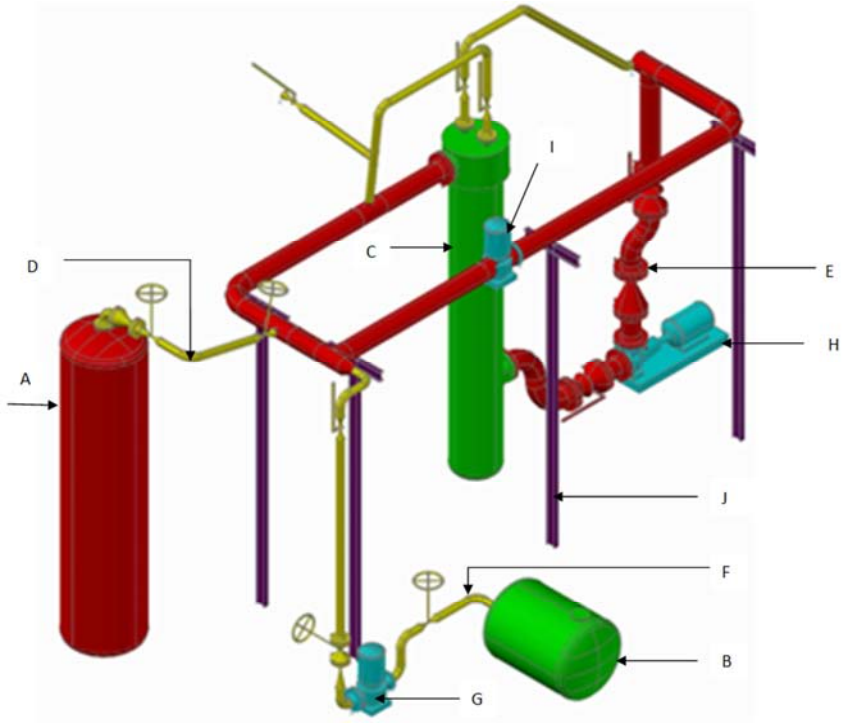

Figure 8. *3D Conceptual design of the Laboratory Hydrate Loop.*

The steel pipe is enclosed in a 4 inch Poly Vinyl Chloride (PVC) jacket where cold water is continuously circulated to cool the steel pipe to temperatures between 0-50°C.This mimics the offshore pipeline that is constantly being cooled by surrounding water.Other components of the loop are: a refrigerator, one variable screw pump for agitating the multiphase fluid in the loop, one inhibitor pump, one cooling water pump, 2 digital differential pressure transmitters, four temperature gauges, and 4 pressure gauges for monitoring the hydrate formation process.

Table 1. *Loop Components and their functions.*

LABEL	COMPONENT	FUNCTION
A	Natural gas storage cylinder	This unit stores the natural gas at a given pressure for use in the loop experiments.
B	Water/ Inhibitor Vessel	This vessel stores water or the premixed inhibitors with water before being introduced to the loop for experimentation.
C	Heat exchanger	This is a refrigerator which is used to regulate the temperature of the loop to hydrate formation temperature during the experiment.
D	Gas stream	This is the stream conveying gas from the natural gas cylinder into the loop.
E	Cooling water stream	This stream conveys chilled water generated from the refrigerator.
F	Inhibitor and water stream	This stream conveys water and inhibitor into the loop to kick start the constant volume experiment
G	Inhibitor/water pump	Pumps water or a mixture of inhibitor and water from the inhibitor/water vessel into the loop through the inhibitor/water stream
H	Cooling water pump	This pump is used to properly circulate cooling water around the loop.
I	Screw pump	This pump is installed for agitation and mixing of the gas and water phases
J	Supporting beams	These support the weight of the loop and helps to keep the loop pipes in place.

Notice that the equipment does not have a gas make up tank because it is designed to operate constant volume experiments which, unlike the constant pressure experiments, does not require gas make up systems. Table 1 discusses the functions of various components in the loop. Details of the equipment design is seen in the first angle orthographic projection showing the front view, side view and plan in 2D (Figue 9).

