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Using Biofuels as Lubrication Oil

By Sabah Alali, Meshal Aldaihani, Waleed Abuhaimed & Khaled Alanezi

College of Technological Studies

Abstract- As environmental concerns grow, vegetable oils are finding their way into lubricants for industrial and transportation applications. These oils offer significant environmental benefits with respect to resource renewability and biodegradability, as well as providing satisfactory performance in a wide array of applications.

This paper aims to determine the properties of different biofuel lubricants prepared from vegetable oils. The investigations of temperature and viscosity properties of the lubricating oil were established and their effect on the property changes of the lubricant.

Experiments are created based on SVM test machine, the results show density, absolute viscosity, and kinematic viscosity of three vegetable oils, the tested oils are Palm, Corn, and Almond oils.

Keywords: *biofuels, lubricants, vegetable oils, environment.*

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Using Biofuels as Lubrication Oil

Sabah Alali ^α, Meshal Aldaihani ^σ, Waleed Abuhaimeed ^ρ, Abdulqader Alfuraj ^ω & Khaled Alanezi [¥]

Abstract- As environmental concerns grow, vegetable oils are finding their way into lubricants for industrial and transportation applications. These oils offer significant environmental benefits with respect to resource renewability and biodegradability, as well as providing satisfactory performance in a wide array of applications.

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I. INTRODUCTION

The use of natural fats and oils by man date back to very ancient times. Fats and oils have specific chemical properties which make them very useful as lubricants. Vegetable, animal and marine sources are the main sources of natural fats and oils and their chemical nature would make them very useful in many applications. Fats and oils are naturally occurring substances that consist predominantly of mixtures of fatty acid esters derived glycerol [1].

Vegetable oils are a renewable energy source in that they are created from plants that can be regrown and they consist mainly of tri-esters of straight-chained, mostly unsaturated fatty acids with glycerol. [2, 3]. What distinguishes these vegetable oils is that they have higher levels of biodegradability and lower toxicity than conventional mineral or synthetic oils. Therefore, these biofuels can be utilized with ease in cars and other places since they do not require much chemical or physical property changes. Many scientists believe that the use of biofuels as carbon neutral would be beneficial to the environment since the carbon produced when burning them is offset by the carbon consumed by the plants they came from. Research suggests that using biofuels would help in reducing carbon emissions by 50-60%. They are other benefits for using vegetable oils as biofuels, for example biofuels from vegetable oils have a very low volatility, good high lubricity and high viscosity index, as well as lower cost than most synthetic oils [4, 5, 6]. Many researchers have pointed out that the use of

biofuels as lubricants has its own drawbacks for example using land for biofuel crops this will mean less land for food production. Also, vegetable oils have low thermal and oxidative stabilities, narrow viscosity range and higher pour points than both mineral or synthetic oil-based lubricants, that's why vegetable oils have a limited use as lubricants and their use in the industry is not yet extensive [7, 8, 9].

- Lubricant's performance for a certain system is a very important characteristic that should be quantified and it can be determined by carrying out specific experimental tests. The lubricity of a substance is not a material property which cannot be measured directly. The lubricity of the fuel is an indication of the amount of wear or scarring which happens between two metal parts covered with the fuel as these metal parts come in contact against each other. Low lubricity fuel may cause high wear and scarring and high lubricity fuel may provide reduced wear and longer component life. Biofuels such as vegetable oils are used as lubricants, specifically, when there is a leakage of equipment or where the system is designed to function by loss lubrications. Systems, such as: Open gear lubricants, arming, mining, and forestry equipment, hydraulic oils etc.

The viscosity index (VI) is a very important property of the biofuels, which is a relative measurement in change of base fluid viscosity between 40°C and 100°C. The viscosity index indicates the change in viscosity over an extended temperature range. It is well documented that vegetable oils display very high viscosity indices (VI) compared to mineral oils. Also, vegetable oils can afford higher flash points as compared to mineral oils [10].

Vegetable oils are high-performance base oils which are extracted from coconut, palm and soy. These vegetable oils are considered as lubricants and they are compatible with additives currently used in the lubrication industry. These vegetable oils have many advantages such as: low cost, acceptable low-temperature properties, and acceptable oxidative and thermal stability. Fatty acids are normally contained in some vegetable oils and it would improve its lubricity, for example fatty acids that exist in the palm oil, tend to cling to metal surfaces more effectively than mineral oils and therefore provide improved lubricity. The research main point is using biofuels as light lubricant with low heat applications like joints, track, bearings. So, the experiments were carried out at various temperatures

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and the results showed that oil viscosity will change with temperature, so the temperature range is the main point.

Table (1): Several types of vegetable-based lubricants developed for industry applications (11)

Canola oil	Hydraulic oils, tractor transmission fluids, metalworking fluids, food grade lubes, penetrating oils, chain bar lubes
Castor oil	Gear lubricants, greases
Coconut oil	Gas engine oils
Olive oil	Automotive lubricants
Palm oil	Rolling lubricant,-steel industry, grease
Rapeseed oil	Chain saw bar lubricants, air compressor-farm equipment, Biodegradable greases
Safflower oil	Light-colored paints, diesel fuel, resins, enamels
Linseed oil	Coating, paints, lacquers, varnishes, stains
Soybean oil	Lubricants, biodiesel fuel, metal casting/working, printing inks, paints, coatings, soaps, shampoos, detergents, pesticides, disinfectants, plasticisers, hydraulic oil
Jojoba oil	Grease, cosmetic industry, lubricant applications
Crambe oil	Grease, intermediate chemicals, surfactants
Sunflower oil	Grease, diesel fuel substitutes
Cuphea oil	Cosmetics and motor oil
Tallow oil	Steam cylinder oils, soaps, cosmetics, lubricants, plastics

The experimental data obtained of vegetable oils showed that low values of kinematic viscosities at 40°C but higher values of kinematic viscosities at 100°C (12) with higher viscosity index range from 203 to 263. Also, vegetable oils viscosity showed much variation over temperature range and have instability and poor temperature properties. Results showed that corn oil and soybean oil were more viscous at high temperatures than synthetic oil.

II. THE SVM MACHINE

Absolute and kinematic viscosity measurements procedure were used to find the viscosity with temperature relationship from different bio-fuels. The device shown in figure (1) is the SVM 3000 and its measuring process depends on torque and speed measurements. The SVM 3000 uses a rotating magnet which produces eddy current field.



Figure (1): SVM 3000 device

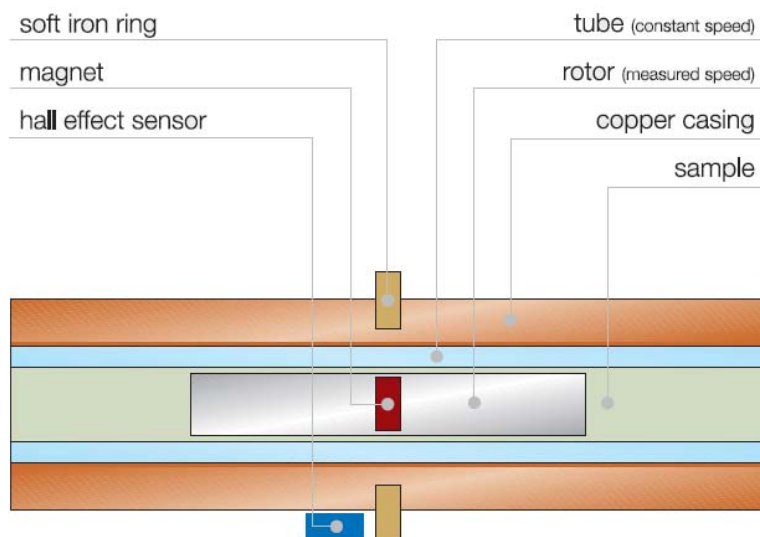


Figure (2): SVM 3000 measuring unit main components

III. DATA AND RESULTS

Experiment and results are created based on certain procedure related to SVM3000 machine. Corn, Palm, and Almond oils were tested based on Absolute viscosity, kinematic viscosity, and density. The oil samples are taken from commercial oil products available in market, the test is done in College of Technological Studies (CTS) in PAAET (Public Authority of Applied Education and Training-Kuwait).

The first group of results showed the relation between temperature and oil specific gravity as shown in figure (3). The results show that as the temperature increases a decrease in the Corn, Palm, and Almond oils specific gravity was noticed. Therefore, it can be deduced that the specific gravity of these vegetable oils (density) is dropped with increasing temperature.

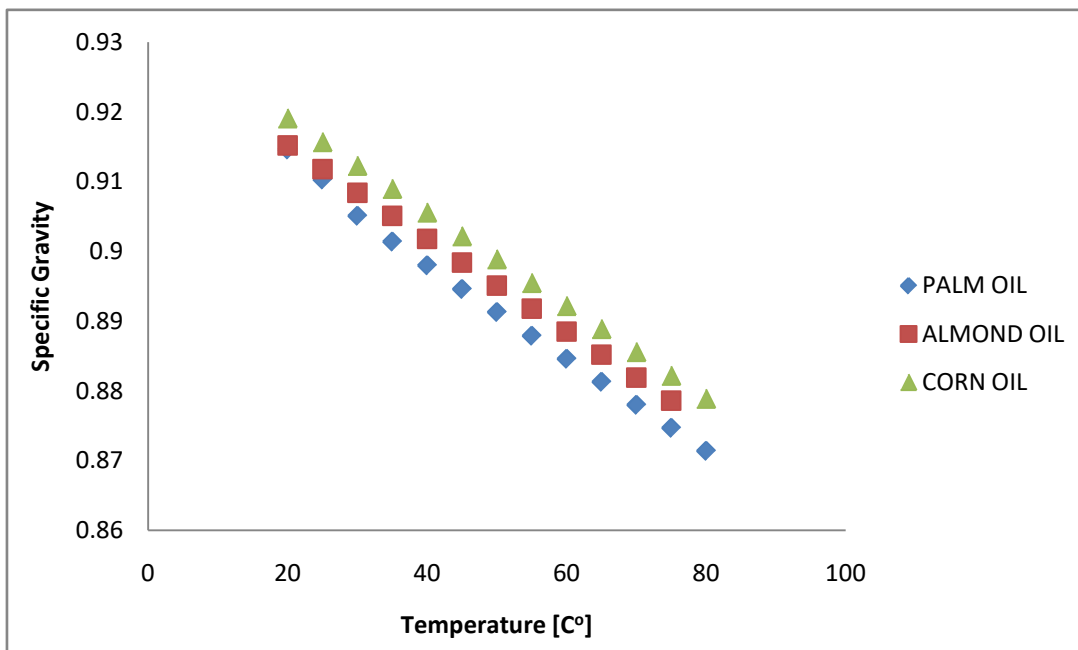


Figure (3): Bio Fuel (oils) Specific gravity vs. temperature

The relation between vegetable oils specific gravity and temperature is linear, by using regression the linear relation is obtained for all oils (Corn, Palm, and Almond). In figure (4) all types of Bio fuels have the

same linear inclination (slope), so the behavior of the tested Bio-fuels with temperature is the same if the view point is the oil density or specific gravity.

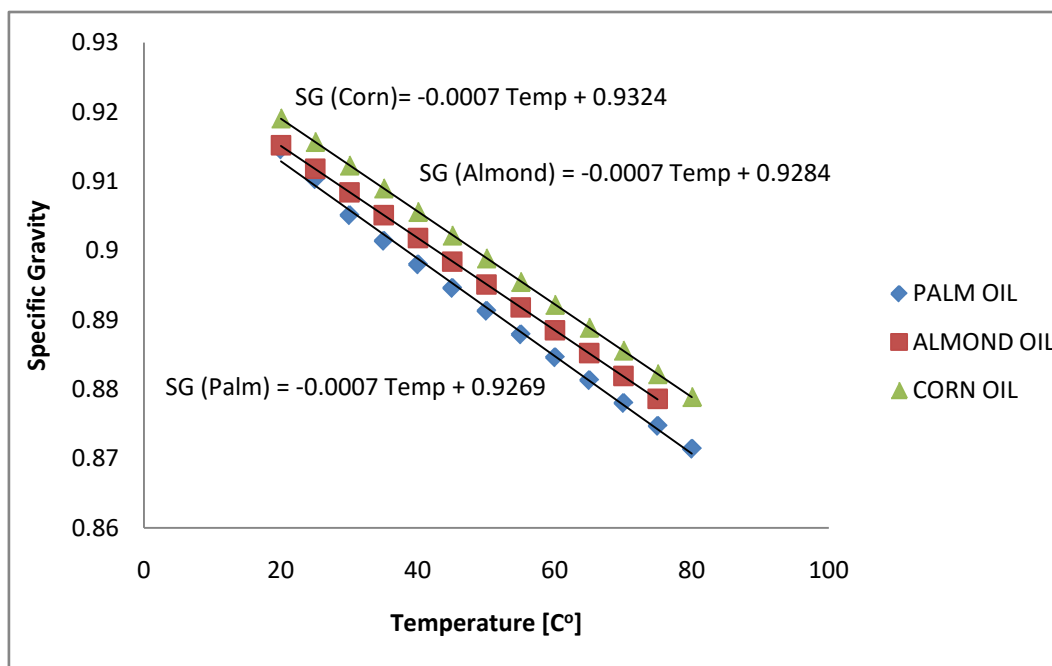


Figure (4): Linear Regression of Bio Fuel (oils) Specific gravity vs. temperature

