# **Design and Operational Procedures for a Locally Made Steam Distillation Apparatus**

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## Abstract

In Trinidad, oil production started just over 100 years ago and steam flood operations started just under 50 years ago. In steam flood operations, oil recovery by steam distillation can be in the range of 5 to 60 % and therefore requires separate experimental and mathematical studies for accurate steam flood predictions. The steam distillation apparatus required for the experimental study can be quite costly. In order to conduct steam distillation studies in the Petroleum Studies Unit in Trinidad, a steam distillation apparatus was designed, fabricated and tested to perform these studies, as an integral part of the experimental steam flood studies on Trinidad crude oils.

The apparatus consist of a positive displacement pump, a steam generator, a steam distillation cell, a temperature measurement and control system, a back pressure valve and a condensing and liquid collection system. The steam generator and steam distillation cell were fabricated inhouse, from stainless steel and were designed to conduct steam distillation studies at a safe working pressure of 4.654 MPa and temperature of 260 °C. From the operational procedures outlined in this study and from repeat test runs conducted at 100 °C and 260 °C steam distillation results were reproduced with differences of less than  $\pm 4.0$  % between the original and repeat runs. Details of the apparatus design and operational procedures from this study can provide a useful guide for other Researchers on crude oil steam distillation studies.

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# INTRODUCTION

Steam distillation is a term applied to a distillation process wherein steam is in direct contact with the distilling material (Perry, 1950). Steam distillation of crude oil occurs during steam flooding of oil reservoirs and has been recognized as one of the major mechanisms responsible for high oil recovery by the steam flooding process (Duerksen & Hsueh, 1983). From steam flood studies, Farouq Ali (1968) estimated the recovery range to be about 5 to 10 % of the original oil in place for heavy oils and as much as 60 % for light oils. Steam distillation data is therefore included in the evaluation of an oil reservoir for steam flooding, steam flood simulation studies and for the optimization of a steam flood design. This data is best determined from experimental studies or from mathematical models developed from experimental data.

The effect of steam distillation on oil recovery has been investigated experimentally in several laboratories with and without a porous media (Willman *et al.*, 1961; Ozen & Farouq Ali, 1969; Quinones, 1971; Wu & Brown, 1975; Wu & Elder, 1980; Duerksen & Hsueh, 1983). The conclusions drawn from each study, led to modifications in apparatus design and laboratory procedures for conducting steam distillation experiments quickly and accurately and also for the development of models for predicting steam distillation yield accurately.

An early attempt to study the steam distillation process by simulating steam zone conditions was conducted by Quinones (1971). In his investigation he designed experimental techniques for studying the steam distillation effects of crude oils with and without a porous medium present. The apparatus was designed to conduct experiments under constant run temperatures and pressures using saturated steam. In his experimental procedure, the hydrocarbon was first heated to the distillation temperature as suggested by Hengstebeck (1961). Next steam at the same distillation temperature was made to pass through the hydrocarbon until the distillation pressure was reached. The effluent vapors were condensed and collected and their respective volumes measured. Pre-heating the hydrocarbon charge reduces the experimental time. From the experimental runs conducted in the core, Quinones (1971) stated that the steam distillation effect in a porous media is similar to that for a hydrocarbon batch. However, the properties of the porous medium must be taken into account when developing steam distillation prediction methods.

Conclusions drawn by Quinones (1971) led Wu and Brown (1975) to study the effects of porous media and process parameters on the steam distillation process. The steam distillation studies were conducted in a verticallyoriented, 91.44 cm long 7.62 cm diameter cell designed so that steam distillation tests were performed in either an open cell (without porous media) or in a packed cell (with sand). By plotting steam distillation yield,  $V_0/V_{0i}$  (the ratio of the collected oil condensate, V<sub>0</sub> and initial oil volume, V<sub>0</sub>) against steam distillation correlation parameter, V<sub>w</sub>/V<sub>oi</sub> (the ratio of the collected steam condensate, Vw, and initial oil volume,  $V_{oi}$ ), Wu and Brown (1975) observed that yields were independent of the porous medium used, steam-injection rate, and initial oil volume. However, it is necessary to select an oil volume so as to obtain measurable distillate volumes, and a steam injection rate so as to allow enough time to measure and record distillate volumes from time to time. More importantly enough time for mixing of steam and oil is necessary so as to avoid or reduce steam channeling to a minimum.