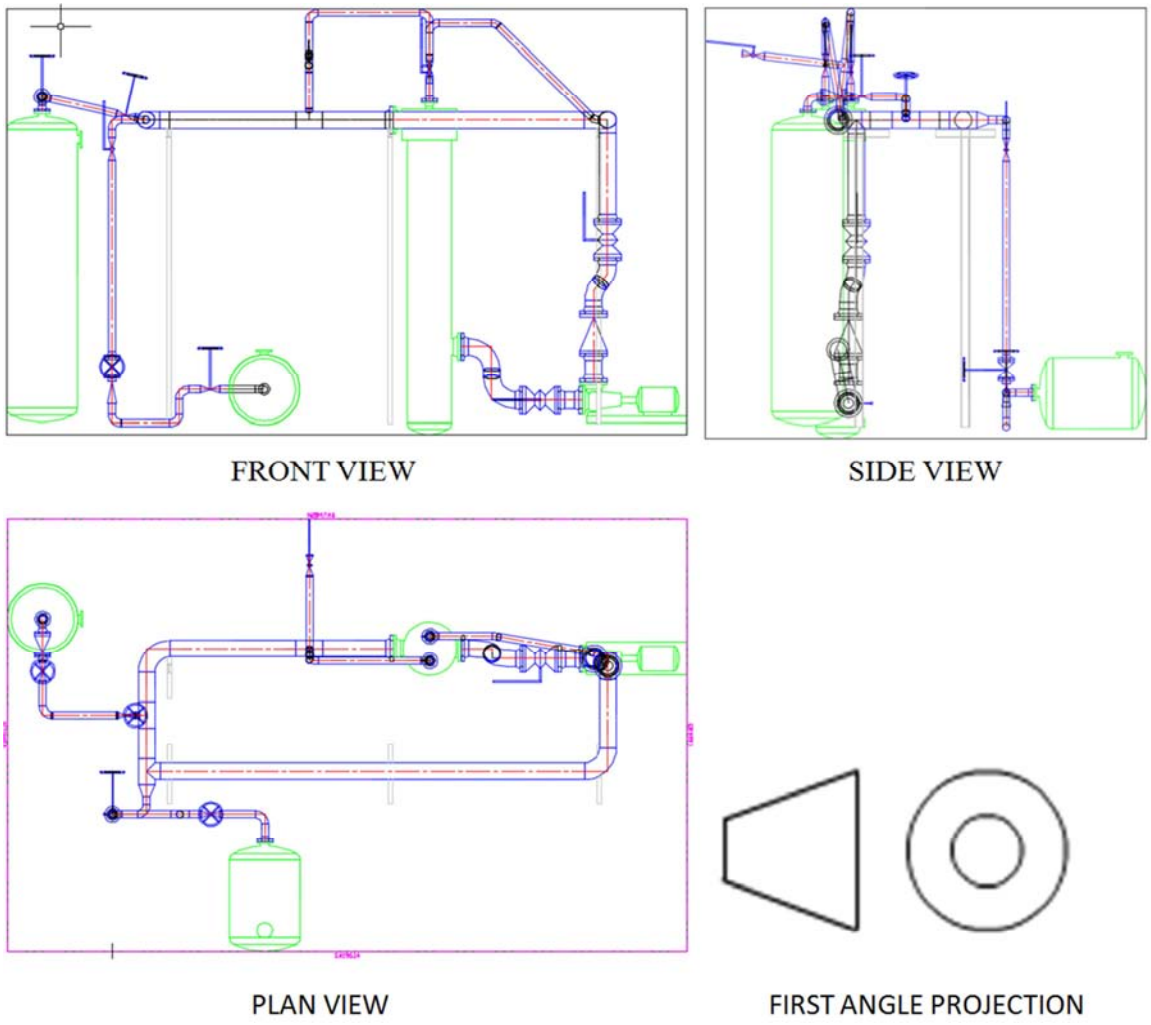

Figure 9. *First Angle Projection of Laboratory Flow loop.*

Figure 10. *Frame Work of Laboratory Loop during construction.*

During fabrication, the loop was placed on a vertical support (Figure 10) capable of withstanding the weight of the 12meters stainless steel pipe, and the weight of the 12 meters 4inch PVC pipe when filled with water. The possible disturbances on the support from screw pump agitation during the experimental procedure was also considered when selecting an appropriate support for the loop.