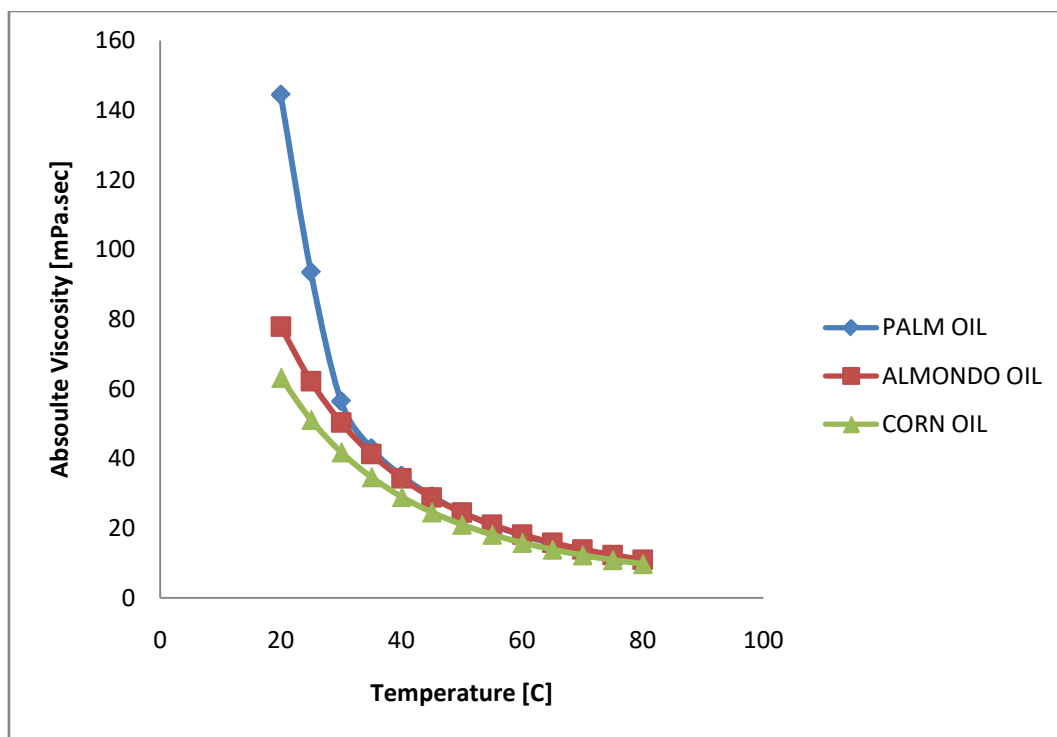


Figure (5): Bio Fuel (oils) absolute viscosity vs. temperature

The oil and new engine oil viscosity is mathematically simulated in sixth order polynomial using Excel software, the mathematical equation is in the form:

$$\mu = a_0 + a_1T + a_2T^2 + a_3T^3 + a_4T^4 + a_5T^5 + a_6T^6$$

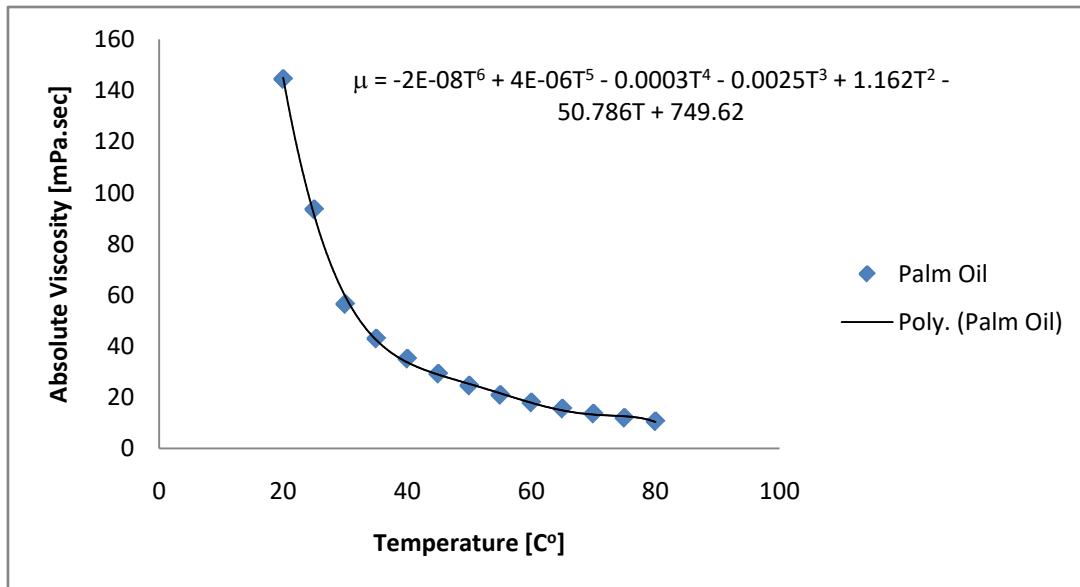


Figure (6): Palm oil absolute viscosity vs. temperature

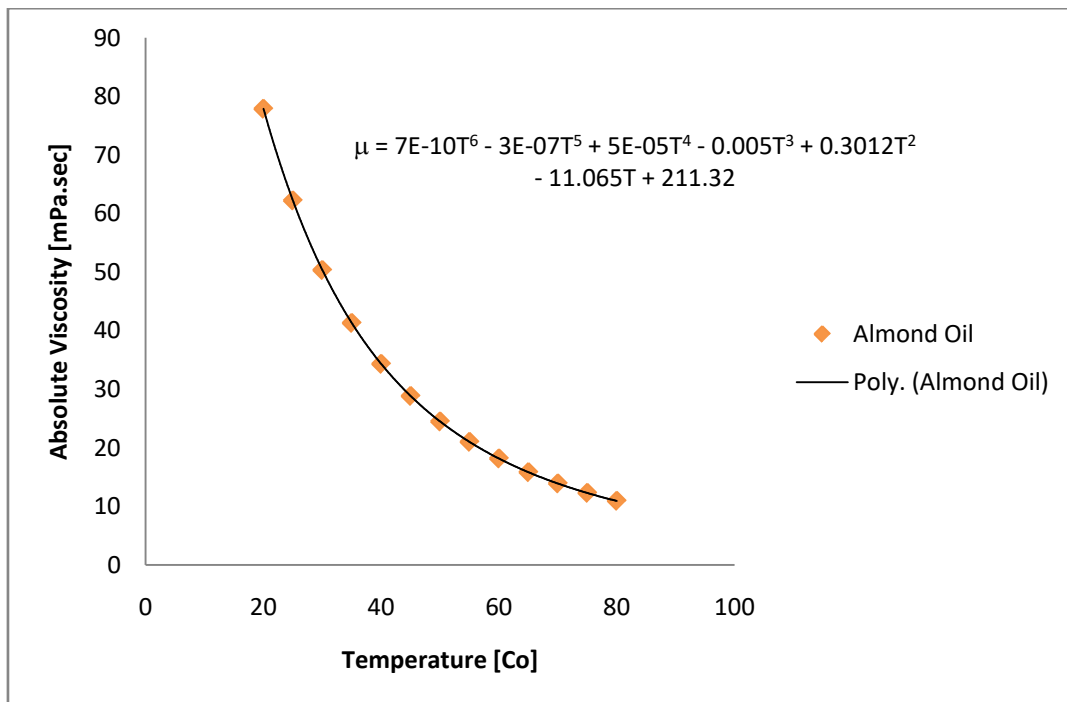


Figure (7): Almond oil absolute viscosity vs. temperature

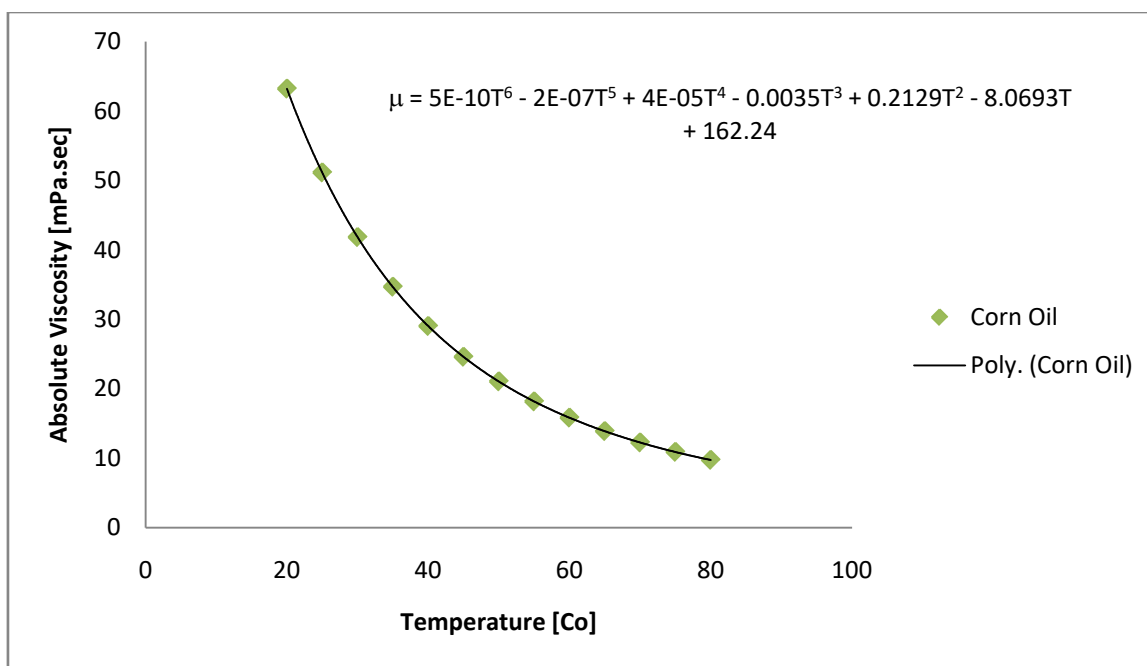


Figure (8): Corn oil absolute viscosity vs. temperature

Figure (6) shows mathematical modelling of Palm oil with temperature, the polynomial of absolute viscosity is of order six, also figures (7) and (8) show mathematical model of Corn and Almond oil, the coefficients of all oil polynomials are obtained.

IV. CONCLUSIONS

Different vegetable oils were tested using SVM3000 viscosity machine, this paper aims to create viscosity modelling of these vegetable oils. The tested oils are Palm, Almond, and Corn, the experimental results obtained are oil absolute viscosity, kinematic viscosity, and specific gravity. A sixth order polynomial for absolute viscosity was obtained to show the oil mathematical model, also a linear equation is created using Excel software for the tested vegetable oils specific gravity with temperature.

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Synthesis of Polymers by a Solar Photocatalytic Microreactor System

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Synthesis of Polymers by a Solar Photocatalytic Microreactor System

Mohammad F. Abid^α, Raid N. Howyeen^σ, Hiba M. Abdulla^ρ, Mohamed H. Hilal^ω, Ahmed S. Hamod^χ & Abeer Sameer^ξ

Abstract- The present work aimed to study the feasibility of microreaction process for polyaniline synthesis under solar light. A chemical method to prepare polyaniline is presented in which a continuous operated solar photocatalytic microreactor is utilized to promote the polymerization of aniline. The photopolymerization method yields a polymeric material that has been scanned by electron microscopy (SEM). Results showed that the initial operating temperature has a positive impact on the rate of polymerization but affects negatively the yield of polyaniline. The yielded of polyaniline is decreased as LHSV of the microreactor increased. An optimum (oxidant/aniline) molar ratio of about 1.25 is found to give the maximum yield of polyaniline. Comparison between microreactor's performance and performance of previous published works were presented. Results of the present study confirmed the feasibility of the solar microreaction process for synthesis of polymeric materials.

Keywords: microreaction; solar irradiation; continuous process; photocatalysis; polymerization.

1. INTRODUCTION

Polyaniline (PANI) has been considered a valuable electric-conductive polymer due to its superior characteristics of conductivity, stability, different structures, feasible cost and other excellent performance (Benabdellah *et al.*, 2011). PANI has been applied in many industrial fields such as LED, electrode manufacturing, isomerization of electro-materials, electrodes of storage batteries, enzyme fixation and etc. Langer *et al.* (2001). PANI has been synthesized by different methods such as chemical, co-mixing of solution, and co-blending by agitation (Malinauskas, 2004). The synthesis of PANI using ammonium persulfate (APS) as oxidizing agent was demonstrated by (Stejskal, 2002). Silver nitrate (AgNO_3) was used as oxidizing agent for aniline polymerization in the aqueous solution of HNO_3 by (Blinova *et al.*, 2009). The effect of light adsorption on the macroscopic structure of polymer was investigated theoretically and experimentally by (Daset *et al.*, 2005). They concluded that polymerization of aniline is induced by light of suitable wavelength. A minimization in the time of aniline

polymerization under UV-irradiation was reported by (Gustavo *et al.*, 2010). Most the production processes of polyaniline are using batch mode (Kulkarni *et al.*, 2015). (Kulkarni *et al.* (2015) have used a spiral microreactor for the continuous production of polyaniline nanoparticles of size of 245.3 nm from a mixture of 0.04 M concentration of aniline and 0.05 M concentration of AgNO_3 as oxidizing agent. They reported that AgNO_3 acts as a mild oxidizing agent and it was found to be more effective for obtaining nanosize of PANI particles. The effects of neutron and gamma radiations on the electrical, optical, chemical and structural properties of PANI for the same batch were studied by (Sonkawade *et al.*, 2010). They reported that PANI samples showing increase in conductivity after radiation. They observed the formation of new bands after irradiation which may be due to the cross-linking of polymer chain after irradiation.