Wu and Elder (1980) adopted the above experimental guidelines and conducted steam distillation studies in an

open cell that started with a pre-heating phase, followed by a steam injection rate of 320 cm<sup>3</sup>/h and using an initial oil volume of approximately 200 cm<sup>3</sup>. Their steam distillation runs lasted between 16 and 24 hours at a steam distillation factor of 15. Steam distillation studies conducted by Duerksen and Hsueh (1983) covered a wide range of varying saturated steam conditions; the minimum was at 104 °C and 0.115 MPa and the maximum was at 260 °C and 4.654 MPa.

Trinidad has a heavy oil reserve of about 1.0 billion barrels on-land, of which 40 to 60 % can be recovered by steam flooding (Hosein *et al.*, 2011). In this paper, we describe the design and outline the operational procedures of a steam distillation apparatus that was locally built for performing steam distillation studies, as an integral part of the experimental steam flood studies on Trinidad crude oils.

# 1. THE STEAM DISTILLATION APPARATUS

A schematic diagram of the steam distillation apparatus designed and assembled for the steam distillation of Trinidad crude oils is shown in Figure 1 and a photograph in Figure 2. The apparatus, mainly fabricated from stainless steel (SS 314), comprise: a positive displacement pump and water reservoir, a steam generator and steam line, a steam distillation cell and thermocouples, a temperature control and display system, a back pressure valve and safety valve and a condensing and liquid collection system. The steam generator and steam distillation cell were fabricated in-house, from stainless steel and were designed to conduct steam distillation studies at a safe working pressure of 4.654 MPa and temperature of 260 °C. The sizes of the steam generator and steam distillation column (details given below) were determined in accordance with the specifications stated in American Society for Testing and Materials ASTM standards (Perry, 1950).







Figure 2 Steam Distillation Apparatus

#### 2. DESIGN CONSIDERATIONS

#### 2.1 Steam Generator and Steam Line

A schematic diagram of the steam generator designed and assembled is shown in Figure 1 and a photograph in Figure 2. It was made from a 23.0 cm long by 3.8 cm I.D. stainless steel (SS 314) tubing, of wall thickness 4.8 mm. From these dimensions and a safe working pressure of 4.654 MPa and temperature of 260 °C, a flange thickness of 2.54 cm was selected (allowing for a high safety factor). The inside of the generator was packed with pieces of stainless steel tubing with dimensions 1.3 cm to 2.5 cm long and 3.175 mm I.D. which provide a larger surface area for heating. The flanges were bolted together by applying a torque of 13.8 N on alternate bolts. The gasket material used between the flanges is CHESTERTON 235 (a brand name) which is 3.175 mm thick.



Schematic of Heater Connections

The heat input was provided by four (4) heaters which were connected to each other as shown in Figure 3. The heaters were made from Nichrome coils, each of which is 40.6 cm long. According to specifications, the power rating per heater is 1500 Watts using a 120 Volt supply. The heaters were electrically insulated from each other with ceramic ferrules and wrapped around the stainless steel pipe as shown in Figure 5. The heaters were then cemented in place and covered with ceramic cement BD6 (BD 6 is a brand name) so as to enclose and insulate thermally. Before installing the heaters, it was necessary to provide reinforcement for the cement. This was done by wrapping a 6 mm square wire mesh closely around the stainless steel pipe. The wire was then insulated with a thin layer of cement and allowed to dry for a period of twelve (12) hours before the heaters were installed. Although the cement will crack when operating the generator at high temperatures, this reinforcement prevents it from falling apart.

The generator was then enclosed with an aluminum casing which was lined inside with fiber glass insulation so as to reduce heat losses. It was then mounted in a vertical position so that water from the reservoir enters at the top and steam will leave at the bottom through a steam line. In this position, water build up in the generator was prevented.

Steam leaving the steam generator travels through a 61.0 cm long by 6.35 mm I.D. stainless steel tubing and a check valve and enters the steam distillation cell from the bottom. Two 500 Watt heaters made from Nichrome coils were electrically insulated from each other with ceramic ferrules, and installed along the entire steam line. A thermocouple wire was installed on the steam line near the bottom of the generator so as to monitor steam temperatures and a pressure gauge for monitoring steam pressures, followed by a needle valve. With this

arrangement, constant saturated steam conditions of 100 °C and 0.101 MPa, 149 °C and 0.448 MPa, 204 °C and 1.689 MPa, and 260 °C and 4.654 MPa were attained.