The process Flow Diagram of the loop (Figure 11) shows the stainless steel pipe (thick line) and the surrounding PVC pipe (thin line) as well as various loop connections described above. About 0.7 meters of the loop is curled in spiral form (Figure 11) and immersed in cold water being generated by the refrigerating unit. This is in order to increase the retention time of the test fluid in the coldest part of the loop. Hydrate formation will likely commence in this spiral part of the loop because it is the coldest part of the loop. A test point is connected from the spiral loop to valve V5 outside the refrigerator using a 1/4" tubing (Figure 11).

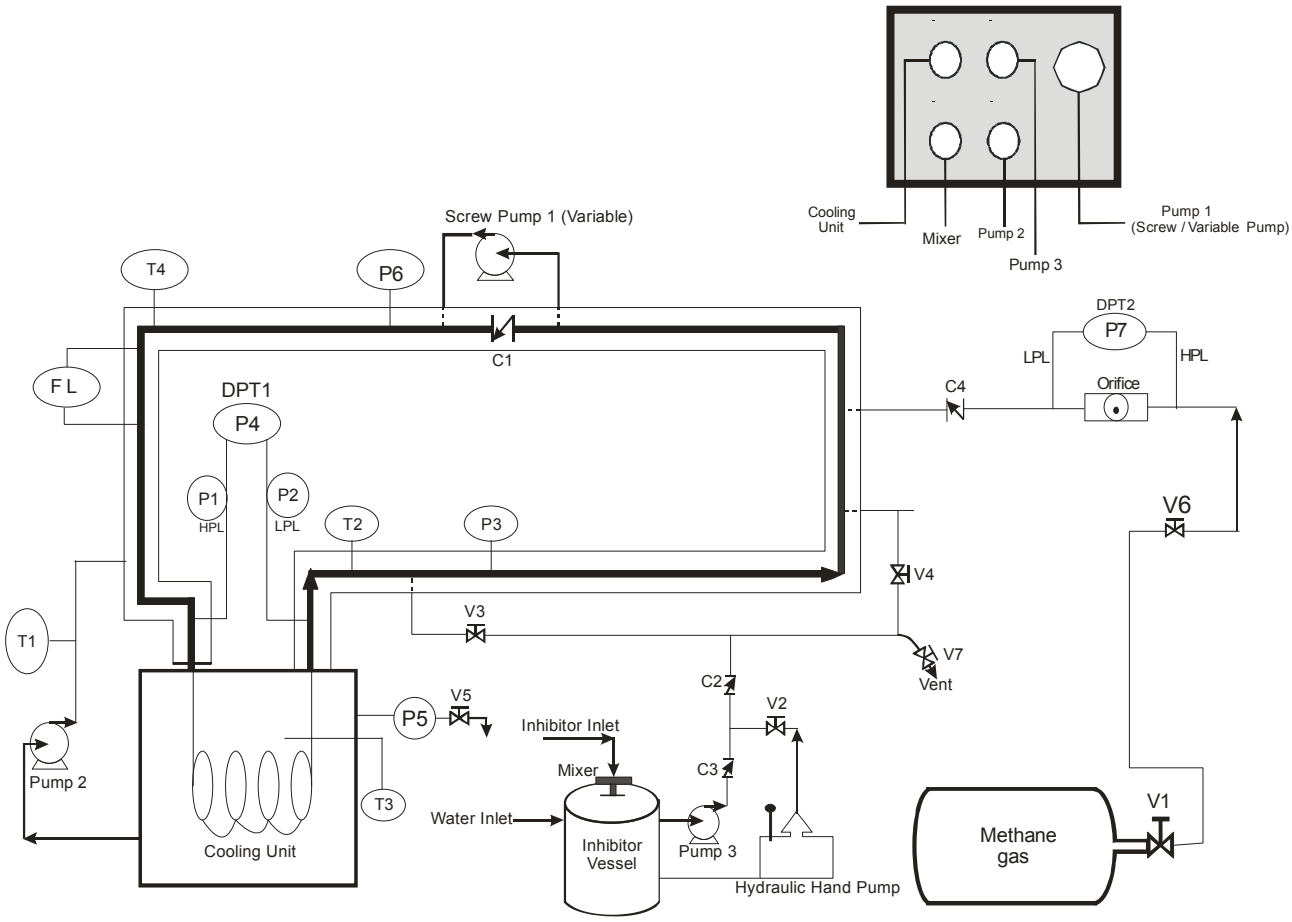

Figure 11. *Process Flow Diagram of Laboratory Hydrate Loop.*

3. Hydrate Formation Experiment

The Laboratory Hydrate loop currently studies hydrate formation in gas dominated systems using a constant volume batch experiment. Gas dominated system was achieved by pumping about 435ml of water into the loop to raise the loop pressure to 25psi and subsequently injecting natural gas(with over 98% methane)into the loop to achieve a pressure of 150psi.The Atlas variable screw pump, Pump 1 (Figure 11) was turned on to effect agitation and circulation in the line. The refrigerating unit was loaded with ice to effect fast cooling of the loop. Cooling water generated from the refrigerator was circulated in the PVC jacket using Pump 2 (Figure 11) in order to lower the temperature of the fluid to hydrate formation temperature. Temperature and Pressure data were acquired every minute throughout the experiment. Hydrate was formed in the loop when natural gas was contacted with water under suitable hydrate forming temperature and pressure conditions. Hydrate formation was detected by direct recording of several events such as: the exothermic peak of the temperature, an increase in differential pressure between the inlet and outlet of the refrigerating unit, and a drastic reduction in loop pressure. Hydrate formation is exothermic in nature (Sloan and Koh, 2008) hence the increase in loop temperature at the onset of hydrate formation. The differential pressure increases due to constriction in the pipe caused by deposited hydrate particles. The loop pressure drops because gas molecules are being used up to form hydrate crystals in the constant volume batch experiment. At the end of each experiment, the loop was depressurized from Valve V5 (Figure 11) and the effluent from Valve V5 examined for evidence of hydrate formation.

4. Loop Validation

The Laboratory hydrate loop was validated by conducting constant volume batch experiment in the loop using single phase fluid of water, single phase fluid of gas and a two phase fluid of gas and water respectively in three different experimental runs. The results of this analysis are presented in Figure 12 to Figure 22.