Recently, microreaction process has been evolved as a novel technology which has been utilized to establish new concept for design, trouble shooting, and control in many fields of industry (Šalić *et al.* 2012, Rebrevet *et al.*, 2003; Kusakabe, 2002). It is well-known that countries of the Arabian Gulf have received solar incident energy for almost 300 day per year. The measured average incident UV-energy is 45 W.m^{-2} – 64 W.m^{-2} for class UV-A, and for class UV-B, the average VU-solar energy is 0.19 W.m^{-2} - 0.33 W.m^{-2} (ISERC, 2012). This makes it promising to use the UV-solar energy for many environmental and industrial applications such as wastewater treatment and synthesis of polymeric materials. Solar irradiation for desulfurization of gas oil in a Y-shaped catalytic microreactor was utilized by (Abid, 2015). He investigated the effect of different operating variables (e.g., concentration of dibenzothiophene, liquid hour space velocity, initial feed temperature, and hydrogen peroxide: dibenzothiophene ratio) on the reactor performance. He reported that the percentage removal of sulfur in the Y-shaped microreactor was 65% in approximately 9 (min) comparing to 340-400 (min) in a batch reactor of macroscopic scale used for desulfurization operation of gas oil.

Aniline in 1 M HCL solution could be polymerized using (APS) as oxidant through two steps. The first is the oxidation to form PANI (emeraldine) hydrochloride. The second step is the deprotonation in

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ammonium hydroxide solution to yield PANI -emeraldine base (Faris, 2007; Sapurina and Stejskal, 2010). An overview on formation of image with PANI by photo-induced reaction was presented by (Kobayashi *et al.*, 1998). They proposed that photogenerated holes on the irradiated surface of the sensitizer could oxidize the aniline derivatives initiate polymerization while the metal complexes reduced by the photogenerated electrons and precipitated on part of the sensitizer which needed to regenerate (flushed) after periods of operating time. The fractional degradation (x) of aniline was calculated according to equation 1. The percentage yield of PANI was calculated on the basis of the unreacted aniline and estimated according to equation 2:

$$\text{Aniline conversion } (x) = \frac{C_0 - C(t)}{C_0} \quad (1)$$

Where C_0 and $C(t)$ are the initial and instantaneous concentration of aniline in solution, respectively.

$$\% \text{ PANI yield} = \frac{\text{weight of PANI produced}}{\text{Initial weight of aniline}} \times 100 \quad (2)$$

The aim of the present study was to design, fabrication and application of a photocatalytic microreactor for a continuous operation of PANI synthesis under incident solar energy. Another objective was to investigate the influence of the operating variables (e.g., APS/aniline molar ratio, inlet mixture flow rate, and initial feed temperature) on the morphology of the produced aniline and also on performance of the microreactor.

II. MATERIALS AND METHODS

a) Materials

Chemicals used in the present study are: TiO_2 (80% (w/w) anatasa, average particle size 20nm, and

specific surface area $60 \pm 10 \text{ m}^2/\text{g}$) was supplied from Xinyue Chemical Co., China and used as a sensitizer. Aniline (99% w/w) was obtained from Thomas Baker Chemicals. Ammonium peroxydisulfate ($(\text{NH}_4)_2\text{S}_2\text{O}_8$, 98% w/w) was purchased from Fluke Chemical Co. Cobalt (II) chloride-6-water ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$) was obtained from Merk and used as an electron acceptor. Hydrochloric acid (36% w/w) and caustic soda (Flakes) were obtained from Bayer. Water used was obtained from the RO plant.

b) Methods

i. Analytical methods

UV-Visible spectrophotometer type Shimadzu (UB-1201 PC) was used to record the UV-Vis spectrum of aniline in HCl. Scanning electron microscope (SEM) (Model: Inspect 50S, supplied by FEI-USA) was used to test the deposition process of photo-catalyst nanoparticles on the microchannel and also to examine the surface morphology of the polyaniline produced. To construct a calibration curve of aniline concentration vs. light absorbance, using UV-Vis spectrophotometer, different amounts of aniline (0.0027, 0.005, 0.0107, 0.032, 0.054, 0.086 and 0.107 mmole) is dissolved in 10 ml of 1 M HCl, respectively. Figure 1 illustrates the obtained calibration curve of aniline in HCl.

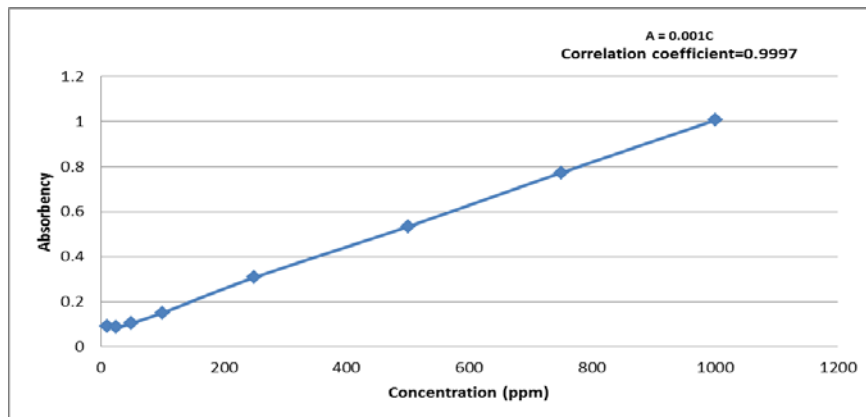


Figure 1: Calibration curve of aniline in HCl

III. MICROREACTOR DESIGN AND FABRICATION

Diffusion concepts were used to correlate the microreactor's dimensions with the time scales of mass and heat transports according to the following equations (Wegenet *et al.*, 1996; Branebjerget *et al.*, 2005):

$$\text{Transport of heat:} \quad \tau \sim \frac{L}{\gamma} \sim \frac{L}{u} \quad (3)$$

$$\text{Transport of mass:} \quad \tau \sim \frac{L^2}{D_m} \sim \frac{L}{u} \quad (4)$$

$$\frac{L^2}{\gamma \tau} = \frac{L^2 \cdot u}{\gamma \cdot L} \sim 1 \quad (5)$$

$$\frac{L^2}{D_m \cdot \tau} \sim = \frac{L^2 \cdot u}{D_m \cdot l} \sim 1 \quad (6)$$

where

l : length of travelling, τ : time, L : diffusion distance, γ : thermal diffusivity of liquid, D_m : mass diffusivity, U : fluid speed

The design steps of the microreactor are found elsewhere in (Abid, 2015; Al-Raie, 2005; Wang et al.,

2013; Yong and Sangmo, 2003). Figure 2 shows the dimensions of the reactor pattern as designed and schemed using 2D AUTOCAD software. CNC milling machine type C-tek, at the workshops of the University of Technology (UOT), was used to fabricate the microreactor which consisted of two parts, the upper one was made of a transparent elastic polymer and the lower one was made of aluminum alloy.

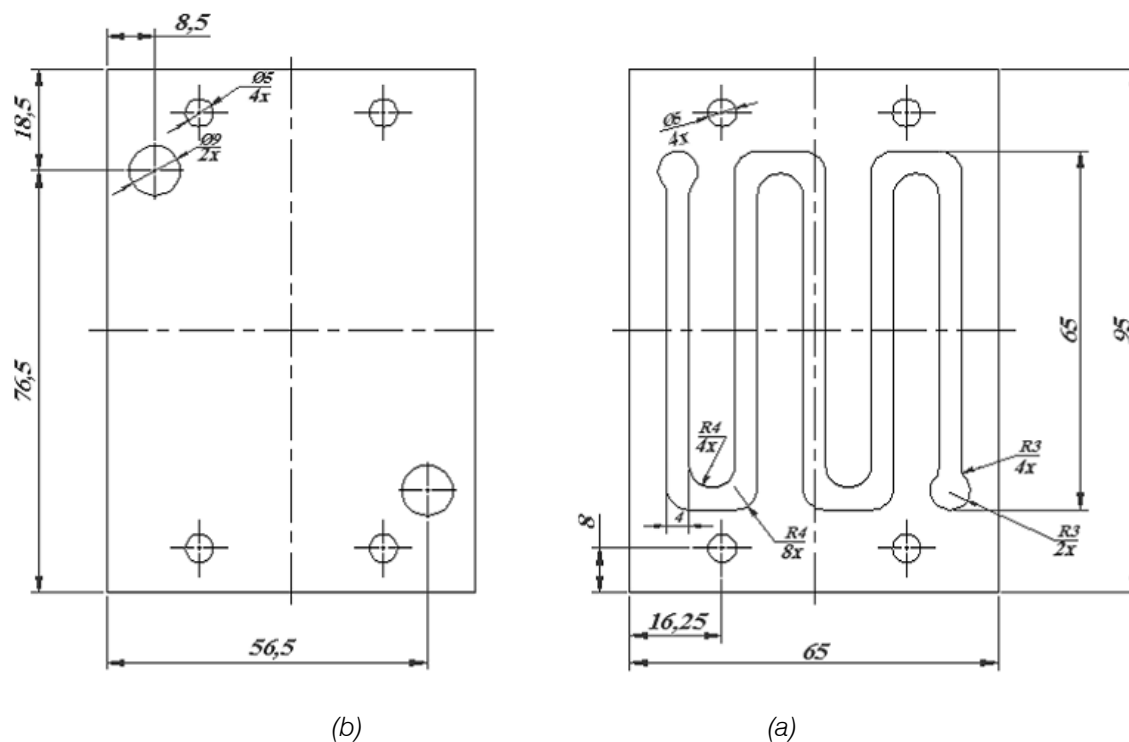


Figure 2: 2D AUTOCAD schematic of the microreactor, (a) lower part and (b) upper part (all dimensions are in mm)

a) Catalyst

To enhance the polymerization reaction, TiO_2 particles were deposited onto the microchannel of the lower part of microreactor as follows (Abid, 2015):

1. The microchannel was first washed with a 5 wt% of caustic soda solution.
2. In a 25 ml beaker, half gram of TiO_2 catalyst was added to a mixture of 5 mL (5 % w/w HNO_3) and 10 mL of 50% (w/w) methanol. The mixture was intensively homogenized with a magnetic stirrer at 400 rpm.
3. After 30 minutes of agitation, a syringe was used to draw a 5 ml of the well-dispersed mixture and injected it into the microchannel. The microchannel which was filled with suspension allowed drying at 120°C for two hours into an electric oven.
4. The deposition process could be carried several times so as to avoid naked batches. Microchannel was scanned using SEM (model Inspect S50, S/N 9922650, FEI Company, USA). Scanning procedure was carried out at the Department of Applied Sciences, UOT.

b) Experimental setup

Figure 3a and Figure 3b show a schematic and an image of the experimental setup, respectively. The experiments were carried out under solar light and being repeated under indirect solar irradiation conditions by installing the experimental setup under shadow. The operating conditions (i.e., APS: aniline molar ratio, inlet mixture flow rate, and initial feed temperature) are kept the same so as to obtain a fair comparison between the two cases. 200 mL of a mixture of aniline in HCl was contained in a 250 mL graduated glass bottle connected to a micropump (model 200.015.230.016 by Williamson Co., England) via a regulating valve. Another 250 mL glass graduated bottle contained 200 mL of APS in HCl with 0.2 g of $(\text{CoCl}_2 \cdot 6\text{H}_2\text{O})$ was connected to a second micropump. Temperature of the two bottles was kept constant by a temperature-controlling water bath. The mixtures into the two bottles were kept in good homogeneity by placing the water bath on a magnetic stirrer as shown in Figure 3a. The suction valves were calibrated against the level in each container, so different flow rates could be delivered to the

microreactor separately in each run. The feed reactants fed to the microreactor via the two micropumps. The initial feed temperature was measured by two mercury thermometers immersed into the bottles. Microreactor effluent was collected in a graduated 500 mL container. After passing through the microreactor, the solution was collected in a product vial containing 1 M NH_4OH for converting polyaniline hydrochloride to a polyaniline

base. Reaction mixture was filtered under vacuum and washed with ethanol and then dried at 60 °C for 6 h to obtain PANI emeraldine salt (ES) as a green powder. All containers and tubing outside the microreactor were shielded from UV-exposure. Flowrates of 0.75, 1, 1.5, and 2 $\text{L}\cdot\text{min}^{-1}$ were used which correlate to 6.9, 5.2, 3.46, and 2.6 min residence times, respectively. Each run was repeated at different molar ratios of (APS: aniline).