## 2.2 The Steam Distillation Cell

A schematic diagram of the steam distillation cell designed and assembled is shown in Figure 6 and a photograph in Figure 7. It has a similar construction to that of the steam generator. It was made from a 91.0 cm long by 3.81 cm I.D., 314 stainless steel tubing of wall thickness 6.35 mm. With these dimensions and a working pressure of 4.654 MPa and temperature of 260 °C, a suitable flange thickness of 3.81 cm was selected (allowing for a high safety factor). The inside of the cell was lined with a 1.58 mm thick copper jacket, welded inside so as to provide a uniform temperature distribution. The cell was mounted in a vertical position and the flanges were bolted together by applying a torque of 20.7 N on alternate bolts. The gasket material is also CHESTERTON 235, but is 6.35 mm in thickness. A total of twenty-one (21) heaters were installed on the cell. Each heater is 61 cm long and has a power rating of 1000 Watts according to the heater specifications. The method of installation was similar to that for the steam generator. Six thermocouples were installed, six inches apart along the entire length of the cell as can be seen in Figures 6 and 7. The ends of these thermocouples were positioned at varying points inside the cell. A further two (2) thermocouples were installed on the bottom flange and their ends were positioned 1.2 cm and 3.8 cm inside the cell.

# 2.3 Thermocouples

The thermocouples were locally made from chromel and alumel (type k) wires. The ends of these two thermocouple wires were fused together so as to form a junction. This junction was then insulated with a fine bore capillary glass tubing. The insulated junction was housed inside a 3.175 mm I.D. stainless steel tubing with one end sealed so as to withstand high pressures. A 3.175 mn swagelok-NPT (National Pipe Thread) fitting was drilled through so that the 3.175 mm tubing can pass through it. The fitting was bolted in position on the steam distillation cell. The 3.175 mm stainless steel, housing the thermocouple was then positioned inside the cell through this fitting. The cap-nut with ferrules held the thermocouple housing fixed with a high pressure seal. Installation of the thermocouple for measuring steam temperature was much simpler. A reducer, from 6.35 mm swagelok to 3.175 mm swagelok was used on the steam line near the generator.

## 2.4 Condenser and Liquid Collection System

Vapors leaving the steam distillation cell entered a 61.0 cm long, locally made water cooled condenser, which was attached to a liquid-gas separator. The liquid from the separator was collected in 10 cm<sup>3</sup> measuring cylinders and then placed in a 1000 cm<sup>3</sup> Erlenmeyer flask. Surrounding the separator is an ice jacket which cools any vapor escaping from the condenser, Embedded in the ice is a copper coil through which tap water flows, and is cooled by the ice. This cold water flows up the condenser and cools the hot effluents from the distillation cell.



Figure 4 Schematic of Steam Generator



Figure 5 Steam Generator

#### 3. OPERATIONAL PROCEDURES

Basically, the operation involved changing a predetermined volume of oil into the distillation cell and heating to the desired temperature. Steam at the same temperature was then injected into the cell until the desired run pressure was achieved. Water and hydrocarbon vapors were condensed and their respective volumes measured. From this operation, a laboratory procedure was developed to determine crude oil steam distillation yields. In general, the procedure was divided into two phases: the first phase involved cell preparation and the second involved the actual distillation tests (Hosein, 1989).

The procedures for the distillation test runs were as follows: An oil sample of volume 200 cm<sup>3</sup> was injected from the bottom of the distillation cell using an ISCO pump. This was followed by the injection of 500 cm<sup>3</sup> of water into the steam generator and steam line. The distillation cell and the steam generator were then heated to the run temperature, after which water was injected continuously into the steam generator at 360 cm<sup>3</sup>/h. The steam bleed off valve was regulated until the run pressure and temperature were achieved with the saturated steam. The steam bleed off valve was then closed off. For safe operation, the back

pressure valve and variable transformers were set to maintain a constant run pressure and temperature and the valve connecting the steam generator and steam distillation cell was then opened (see valve arrangements in Figure 1). When the desired run temperature and pressure were achieved in the steam generator and steam distillation cell, the valve at the top of the distillation cell was opened to allow vapors to enter into the condenser and oil and water volumes were measured. A run was terminated after a produced oil/water ratio of 0.01 cm<sup>3</sup> has been recorded at least ten (10) times.