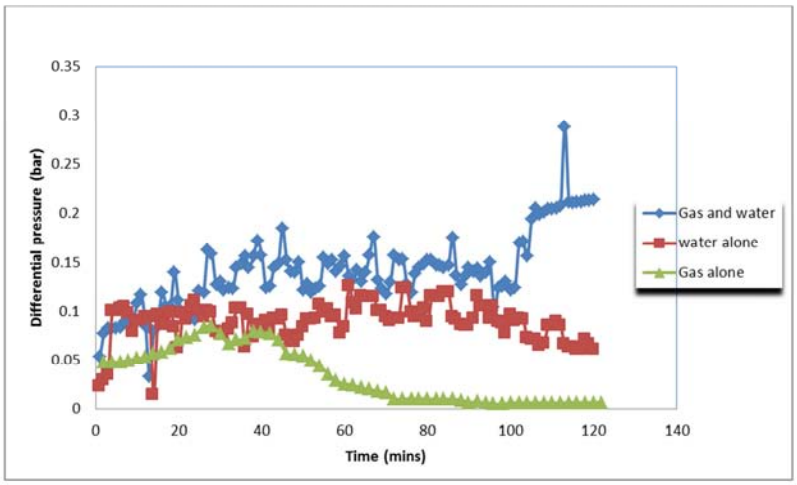

Figure 12. *Plot of Differential pressure against time for loop validation.*

Each experimental run was conducted for 2 hours. The plots of differential pressure versus time for the experimental run conducted with water did not show any significant rise in differential pressure (Figure 12). The differential pressure only oscillated around a value of 0.1 bar but never increased rapidly (Figure 12).This implies that hydrates did not form hence there was no constriction in the pipe due to the absence of hydrate build-up in the loop.

For experimental run conducted with gas, the differential pressure consistently increased to0.084bar (Figure 12)in the first 30 minutes (Figure 12) due to hydrate particles building up around the internal walls of the loop, reducing the effective internal diameter of the pipe. Experimental gas pressure sweeps off hydrate crystals from the pipe walls and this causes a resultant reduction in differential pressure (to 0.0667bar). However, the displaced hydrate crystals soon agglommerate (due to the spiral nature of the coldest part of the loop) and cause pipe restriction as indicated by an increased differential pressure from 0.0667 bar to 0.0799bar at 38 minutes (Figure 12). This differential pressure increase lasted for about 5 minutes after which there was a continuous decline in differential pressure implying that the hydrate particles have been moved out of the spiral part of the loop.

The differential pressure for the experimental run conducted with two phase fluid (gas and water) gradually increases with time until the 100th minute (Figure 12) when the differential pressure increased rapidly to a value of 0.29bar at 112th. This implies that the hydrate crystals formed during the experiment and reduced the pipe internal diameter.

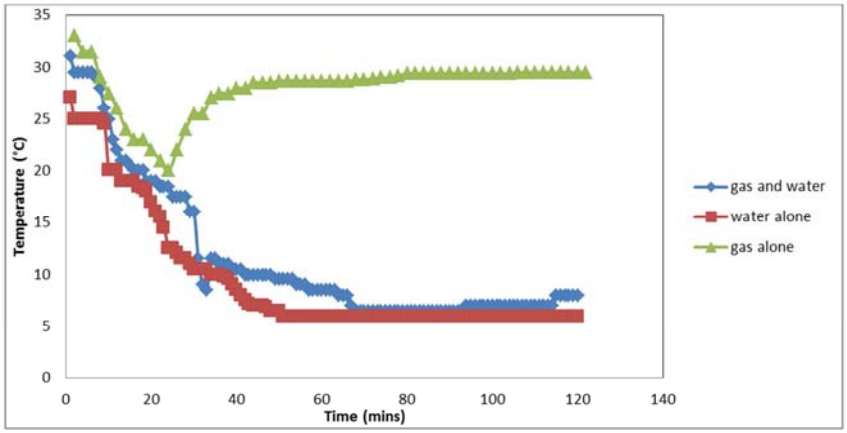

Figure 13. *Plot of temperature versus time for loop validation.*

The plot of temperature versus time for the experimental run conducted with water (Figure 13) shows a continuous decline in temperature of water throughout the experiment. The temperature was initially at 27°C at start of the experiment and it rapidly declined to 6°C within 51 minutes. The loop temperature remained at this value (6°C) until the 2 hours experiment ended. No significant rise in temperature was observed. Hence, no hydrate formation was indicated in

the experimental run conducted with water. The effluent obtained from Valve V5 at the termination of the experiment was clear water (Figure 14).

Figure 14. *Effluent from sample point after water alone was used in experiment.*

When temperature was plotted against time in the experimental run conducted with gas alone (Figure 13), temperature initially declined with time from 33°C to 20°Cat 24 minutes. As the gas was being cooled, water condensed out of the natural gas. The hydrogen bonded water molecules formed cages which were stabilized by the natural gas molecules, forming hydrate crystals as indicated by a rapid rise in temperature from 20°C to 28.5°C at 44minutes (Figure 13). The rise in temperature signifies hydrate formation in the experimental run conducted with gas alone. Notice that the temperature decline after hydrate formation for the gas phase is very slow despite the continuous circulation of cooling water by the cooling water pump. This is because gas is a poor conductor of heat, hence heat gained from the exothermic reaction of hydrate formation was not easily dissipated.