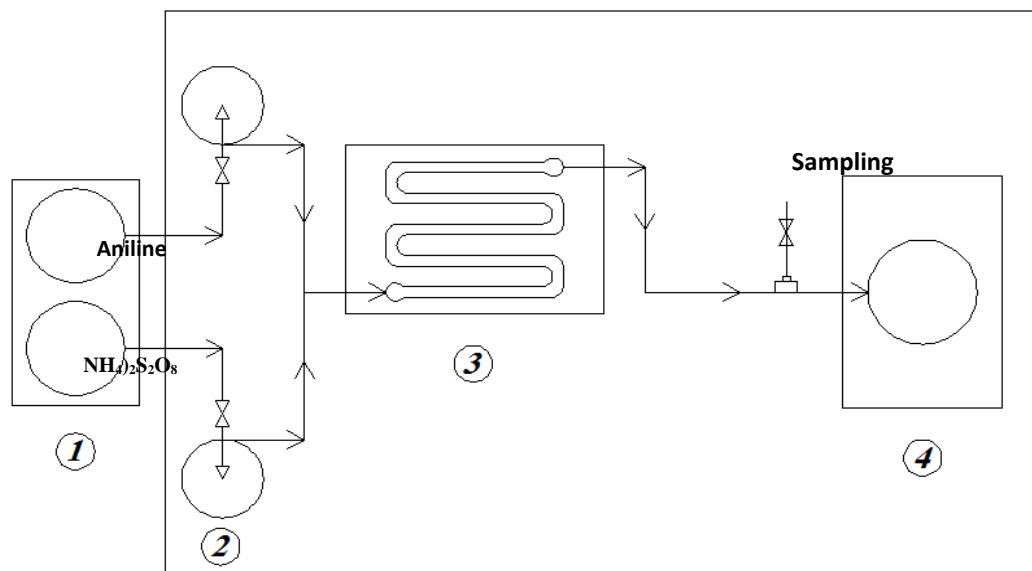


Figure 3 a: Schematic diagram of the experimental setup

1: Magnetic stirrer with temperature-controlling bath; 2: Micropumps; 3: Microreactor; 4: Product collector

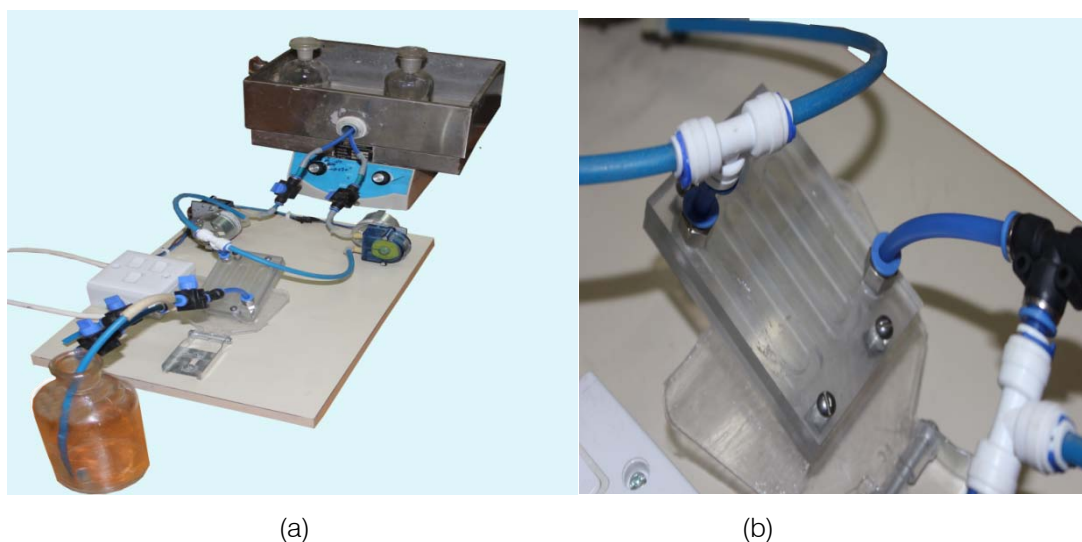


Figure 3 b: Images of the experimental setup (a) and microreactor (b)

IV. EXPERIMENTAL DESIGN

The experimental runs were designed using the factorial method which has a high reliability. Real values of the controlled variables (F) such as (initial feed temperature, mixture flow rate, and molar ratio of APS: aniline) and their corresponding levels (Le) are shown in

Table (1). Each experiment was repeated twice to get accurate results.

Table 1: Selected levels and factors

F Le	Real variables		
	Initial feed temperature [°C]	Mixture flow rate [ml. min ⁻¹]	(APS: aniline) [molar ratio]
1	25	0.75	0.8
2	5	1.00	1.0
3		1.5	1.2
4		2	1.4

V. RESULTS AND DISCUSSION

a) Catalyst deposition

As can be seen in Figure 4 (a and b), the third coating with TiO₂ nanoparticles onto the microchannel lead to complete coverage. The total thickness of

coating was approximately calculated by estimating the actual weight of TiO₂ nanoparticles used for coating, the area covered, and the known bulk density of dried nanoparticles. The total thickness was approximately estimated to be 6 μm.

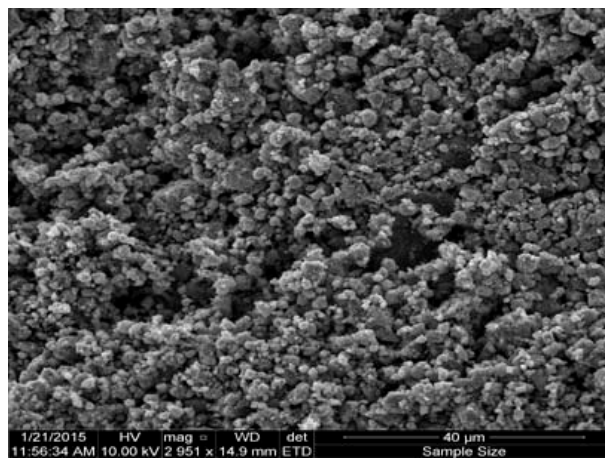
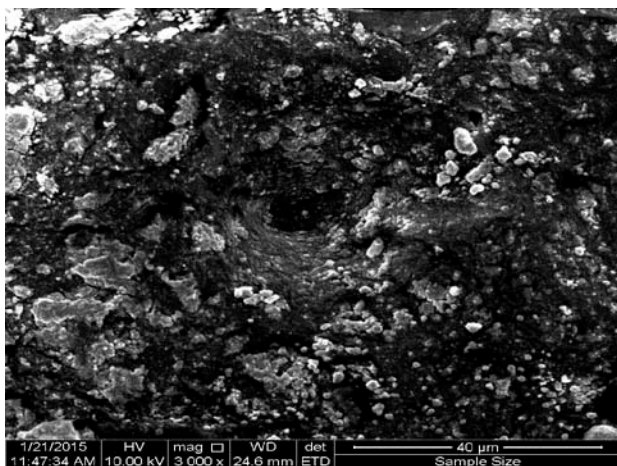


Figure 4: SEM images of different coatings of nanoTiO₂, after 1st coating (left) and after 3rd coating (right)

b) Morphological Analysis

Figure (5) shows the morphology of PANI powder synthesized under indirect solar light at 25°C and under direct solar light at 5 and 25 °C, respectively. As can be seen in Figure (5a) a surface of porous structure composed mainly of granular sharp edges in size ~ 15 μm. Figure (5b) shows a granular sharp edge surface but with less porosity. This may be attributed to the effect of solar irradiant which indicated that the morphology of the conducting polymer is strongly dependent on the irradiated excitation wavelength (Sonkawade, 2010; de Barros *et al.* 2003). Micrograph of Figure (5c) shows an agglomeration of nano-crystals in size ~ 2 to 4 μm. This agglomeration may be increased as the operating temperature is reduced further (Faris, 2007). The SEM micrographs of Figures (5b) and (5c) confirmed that the morphology of PANI depends on the solar incident energy and initial feed

temperature used for the preparation. The data reported by (Faris, 2007; Mattoso *et al.*, 1994) indicated that decreasing of synthesis temperature may give a polymer of shorter chains with higher molecular weight.

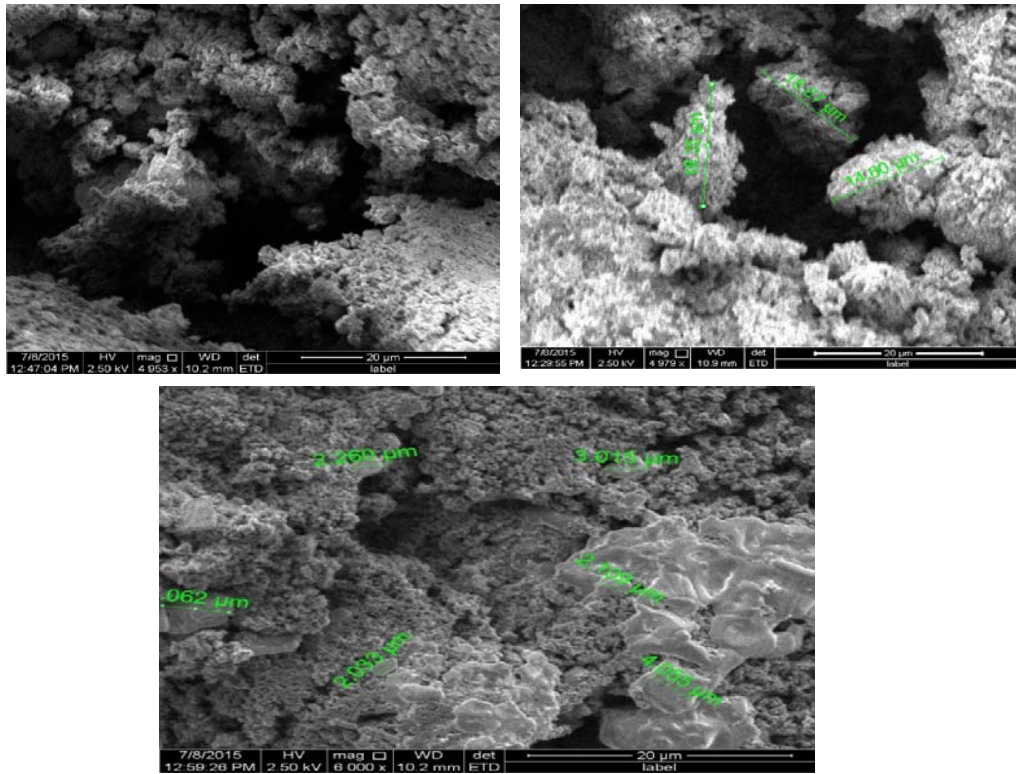


Figure 5: SEM micrographs of produced samples (a) Upper left: indirect solar light at 25 °C; (b) Upper right: direct solar light at 25 °C; (c) down middle: direct solar light at 5 °C

c) Influence of solar incident energy on polymerization process

Figure (6) plots the effect of solar irradiant intensity on normalized aniline concentration at different reaction time at (oxidant-to aniline molar ratio ~ 1.25 and $LHSV = 0.145 \text{ min}^{-1}$). As can be seen in Fig.6, the photocatalytic process affects negatively the time span of polymerization and positively impacted the yield of PANI. Table 2 list the influence of solar incident energy

and initial feed temperature on % yield of the microreactor at 11.5 min of polymerization time. It could be observed that solar incident irradiation enhanced the average yield by 5% over that of indirect solar while the average time of polymerization is reduced by 17%. The polymerization time was reduced by 20 to 27% when increasing temperature from 5 to 25 °C under indirect solar and direct solar, respectively.

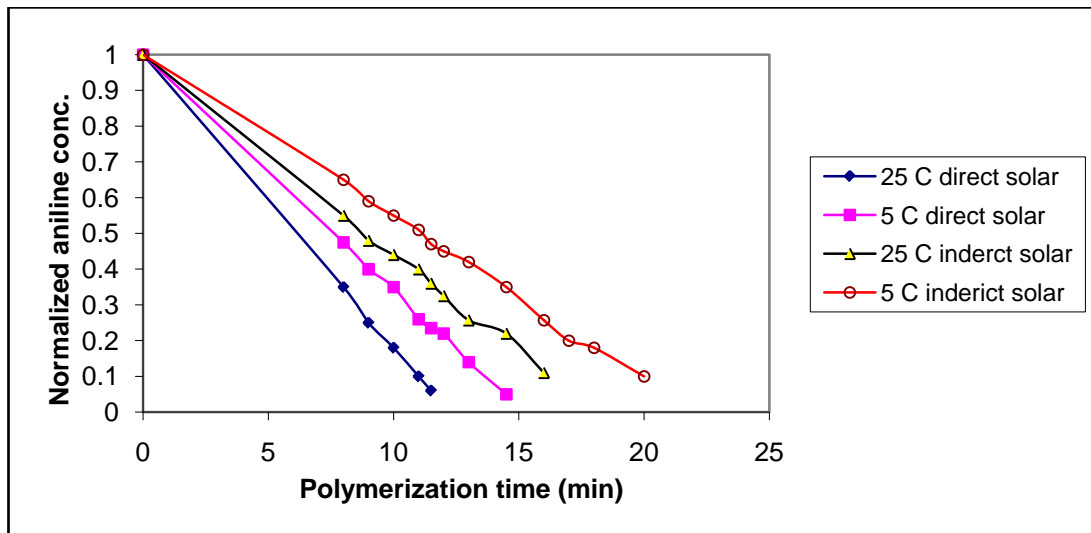


Figure 6: Effect of solar intensity on normalized aniline concentration at different reaction time at (molar ratio of APS: aniline=1.25, and $LHSV = 0.145 \text{ min}^{-1}$)

Table 2: Effect of solar energy and initial feed temperature on yield at 11.5 min of polymerization time

	Direct solar		Indirect solar	
Temperature [°C]	5	25	5	25
yield [%]	76.5	94	53	64

d) *Influence of LHSV and Initial feed temperature*

The effect of LHSV and initial feed temperature on yield of polyaniline synthesized under solar light is illustrated in Figure 7. As can be observed, yield is reduced as LHSV is increased while initial feed temperature shows a different image. The explanation for these trends is that increasing LHSV resulted in decreasing retention time of reactant in the photocatalytic microreactor, consequently the rate of oxidation within the reactor is reduced. The yield of

polyaniline is reduced from 94.0 to 84.2% as LHSV is increased from 0.145 to 0.385 min^{-1} at temperature of 25 °C. The influence of initial feed temperature on polyaniline yield could be also seen in Figure 7. The yield increased from 94.0 to 95.1% as the temperature is reduced from 25 to 5 °C at LHSV = 0.145 min^{-1} . As can be observed, at lower temperature, the increase of polyaniline yield was predominant. This could be due to the molecular chains which have less structural defects at lower temperature.