# 4. SAMPLE AND TEST RUN DESCRIPTION

The oil sample used to test the equipment has a gravity of 30.6 °API and falls under the description of intermediate oils (between light and heavy oils) and a distillation yield as high as 60 % is to be expected (Farouq Ali, 1968). Each test run that was conducted (original run) was repeated (repeat run) so as to ensure that similar results can be obtained with the equipment and the operational procedures that were outlined for the equipment.



Figure 6 Schematic of Steam Distillation Cell

Figure 7 Steam Distillation Cell

#### 5 RESULTS AND DISCUSSION

#### 5.1 Original and Repeat Runs

#### Table 1

Comparison of Steam Distillation Data for Original and Repeat Runs at Saturated Steam Temperature,  $T = 100 \text{ }^{\circ}\text{C}$  and Pressure, P = 0.101 MPa

<b>Steam Distillation</b>	Steam Distillation Yield (V <sub>o</sub> /V <sub>oi</sub> )			
Factor (V <sub>w</sub> /V <sub>oi</sub> )	Original Run	Repeat Run	Diff.,%	
0.10	13.00	13.40	3.08	
0.25	17.15	17.35	1.17	
0.50	21.35	21.50	0.70	
1.00	25.05	25.00	-0.20	
2.00	28.75	28.80	0.17	
3.00	31.65	31.90	0.79	
4.00	33.05	33.25	0.61	
6.00	34.90	35.25	1.00	
8.00	36.25	36.55	0.83	
10.00	36.50	36.80	0.82	

Table 2

Comparison of Steam Distillation Data for Original and Repeat Runs at Saturated Steam Temperature,  $T = 260 \text{ }^{\circ}\text{C}$  and Pressure, P = 4.654 MPa

<b>Steam Distillation</b>	Steam Distillation Yield (V <sub>o</sub> /V <sub>oi</sub> )		
Factor (V <sub>w</sub> /V <sub>oi</sub> )	Original Run	Repeat Run	Diff.,%
0.10	11.85	11.60	-2.11
0.25	16.90	16.45	-2.66
0.50	24.60	24.30	-1.22
1.00	33.10	32.90	-0.60
2.00	41.55	41.30	-0.60
3.00	49.70	49.55	-0.30
4.00	53.90	53.65	-0.46
6.00	56.05	55.95	-0.18
8.00	57.25	57.00	-0.44
10.00	58.75	58.55	-0.34

Tables 1 and 2 show the data obtained for the original and repeat runs obtained for the 30.6 °API Trinidad oil sample at saturated steam temperatures of 100 °C and 260 °C. Steam distillation yield,  $V_o/V_{oi}$  (%) is the ratio of the collected oil condensate,  $V_o$  and initial oil volume,  $V_{oi}$  and steam distillation factor,  $V_w/V_{oi}$  is the ratio of the collected steam condensate,  $V_w$ , and initial oil volume,  $V_{oi}$  (Wu and Brown, 1975) The differences in the steam distillation yield data between the original and repeat runs were less than  $\pm$  4.0 %. This small difference indicated that results can be almost reproduced from the repeat runs by following the operational procedures outlined for the equipment. From these test runs, the highest distillation yield obtained with this sample was 58.0 % (see Table 2), which is similar to the results obtained by Farouq Ali (1968).

# 5.2 General Operational and Experimental Considerations

All distillation runs were conducted with the cell held in a vertical position, so as to avoid steam displacement of oil during steam distillation. As discussed by Wu and Brown (1975) a porous media was not used in the cell for conducting runs. As such, the difficulty of core packing before runs and core removal after runs was eliminated. Pre-heating the crude sample to the desired run temperature before injecting steam, reduce experimental time considerably. A steam injection rate of 360 cm<sup>3</sup>/h allowed enough time to monitor and regulate constant run temperature and pressure, record distillate volumes and change measuring cylinders when filled. With these experimental considerations, and following the experimental procedures outlined above, an experimental run lasted between twelve (12) to sixteen (16) hours.

#### CONCLUSIONS

(1) A steam distillation apparatus was locally made and tested for conducting steam distillation experimental studies on Trinidad oil.

(2) Details of the apparatus design and operational procedures from this study can provide a useful guide for other Researchers on crude oil steam distillation studies.

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