Figure 15. *Effluent obtained on completion of gas and water loop validation experiment.*

In the two phase experimental run conducted with gas and water, the temperature consistently reduced from 31°Cto about 8.5°C at about 32 minutes (Figure 13), after which the temperature increased from 8.5°C to 11°C at 34 minutes. Subsequently, the loop temperature gradually reduced to 6.5°Cat about 67 minutes. The temperature stayed constant at 67 minutes until the 93rd minute where another slight increase in temperature is observed form 6.5°C to 7°C.Temperature then remained at 7°C until the 113th minute when another temperature rise was observed from 7°C to 8°C.The loop temperature remained at 8°C until the termination of the experiment (Figure 13). The observed temperature increase indicates the occurrence of the exothermic reaction of hydrate nucleation and growth in the loop. Hydrate formation was confirmed by observing the loop effluent obtained after loop depressurization (Figure 15) and how it quickly dissociates at atmospheric conditions (Figure 16).

Figure 16. *Hydrate dissociation when exposed to atmospheric conditions.*

The hydrate formation trend can be seen clearly from the plot of Differential Pressure against Time superimposed on the Plot of Temperature against Time in single phase water experiment (Figure 17), single phase gas experiment (Figure 18) and the two phase (gas and water) experiment (Figure 19).Notice that Figures 17, 18 and 19 are the expanded form of Figure 12 and 13.

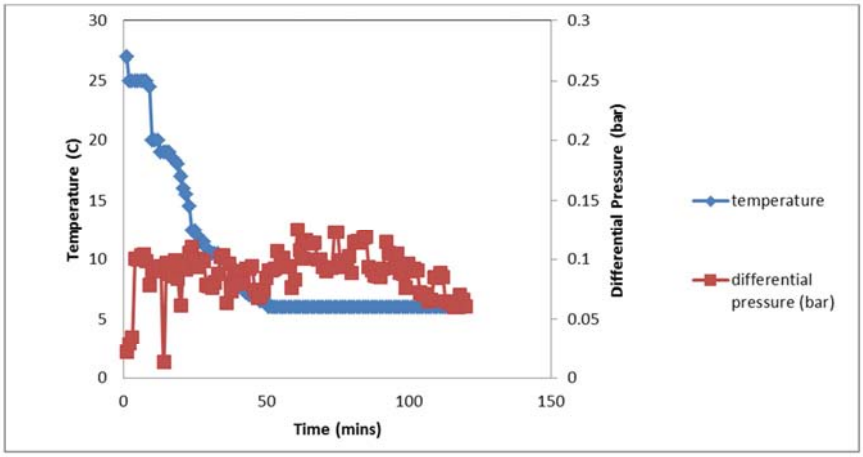

Figure 17. *Plot of differential pressure and temperature versus time for water alone loop validation experiment.*