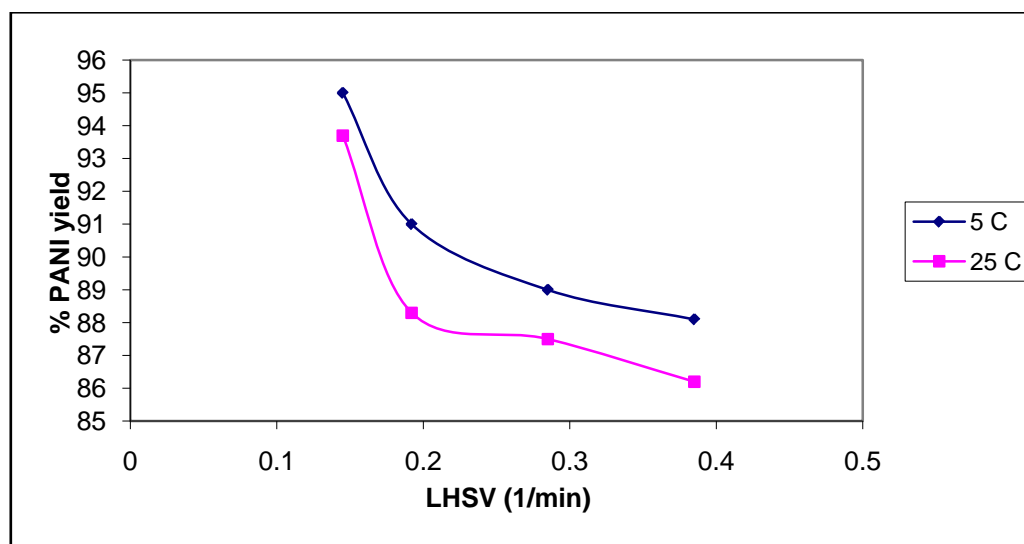


Figure 7: Variation of polyaniline yield against LHSV at different initial feed temperature and APS-to-aniline molar ratio = 1.25

e) *Influence of (APS: aniline) molar ratio on yield*

Figure 8 shows the influence of (APS: aniline) molar ratio on polyaniline yield. As can be seen when the APS: aniline ratio gradually increased, the yield of PANI increases correspondingly keeping other operating parameters unchanged. The yield reaches maximum value (i.e., 95.1%) after then further increase in molar ratio resulted a gradual decreasing of yield. It is expected that all the aniline which consumed by thereaction at the ratio higher than 1.25 would not increasethe yield but may cause its decrease because the over- oxidationmay convert part of the PANI to quinone. Furthermore, color of the filtrate solution was changed from green-blue to purple when the APS: aniline ratio was more than 1.25: 1. This could be an indication that chain of polyaniline begin to break up. These results agree well with the findings of (Natalia *et al.*, 2007; Adams *et al.*, 1996).

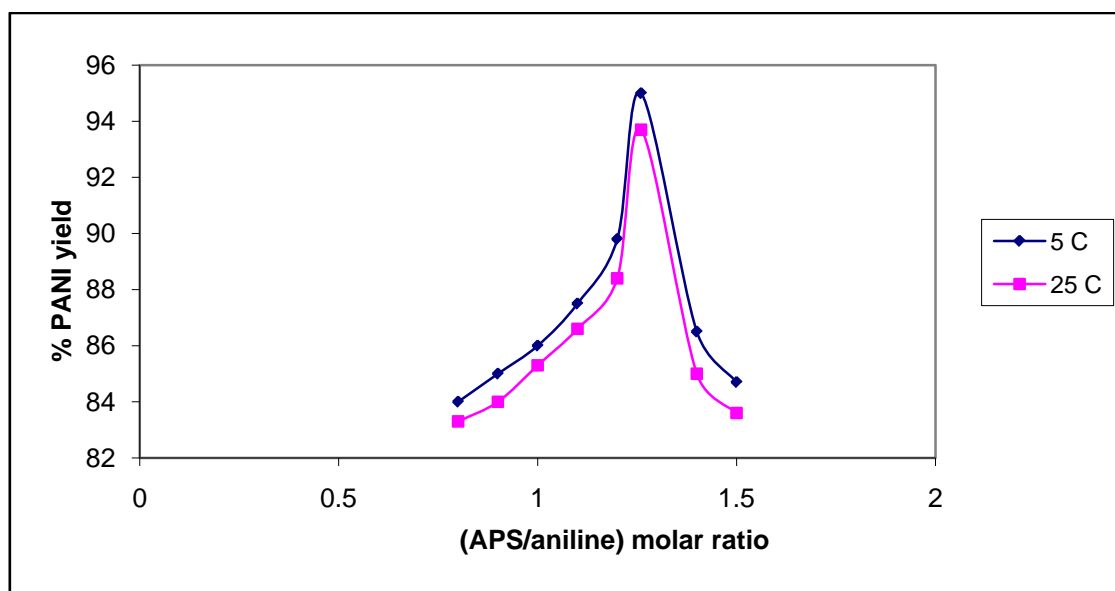


Figure 8: Influence of (APS: aniline) molar ratio on the yield of polyaniline at different initial operating temperature and LHSV = 0.145 min⁻¹

f) Comparison of performance between present study and previous published works

Comparison of performance between present study and previous published works of (Faris, 2007; Adams *et al.*, 1996; Rodolfo *et al.*, 2005; Gordana *et al.*, 2006) is presented. Different operating temperatures to synthesis chemically polyaniline with APS at equal molar ratio of (aniline: oxidant) and 1 mol/L hydrochloric acid in a stirred batch reactor was used by (Faris, 2007). He reported yields of (86.4, 89, and 90.4%) at operating temperatures of (25, 0, and -25 °C), respectively. The Synthesis of polyaniline (PANI) catalyzed by soybean peroxidase (SBP) at 1°C in either aqueous or partially organic media in a batch stirred reactor was studied by (Rodolfo *et al.*, 2005). They claimed a yield of 71% at the optimum conditions of their experimental run. Gordana *et al.* (2006) investigated the kinetics of aniline polymerization in acid solution with ferric chloride as oxidant. They performed the polymerization in a special electro-cell batch reactor at 25 °C. They reported a

maximum yield of 50%. The oxidative chemical polymerization of aniline in hydrochloric acid solution at low temperature was studied by (Rodolfo *et al.*, 2005). They reported that among the reaction parameters studied which affected the molecular weight and yield of the polymer were: (a) reaction temperature; (b) solution pH at the start of the reaction; (c) molar ratio of oxidant: aniline; (d) total reaction time. They concluded that polyaniline of high molecular weight may be obtained by carrying out the polymerization at low temperatures, typically between - 25 and - 30 °C which required the use of an inert solute (lithium chloride was the preferred choice) to keep the aqueous reaction medium mobile. They reported that molecular weight (daltons) and yield% of synthesized PANI were (133000±10000, 94.2%), (153000±9000, 97.5%), (138000±7000, 94.9%) at reaction temperature -20, -25, -30 respectively and reaction time of approximately 15 h. Table 3 summarizes the results obtained by different authors.

Table 3: Comparison of performance between present study and previous published works

Substrate	Process	Type of reactor	Retention time, [min]	Yield [%]	reference
Aniline	Chemical (APS)	batch	120 (0 °C)	89	Faris (2007)
	Enzymatic (SBP) + H ₂ O ₂	batch	480 (1 °C)	71	Rodolfo <i>et al.</i> , 2005
	Electrochemical (FeCl ₃)	Batch electro-cell	180 (25 °C)	50	Gordana <i>et al.</i> , 2006
	Chemical (APS)	batch	~900 (-20, -25, -30 °C)	94.2, 97.5, 94.9 (respectively)	Adams <i>et al.</i> , 1996
	Solar photocatalysis (APS)	S-shaped microreactor	14.5 (5 °C)	95	Present work

V. CONCLUSION

The present work aimed to study the feasibility of microreaction process for PANI synthesis under solar light. The chemical polymerization of aniline in aqueous HCl solution with APS as the oxidant was investigated in a continuous operated solar photocatalytic microreactor with TiO₂ nanoparticles as photocatalyst. The performance of the system was studied at different operating conditions (e.g. initial feed temperature, LHSV, and (APS/aniline) molar ratio). Scanning electron microscopy (SEM) was used to test the surface characteristics of the polymeric material produced. It was shown that surface morphology of PANI samples affected by solar incident energy characterized by reduced surface porosity. This could improve the conductivity of the polymer as confirmed by other published data. Results showed that initial temperature has positively affected the rate of polymerization but it affects negatively the yield of polyaniline, yield of polyaniline was decreased as LHSV of the microreactor increased, and an optimum (APS: aniline) molar ratio of 1.25 was found to give the maximum yield of polyaniline. Results indicated that solar incident irradiation enhanced the average yield by 5% over that of indirect solar while the average time of polymerization was reduced by 17%. The polymerization time was reduced by 20 to 27% when increasing temperature from 5 to 25 °C under indirect solar and direct solar, respectively. Data of batch reactors from published literature were used for performance comparison with the present work. Results showed that the solar microreaction system offered an excellent result (i.e., a yield of 95% under solar light at 5 °C within 14.5 min.) compared with other published data presented in this study. Results of the present work confirmed the feasibility of the microreaction process for aniline polymerization under solar irradiation.

VI. ACKNOWLEDEMENT

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Keywords: *alizarin red S, norit GCA830, fixed bed, adsorption capacity.*

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Dynamic Sorption of Alizarin Red S by Fixed Bed Activated Carbon

Ali Benhmidene ^α, Khaoula Hidouri ^σ, Hedi Ben Amor ^ρ & Bechir Chaouachi ^ω

Abstract- Dynamic removal of Alizarin Red S (RAS) by activated carbon Norit GCA830 has been experimentally studied in fixed bed column. We can predicted the value of column parameters as a function of inlet solution dye concentration, flow rate and bed height. A static study has been first conducted, from which maximum adsorption capacity is defined; it's of 385mg/g. Both Freundlich and Langmuir models were found to fit the sorption isotherm data well. For dynamic experimental study; series of column tests using activated carbon were performed to determine the breakthrough curves with varying the bed height, inlet solution dye concentration and flow rate. Adsorption capacity in fixed bed is defined from breakthrough curves is similar of that defined in static study. In addition adsorption capacity of fixed bed is correlated in function of operating conditions cited above. The kinetic model "Bed Depth Service Time" (BDST) is another method used to defined the adsorption capacity of AC in fixed bed in addition of other kinetic parameters such as constant kinetic, the thickness of mass transfer zone and the velocity of migration of adsorption zone. According to operating conditions the adsorption capacity is mostly depend of residence time.

Keywords: alizarin red S, norit GCA830, fixed bed, adsorption capacity.

I. INTRODUCTION

Due to the rapid urbanization and rapid growth of industrialization, large quantities of waste containing pigments and dyes are discharged into the receiving aquatic environment. It is expected that the dyeing industry is responsible for the release of 100 tons of dyes per year into the environment, contaminating rivers and springs [1]. Of these dyestuffs, 5–10% is lost in industrial effluents, and consequently, wastewater treatment is one of the biggest problems we face today [2]. In fact, due to ineffective treatment processes, the presence of these dyes will have a negative impact on environmental life in general [3]. Knowing that synthetic dyes are not biodegradable and toxic in nature; their presence in the aquatic ecosystem hinders the growth of biota and affects the food web [4]. In addition, long term human exposure to dye pollutants increases the risk of tumours, cancers, cerebrovascular and lung diseases [5].

Nowadays, several industrial sectors use dyes in their product such as leather, textiles and food processing industries, there is a primary concern of

decolorization and treatment of wastewater [6]. In addition to adsorption which has been widely used, there are various methods of physical and chemical treatment for organic dyes. The most important treatment processes are coagulation and flocculation [7], photodegradation, biosorption, oxidizing agents, membrane and ultrafiltration. The advantages, disadvantages and limitations of each technique have been widely studied by many researchers [8-10]. However, those technologies are costly, less effective and lack adaptability to a wide range of dyes present in the wastewaters. As a technique obeyed by environmental rules, capable of eliminating a wide range of contaminants even at very low concentrations [11], the adsorption processes presents the most preferable method for the purification of water from synthetic dyes [12, 13].