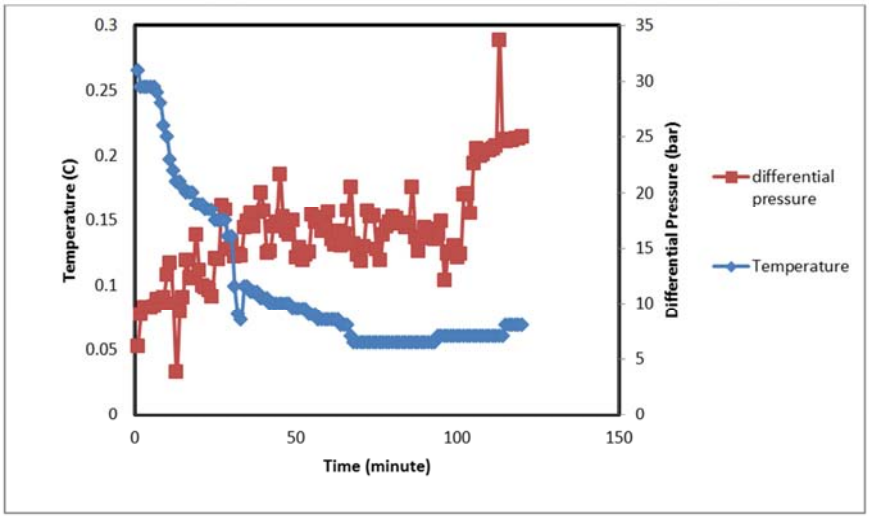

Figure 18. *Plot of Differential Pressure and Temperature versus time for gas and water (2 phase) loop validation experiment.*

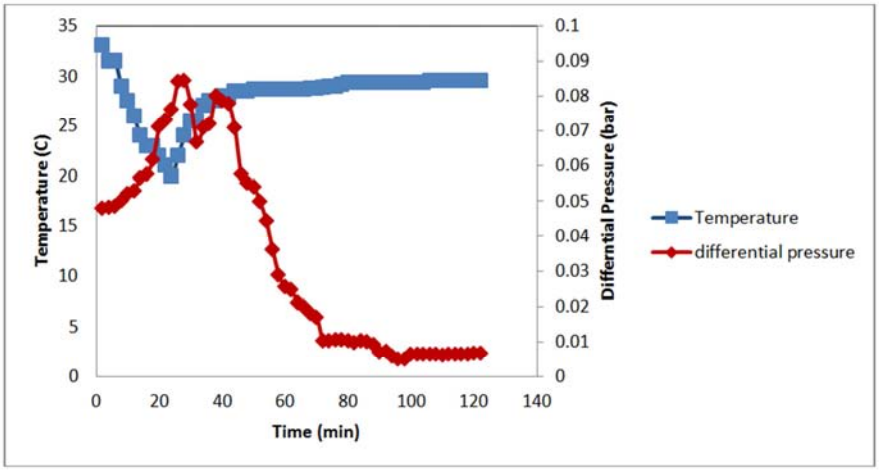

Figure 19. *Plot of differential pressure and temperature versus time for the gas alone loop validation experiment.*

Temperature against time plot was juxtaposed with pressure against time plot for the systems in which hydrates formed: gas and water two phase system (Figure 20) and single phase gas system (Figure 21). A decrease in the pressure of the loop is an indication that gas is being used up to form hydrates. In the two phase experiment (Figure 20), it was observed that the slight rise in temperature from 8.5°C to 11.5°C was accompanied by a decrease in pressure from 80 psi to 77 psi at about 33 minutes.

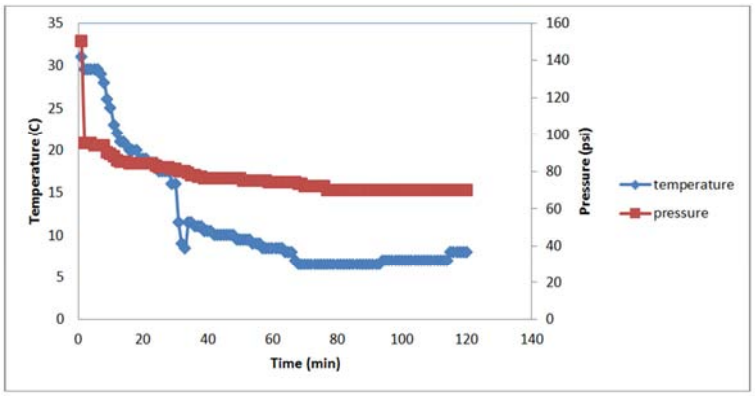

Figure 20. *Plot of pressure and temperature versus time for 2 phase (gas and water) experiment for loop validation.*

In the experimental run conducted with gas (single phase), the loop temperature initially decreases from 33°C to 20°C at about 24minutes after which the temperature rises and stabilizes at about 29°C. Pressure declined from 140 psi to 84psi within the first 24 minutes. Rapid pressure decline (84 psi to 2psi) corresponding to the temperature increase was observed in about 44 minutes (Figure 21). This implies that as hydrates formed, an exothermic reaction occurred causing the increase in temperature which was accompanied bya corresponding decrease in the quantity of gas available in the loop hence, a pressure decline.

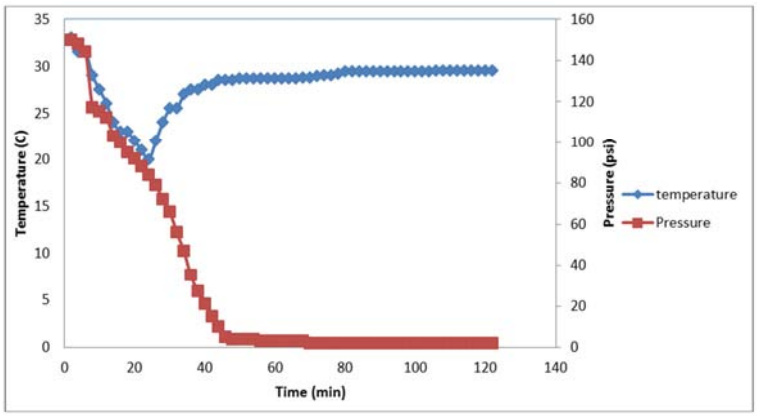

Figure 21. *Plot of temperature and pressure versus time for loop validation experiment using gas alone.*

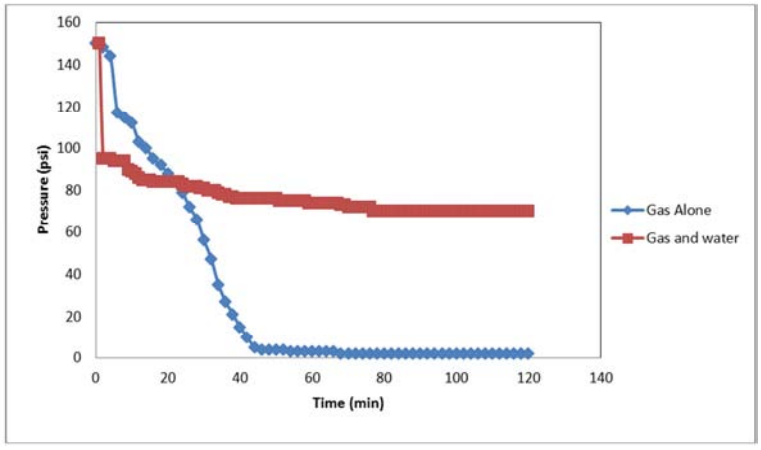

Figure 22. *Plot of pressure versus time for gas alone loop validation experiment.*

A plot of pressure versus time for the systems in which hydrate formed showed the variation in pressure depletion in these systems (Figure 22). In the two phase (gas and water) system, the pressure depletion was from 150psi to about 70psi while in the gas alone experiment, almost all the gas was used up as the pressure decline was from 150psi to 2 psi.

The various plots above showed evidences of hydrate formation in the loop in the presence of hydrate formers (gas) and water at the appropriate pressure and temperature. The effectiveness of N-Vinyl Caprolactom, PolyVinylPyrrolidone and 2-(Dimethylamino)Ethylmethacrylate have been studied using this equipment and documented in Odutola et al [23], [24].

5. Conclusion

A laboratory flow loop has been designed and fabricated. It currently operates as a constant volume batch process at temperature between 0-50°C and pressure up to 200psi. It has been used to study hydrate formation in gas dominated flow. It can adequately predict hydrate formation in gas pipelines and can be used in screening and selecting Kinetic Hydrate Inhibitors.

Appendix

Figure A1. *Current view of Laboratory Hydrate loop.*

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