The adsorption studies are generally divided into two parts. The first is the batch adsorption. It has the advantage to provide useful data and parameters on the adsorbent and the adsorbent-adsorbent mass transfer mechanism such as the maximum adsorption capacities and the kinetic constants as well as the type of the isotherm inducing the type of kinetics controlling the adsorption process [14, 15]. The second technique is that of continuous adsorption, for which the effluent to be treated flows over a bed of active charcoal or other adsorbent placed in a fixed bed. This process is also necessary for access to practical operational information [16-18]. During the adsorption on a fixed bed, the columns can be placed in series or in parallel. In this area, small column studies are started to simulate the potential performances of adsorption, and then the results obtained will be extrapolated in large scale columns or reactors [19,20].

In addition to the kinetic parameters, the adsorption capacity of activated carbon in a fixed bed is of great practical importance because it makes it possible to evaluate the efficiency of the fixed bed with respect to the elimination of the contaminants [21]. This was the subject of several studies. Different methods have been used to access this parameter. We distinguish among others, those who use the curve of breakthrough [22,23]. In addition the BDST model and the Clarks model are frequently used to access in this parameter [14].

In the present work, we have carried out an experimental study for testing the ability of Norit

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activated carbon to remove the Alizarin Red S as an example of anionic dye. Our study is realised for static regime in Batch reactor. However series of columns is used in dynamic regime. The adsorption capacity of activated carbon in static and dynamic is calculated by using different methods for different operating conditions.

II. MATERIAL AND METHOD

a) The Adsorbent

In present study we used activated carbon NORIT GCA830; the main characteristics are extracted from Norit Digital Library is shown in Table 1 [24].

Table 1: main characteristics of NORIT GCA830 activated carbon

Specific surface (m ² /g)	1000
Molasses Index	210 min
Iodine number (mg/g)	920 min
Moisture conditioning (%)	2 max
Mesh size(%) :	
- Greaterthan 8mesh (2.36mm)	8 max
- Lessthan 30 mesh (0.6mm)	4 max
Abrasion number (AWWA)	75 min
Apparent density (mg/l)	0.51
Effective size (mm)	0.86
Density after backwash and drainage (g.cm ⁻³)	0.45

b) The Adsorbate

Our study focused on adsorption of Alizarin Red S dye by NORIT GCA 830 in batch and fixed bed reactor. Alizarin Red S (sodium alizarin sulphonate, CI 58005) is an anthraquinone.

Figure 1 shows its chemical structure molecule:

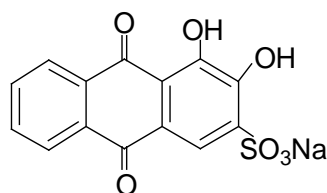


Fig. 1: Chemical structure molecule of RAS

The mains property of RAS is resumed in Table 2.

Table 2: Characteristic of RAS

Number (CI)	58005
Class	anthraquinone
Molar Wight (g)	342.267
Ionisation	acide
Ebullition temperature (°C)	430
Fusion temperature (°C)	287-289

c) Batch equilibrium studies

The study of the adsorption of RAS by the activated carbon NORIT GCA830 in a static condition was carried out according to the following experimental protocol:

1000 ml of RAS aqueous solution of variable initial concentration are stirred with 50 mg of NORIT GCA830 of particle size between 0.4 and 0.63 mm at ambient temperature. After a given stirring time, samples were taken and then centrifuged at 3000 rpm for 5 minutes. The supernatant is analyzed using a spectrophotometer (HATCH DR / 2000).

d) Fixed-bed adsorption experiments

For the experiments under dynamic conditions, we carried out experiments of adsorption in fixed bed by using the prototype given in Figure2:

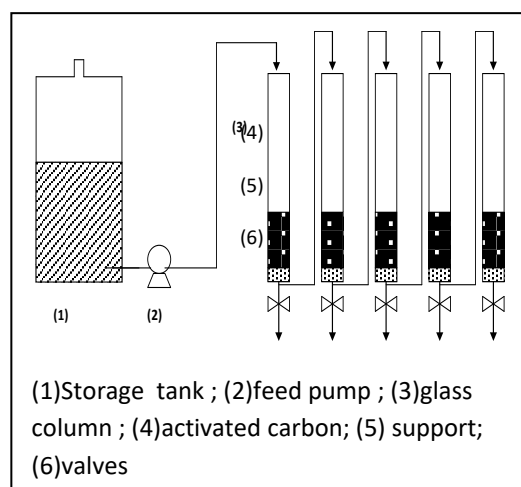


Fig. 2: Fixed bed adsorption set up

Each column of inner diameter of 1.2cm, contains a given weight of active carbon of particle size of 0.4 to 0.63 mm, corresponding to cumulative height respectively of 3.3, 5.3, 7.3, 9.3 and 11.3cm. The inlet solution dye concentration is 25 mg / l to 75 mg / l and its flow rates are 7, 15, 22.5 and 30ml/s.

III. RESULTS AND DISCUSSION

a) Batch equilibrium studies

In order to determine the adsorption parameters of RAS on NORIT GCA830, the sorption isotherms of

Langmuir and Freundlich isotherms in their linearized form were determined. We obtain right lines whose regression coefficient ($R^2 > 0.98$).

The values of Langmuir parameters such as the maximum adsorption capacity q_m and of the equilibrium constant K_l and their of Freundlich (K_F and n) are presented in Table 3.

We can be seen that the n value is between 2-10 the rung indicates a good favourability of sorption [13]. According of applicability of both Freundlich and Langmuir isotherms, we deduce that both heterogeneous surface and monolayer adsorption conditions exist under the actual studied process of sorption

Table 3: Langmuir and Freundlich parameters for RAS Adsorption

Langmuir			Freundlich		
K_l (l/mg)	q_m (mg/g)	R^2	K_F	n	R^2
0.13	384.6	0.9978	78.90	2.55	0.9835

b) Dynamic sorption

i. Breakthrough curve

To obtain these curves, we followed, as a function of the time, the evolution of the concentration of RAS effluents at the columns exit. The curves are represented for different height of the absorbent bed (Figure 3.(a)) and for three concentrations (Figure 3.(b)). The influence of flow rate on the breakthrough curve is given in Figure 3.(c).

It can be seen that, these curves are characterized by two particular points

- The breakthrough time t_b : corresponding to the leakage concentration C_b of $0.1C_0$, which it is chosen arbitrarily, as the extreme value of the RAS solution concentration at the outlet of column.
- The saturation time t_s : corresponding to the saturation of fixed bed activated carbon, where $C_s = C_0$.

According the results illustrated in above curves, the evolutions of breakthrough and saturation times are function of studied parameters. From Figure 3(b), we can be seen that the breakthrough and saturation times are all the shorter as the height of the bed is short. However they are shorter as the initial concentration and flow rates are high (see Figure 3(a) and (c)).

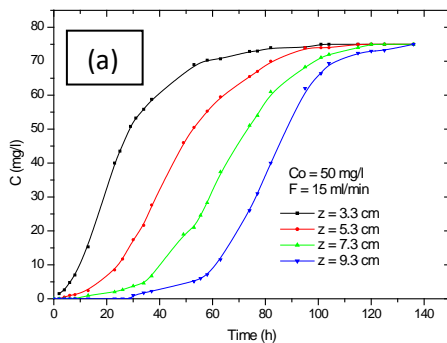
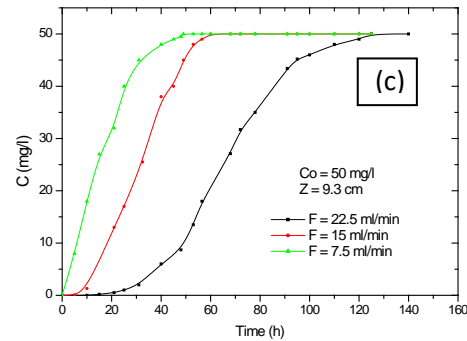
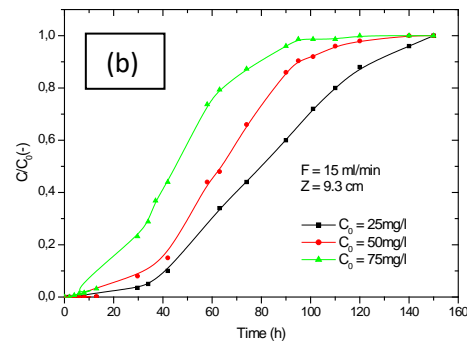


Fig. 3: Breakthrough curves for (a) different bed height, (b) different inlet concentration, (c) different flow rate

In order to understand the evolution of the breakthrough and saturation time as a function of the studied parameters, we have plotted in Figure 4 (a) and (b), those variations respectively versus bed height and the reverse of flow rate. It can be seen, the increase in t_b and t_s is directly proportional to the bed height (Z), but inversely proportional to the flow velocity. However, no relationship could be established to describe the evolution of t_b and t_s the initial concentration.

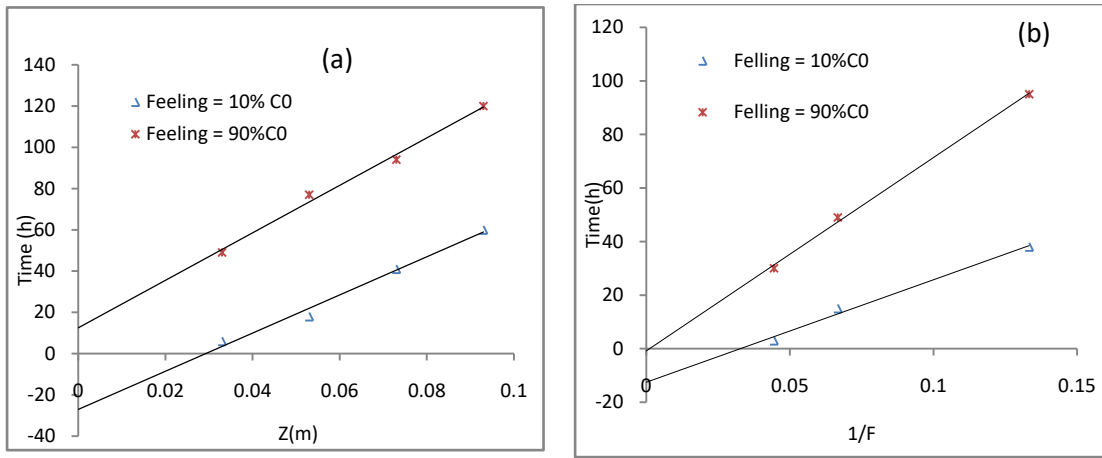


Fig. 4: Breakthrough and saturation times versus (a) bed height (b) the reverse of flow rate.

An important result can be deduced from these curves is that the time difference ($t_b - t_s$) is independent of the bed height (Figure 4 (a)). Result means that the adsorption zone height (where the concentration varies between 0 and C_0) is independent of the bed height. On the other hand, when the flow rate increases the time difference ($t_b - t_s$) decreases (Figure 4 (b)).

ii. Assessment of adsorption capacity

Based on the breakthrough curves, the amount of RAS adsorbed by activated carbon in the fixed bed is calculated by using the following equation [25] :

$$q = \frac{F}{m} \int_0^{t_s} (C_0 - C_s) dt \quad (1)$$

Where F is the volumetric flow and m is the weight of activated carbon.

The integral in Eq. (1) is equal to the area includes the ordinate axis and the breakthrough curve, where it's calculated by using the Matlab software. Values of adsorption capacity calculated from the breakthrough curves at different experimental conditions are grouped in Table 4.

Table 4: Adsorption capacity of fixed bed for different operating conditions

Bed height (m)	Flow rate (ml/min)	Inlet concentration (mg/l)	Adsorption capacity (mg/g)
0.033	7.5	75	360
0.053			380
0.073			382
0.093			385
0.053	7.5	25	222.3
		50	347.6
		75	380
0.053	7.5	50	347.6
	15		318.7
	22.5		276

It is found that the adsorbed amount depends of the bed height, the initial concentration and the flow rate. As the bed height increases the liquid residence time in the bed increases, and therefore the molecules of RAS diffuse deeply into the adsorbent grains. This results in an increase in the adsorbed amount which reaches its maximum value of 380mg / g when the bed height exceeds about 0.08m. Increasing inlet concentration results to increase its gradient in the liquid

film. Consequently, an improvement of the solute diffusion from the liquid phase to activated carbon grains. This results in an increasing of adsorbed amount. However, the adsorbed amount decreases, when the flow rate increases (Figure 5). In fact, an increase in flow rate results in a reduction in the thickness of the liquid film around grain, which has the effect of improving external diffusion, hence adsorption. But at the same time there is a reduction in the

residence time and the molecules of solute doesn't penetrate enough within the adsorbent grains. In sum, a decrease in the adsorbed amount is observed when the flow rate increases.

It should be noted that at zero flow velocity, the adsorbed amount value is about 385 mg / g (see Figure 5). It is equal to that determined by the Langmuir model.

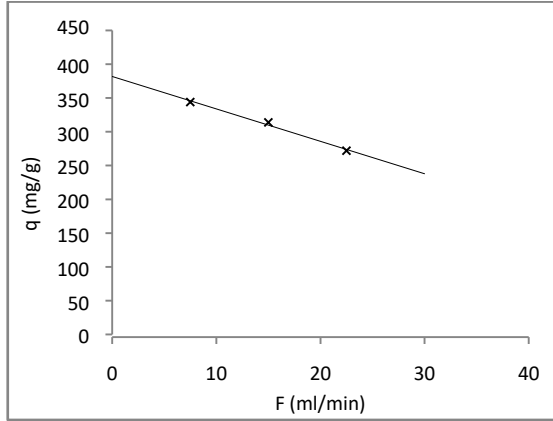


Fig. 5: Absorption capacity versus inlet flow velocity

As we have seen above, the adsorption capacity of the fixed bed is a function of the inlet concentration C_0 , the flow rate F and the bed depth Z . It can be written according to Danny et al. [26] in the form:

$$1 - \frac{q}{q_m} = w.C_0^a.F^b.(S.Z)^c \quad (2)$$

Where: $q_m = 385\text{mg/g}$ is the maximum absorption capacity.

And S is the column section.

The coefficients a , b and c are the slopes of line plotted in Figure 6 (a), (b), (c). The ordinates at the origin are respectively $C_1 = w.F^b.(S.Z)^c$; $C_2 = w.C_0^a.(S.Z)^c$ and $C_3 = w.C_0^a.F^b$, from which we deduce the mean value of w .

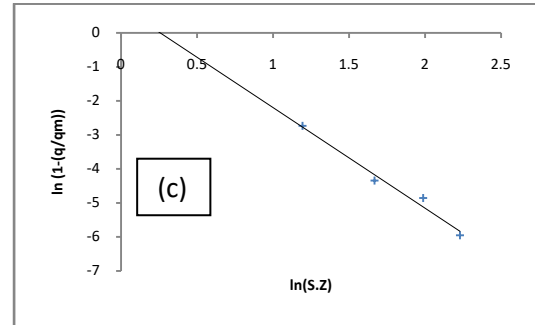
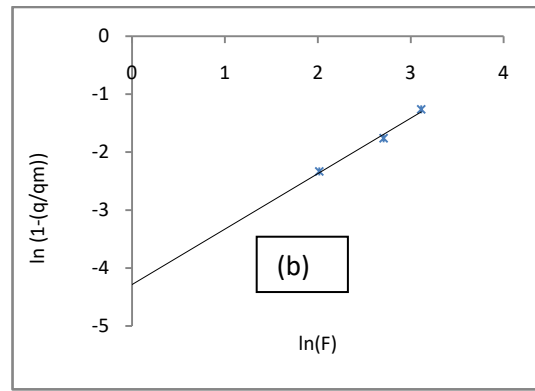
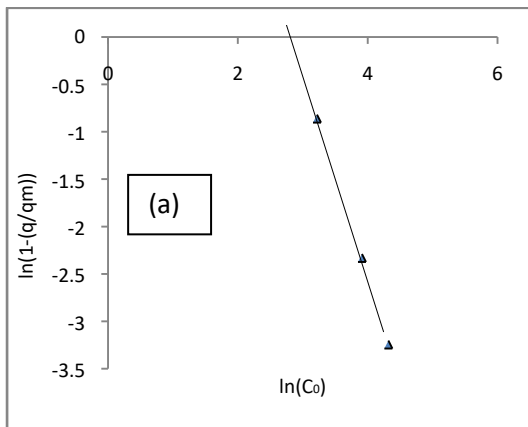


Fig. 6 (a), (b), (c): Determination of Equation 2 parameters

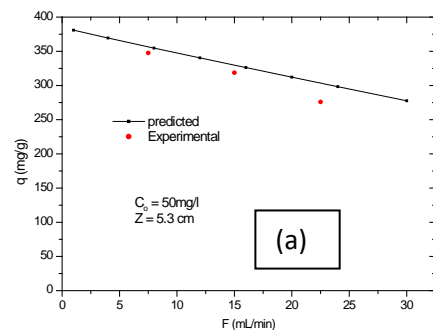
The adsorption capacity of Norit GCA830 in fixed bed for the RAS is given by the following expression:

$$1 - \frac{q}{q_m} = 5000 (C_0^{-2.17} . F^{0.96} . (SZ)^{-2.96})$$

It can be expressed as

$$q = 385 \left(1 - 5000 \left(C_0^{-2.17} . F^{0.96} . (SZ)^{-2.96} \right) \right) \quad (3)$$

Figures. 7 (a), (b), (c) shows a good correlation between the experimental values and those calculated from Equation (3).



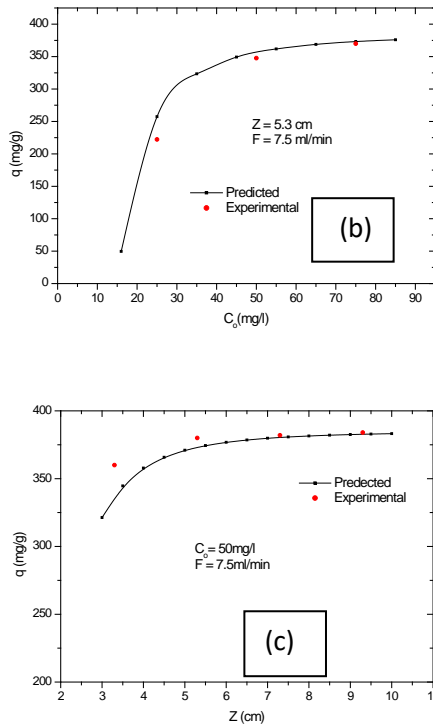


Fig. 7 (a), (b), (c) : Comparison of experimental and calculated absorption capacity for (a) different flow rate, (b) inlet concentration, (c) bed height

iii. *BDST model*

The present model, proposed by Hutchins [27], yields the breakthrough concentration C_b to the breakthrough time.

$$t_b = \frac{q}{C_0 u_0} Z - \frac{1}{k C_0} \ln\left(\frac{C_0}{C_b} - 1\right) \quad (4)$$

Where u_0 is the flow velocity (m/s)

According to the Equation (4), the curve $t_b = f(z)$ is a right line called Bed Depth Service Time (BDST). Its slope is $m_x = \frac{q}{C_0 u_0}$; allowed to calculate the absorption capacity when C_0 and u_0 are defined. In addition it can define the velocity of adsorption zone migration v , where it's the reverse of slope m_x .

The ordinate at the origin $C_x = \frac{1}{k C_0} \ln\left(\frac{C_0}{C_b} - 1\right)$

makes it possible to calculate the kinetic constant k . When C_0 and C_b are known.

In addition the thickness of mass transfer zone Z_0 is defined as the length at $t_b = 0$, it given by the following equation:

$$Z_0 = \frac{u_0}{qk} \ln\left(\frac{C_0}{C_b} - 1\right) \quad (5)$$

BEST model present many advantageous such as it reduce the experiments duration. In fact, it is sufficient to determine the breakthrough time,

corresponding to the breakthrough concentration C_b ($C_b = 0.1C_0$), at different bed height. The curves BDST plotted at different operating conditions shown in figure 8 (a), (b) are linear ($R > 0.988$).

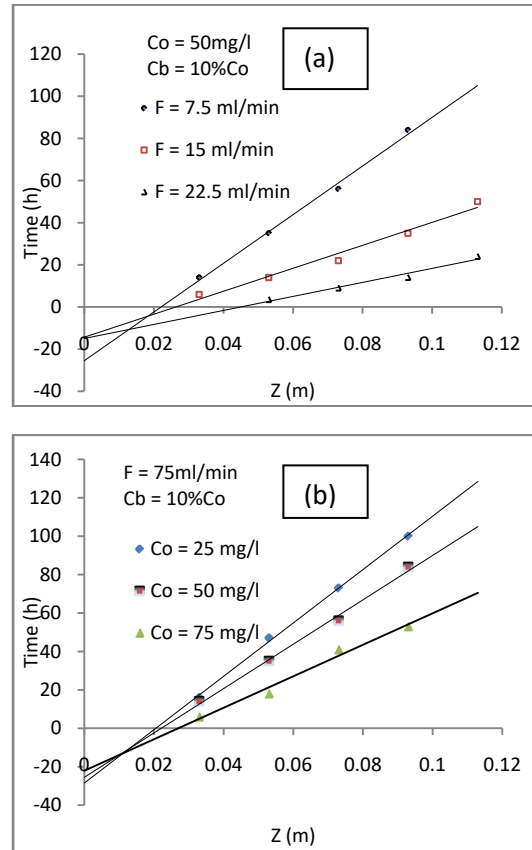


Fig. 8: Curves BDST at (a) different flow rate and (b) different inlet concentration

As it's shown in Figure 8(a), the variation of inlet concentration leads to the variation of the slope m_x , the ordinate at the origin C_x and the thickness of mass transfer zone Z_0 . In addition, increasing the flow rate, results in a decrease in the slope m_x and the ordinate at the origin C_x . Contrary to the thickness of mass transfer zone Z_0 (Figure 8(a)).

If m_x , C_x and Z_0 are defined from BDST curves, adsorption capacity q , velocity of migration of adsorption zone v and the kinetic constant k are defined too, their values are shown in Table 5 for the different operating conditions studied.

Table 5: Influence of initial concentration and flow rate on adsorption capacity q , velocity of migration of adsorption zone v and the kinetic constant k

C_0 (mg/l)	u_0 (m/h)	m_x (h/m)	C_x (h)	q (mg/g)	Z_0 (m)	k (l/kg.s)	v (10^3 m/h)
25	2.92	1390	28.57	209.2	0.02	0.854	0.72
	5.84	680	18.04	204.7	0.026	1.353	1.47
	8.76	415	17.2	187.4	0.041	1.42	2.4
	11.68	270	12.4	162.6	0.045	1.968	3.7
50	2.92	1155	25.515	348	0.022	0.478	0.866
	5.84	545	14.385	328.4	0.026	0.848	1.834
	8.76	332.5	14.973	300.5	0.045	0.815	3
	11.68	167.5	11.093	268.1	0.050	1.1	5.97
25	2.92	1390	28.57	209.2	0.020	0.854	0.72
50		1155	25.515	348	0.022	0.478	0.866
75		830	22.29	374.8	0.027	0.365	1.2

As indicated in Table 5, the absorption capacity increases if we increase the input concentration and if we reduce the flow rate. It reaches its maximum value of 374 mg/g for the lower flow rate studied. For the thickness of mass transfer zone Z_0 , it can be seen that it's proportional of inlet concentration and flow rate. In fact, an increase in the flow rate reduces the residence time of the solution in the bed and consequently the contact time solute-adsorbent. Thus the molecules don't penetrate enough within the adsorbent. This results in a widening of the height of adsorption zone.

Concerning the kinetic constant k , it can be seen that k increases as the flow rate increases. That because, the increase in the flow rate results in a reduction in the thickness of the film through which the diffusion becomes faster. With respect to the influence of the initial concentration, k decreases as C_0 increases. This evolution was also observed by Danny et al. [28] during the study of the adsorption of some metals in a fixed bed of activated carbon.

For the evaluation of the velocity of migration of the adsorption zone, it's clear that an increase in the flow rate or the inlet concentration leads to an increase in the velocity of migration and consequently the exhaustion of the bed and the saturation of the activated carbon, all the faster that u_0 and C_0 are higher.

IV. CONCLUSION

In the present work, our aim is to predict the adsorption capacity of fixed bed activated carbon Norit GCA830 to remove Alizarin Red S. From the

experiments in Batch reactor the adsorption capacity determine using Langmuir model is of 385 mg/g. We are found that both model of Langmuir and Freundlich fit well our experimental results, but the Langmuir model is better.

For fixed bed adsorption process, the breakthrough curves have been plotted at various inlet dye concentration, bed height and flow rate. The obtained results show that both breakthrough and exhaustion times increases with the increase of bed height and the decrease of flow rate and inlet solution dye concentration. According the operating condition, the maximum absorption capacity, calculated from breakthrough curve area, is about 380mg/g. Using breakthrough curve, the absorption capacity of fixed bed is correlated in function of studied operating condition as following:

The adsorption capacity estimated using the BDST model increase with the inlet concentration where it achieved its maximum value of 374mg/g for a lower flow rate studied. In addition from BDST model, allowed to define the thickness of absorption zone it's between 0.02 to 0.05m. The constant kinetic and the velocity of migration of absorption zone estimated by BDST model are mostly influenced by the residence time.

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A Simulation Study of the Factors that Impact Gas-Oil Ratio (GOR) Behavior in Liquid-Rich Shale (LRS) Reservoirs

By Ibukun Makinde

University of Houston

Abstract- The behavior of producing gas-oil ratio (GOR) in unconventional reservoirs like liquid-rich shales (LRS) and conventional reservoirs differ. This is mainly due to major disparity in the permeability – ultra-low in unconventional reservoirs in comparison to that in conventional (higher-permeability) reservoirs. The ultra-low permeability and porosity of shales, among other factors contribute to the complex fluid flow mechanisms in these plays. Therefore, there is a need for a good comprehension of the physics of flow in liquid-rich shale reservoirs. This paper particularly investigates how various factors, ranging from critical gas saturation to compaction affect producing gas-oil ratio behavior in liquid-rich shale (LRS) reservoirs. Ten different moderately volatile and highly volatile (near-critical) oil fluid compositions were considered. Compositional reservoir simulations for a period of 30 years were run on a base case multi-fractured horizontal well (MFHW) model for each fluid type.

Keywords: *unconventional resources; gas-oil ratio; liquid rich shales; volatile oil; production forecasting.*

GJRE-C Classification: FOR Code: 090499



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Abstract- The behavior of producing gas-oil ratio (GOR) in unconventional reservoirs like liquid-rich shales (LRS) and conventional reservoirs differ. This is mainly due to major disparity in the permeability – ultra-low in unconventional reservoirs in comparison to that in conventional (higher-permeability) reservoirs. The ultra-low permeability and porosity of shales, among other factors contribute to the complex fluid flow mechanisms in these plays. Therefore, there is a need for a good comprehension of the physics of flow in liquid-rich shale reservoirs.

This paper particularly investigates how various factors, ranging from critical gas saturation to compaction affect producing gas-oil ratio behavior in liquid-rich shale (LRS) reservoirs. Ten different moderately volatile and highly volatile (near-critical) oil fluid compositions were considered. Compositional reservoir simulations for a period of 30 years were run on a base case multi-fractured horizontal well (MFHW) model for each fluid type. Results showed that the different factors had varying impacts on the production performance and GOR behavior of LRS reservoirs – some more influential than others. Also, the fluid type, whether moderate or highly volatile oil, play a major role in determining how producing gas-oil ratios (GOR) behave in a LRS reservoir.

A proper understanding of unconventional reservoir production mechanisms is necessary for reliable reserves estimation, production forecasting and improving oil recovery. This work contributes to this mission and provides a better understanding of the performance of liquid-rich shale plays.

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I. INTRODUCTION

Liquid-rich shales (LRS) are shale rocks that contain high value oil and gas. Typical examples are the Eagle Ford play in Texas and the Bakken play in North Dakota, among several others. In recent times, LRS reservoirs have become viable sources of oil and gas production. Initially, the ultra-low permeability and porosity of shale formations made producing economic volumes of oil and gas from these reservoirs difficult. However, technological advancement in the form of multi-fractured horizontal wells (MFHW) has significantly improved production from these plays.

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In oil reservoirs, when reservoir pressure drops below the bubble point, solution gas evolves. The degree of undersaturation, production mechanisms of the reservoirs, fluid PVT properties and other factors determine the rate of solution gas production. Gas-oil ratio refers to the ratio of the volume of gas that evolves out of solution to the volume of produced oil at standard conditions. In the work by Beliveau (2004), there are three major factors that impact gas-oil ratio (GOR) performance – gas-oil relative permeability curve, the presence of initial gas cap and the strength of any associated aquifer. In the cases considered in this study, gas caps were absent and there were no associated aquifers. Solution gas drive is the primary drive mechanism in liquid-rich shale reservoirs.

This work studies the impacts of factors and parameters like bottomhole pressure, critical gas saturation, degree of undersaturation, fracture half-lengths, compaction, rock compressibility, etc. on producing gas-oil ratio (GOR). Whitson and Sunjerga (2012) demonstrated through the simulation of multi-fractured horizontal wells (MFHW) that producing GOR can be strongly dependent on the bottomhole pressure (BHP) when permeability is very low (approximately less than 0.001md). Also, Behmanesh et al. (2015) studied the GOR behavior of a multi-fractured horizontal well (MFHW) with constant BHP during linear flow. Jones Jr. (2016) investigated variations in the producing gas-oil ratio behavior of MFHW in tight oil reservoirs.

With a better knowledge of the behavior of producing GOR in liquid-rich shale (LRS) plays, forecasting of solution gas production can be possible. Yu (2014) presented a method for forecasting solution gas production based on predicted oil production. He proposed a specialized plot based on a linear relationship between the logarithm of a well's cumulative gas-oil ratio (GORcum) and cumulative oil production (Np). Makinde and Lee (2016) modified this method by considering a power law relationship between these two variables. Also, Makinde and Lee (2016) presented a different approach to forecasting production from LRS reservoirs – Principal Components Methodology (PCM), based on the statistical data-driven technique of principal components analysis. PCM was also used in another study by Makinde and Lee (2016) to forecast solution gas production from LRS reservoirs.

II. RESERVOIR MODEL DESCRIPTION

A 5000 ft horizontal well, with 20 hydraulic fractures spaced 250 ft apart was modeled. The fractures have half lengths of 150 ft and are all infinitely conductive. Fracture width of 2 ft was used to make simulation easier. Fracture permeability was correspondingly reduced to keep the product of width and permeability (of fractures) at an appropriate level. Reservoir models with the same fracture conductivity but different fracture widths yield similar results (Alkough et al., 2012).

A commercial compositional simulator was used to simulate production with ten different reservoir fluids (moderately and highly volatile oils). Fluids 3 and 4 are near-critical fluids. The well produced for 30 years at a minimum bottomhole pressure constraint of 1000 psia. Logarithmically-spaced local grid refinement (LS-LGR) was used to model pressure drop and fluid flow as accurately as possible. Figure 1 shows a pictorial representation of the reservoir model. Tables 1 and 2 show the reservoir data and the reservoir fluid compositions used.

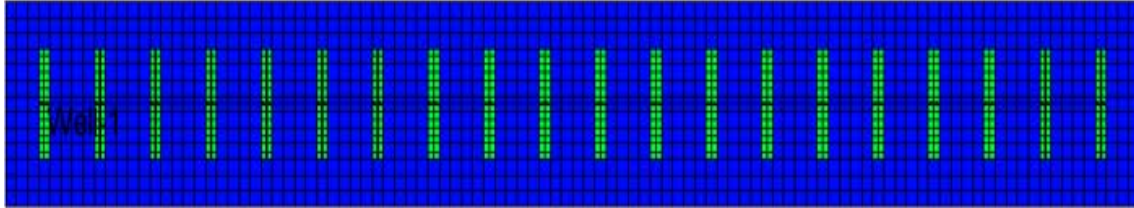


Figure 1: Basecase Multi-Fractured Horizontal Well (MFHW) Model

Table 1 Reservoir Data for MFHW Model

Permeability	0.001 md
Porosity	0.06
Reservoir Temperature	250°F
Initial Reservoir Pressure	5,000 psia
Depth to top of formation	10,000 ft
Reservoir Thickness	250 ft
Corey Relative Permeability Exponent	2.5
Critical gas saturation, S_{gc}	0.05
Residual saturation of oil (gas/oil displacement), S_{orm}	0.2

Table 2 Fluid Compositions

	Fluid 1	Fluid 2	Fluid 3	Fluid 4	Fluid 5	Fluid 6	Fluid 7	Fluid 8	Fluid 9	Fluid 10
Components	Composition (%)	Composition (%)	Composition (%)	Composition (%)	Composition (%)	Composition (%)	Composition (%)	Composition (%)	Composition (%)	Composition (%)
CH ₄	58.77	58.07	61.82	53.47	49.43	49.96	48.78	51.93	44.42	41.52
C ₂ H ₆	7.57	7.43	7.91	11.46	7.28	6.44	6.24	6.64	9.52	6.12
C ₃ H ₈	4.09	4.16	4.42	8.79	8.02	3.48	3.49	3.71	7.30	6.74
I-C ₄ H ₁₀	0.91	0.96	1.02	-	2.31	0.77	0.81	0.86	-	1.94
N-C ₄ H ₁₀	2.09	1.63	1.74	4.56	3.61	1.78	1.37	1.46	3.79	3.03
I-C ₆ H ₁₂	0.77	0.75	0.80	-	1.80	0.66	0.63	0.67	-	1.51
N-C ₆ H ₁₂	1.15	0.80	0.86	2.09	1.79	0.98	0.67	0.72	1.74	1.50
C ₈ H ₁₄	1.75	1.14	1.21	1.51	2.32	1.49	0.96	1.02	1.26	1.95
C ₇₊	21.76	22.59	17.59	16.92	22.41	33.50	34.98	30.78	30.98	34.82
CO ₂	0.93	2.32	2.47	0.90	0.16	0.79	1.95	2.08	0.75	0.13
N ₂	0.21	0.15	0.16	0.30	0.87	0.18	0.13	0.13	0.25	0.73
	Highly Volatile Oils					Moderately Volatile Oils				
GOR, scf/bbl	3,024	3,043	4,081	3,967	2,561	1,806	1,755	2,128	1,873	1,513
API	63.5	63.0	63.5	64.9	65.2	49.2	49.1	46.8	49.7	50.6
Oil FVF, bbl/stb	3.56	3.55	-	4.81	3.26	2.23	2.19	2.42	2.32	2.10

III. SOLUTION GAS DRIVE MECHANISM

LRS reservoirs under consideration in this work are shale volatile oil reservoirs (fluids are moderately and highly volatile oils). Solution gas drive is the primary drive mechanism in shale volatile oil reservoirs. In this study, the reservoir is initially undersaturated i.e., the initial reservoir pressure is greater than the saturation pressure (bubble point pressure). At this time, production is mainly driven by the bulk expansion of

reservoir rock and oil. When reservoir pressure drops below the bubble point, expansion of gases dissolved in oil provide most of the reservoir drive energy. Illustrations of gas-oil ratio history, reservoir pressure and gas saturation with time for one of the fluid samples in the basecase scenario are shown in Figures 2, 3 and 4. Figure 3 is a semi-log plot of the gas-oil ratio history to enable proper visibility of the various critical points of production mechanism of shale volatile oil reservoirs.

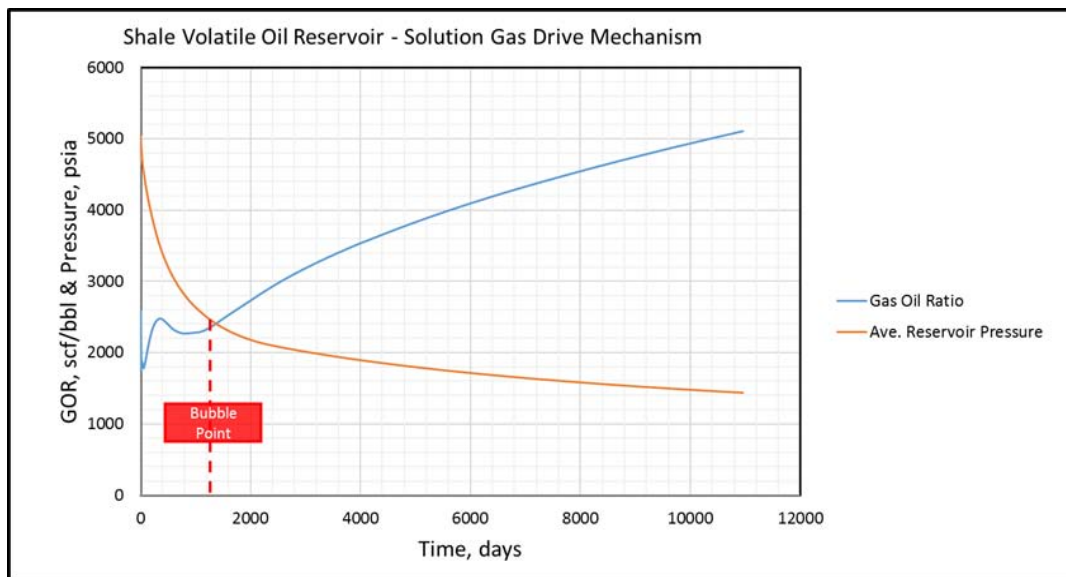


Figure 2: Shale Volatile Oil Reservoir – Solution Gas Drive Mechanism

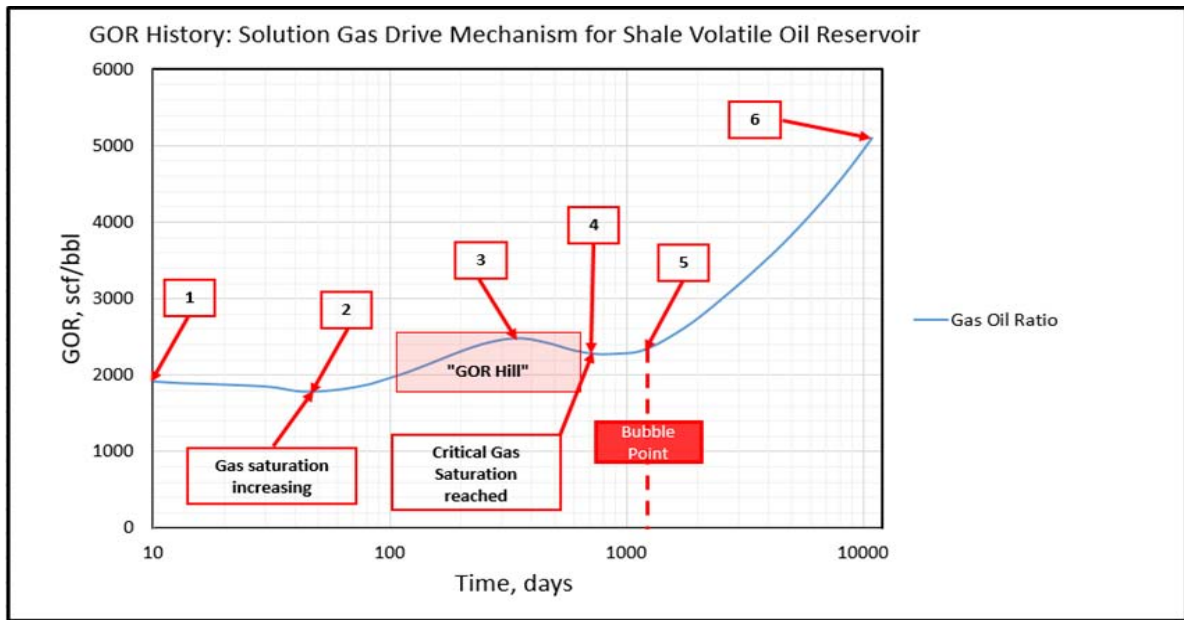


Figure 3: GOR History: Solution Gas Drive Mechanism for Shale Volatile Oil Reservoirs

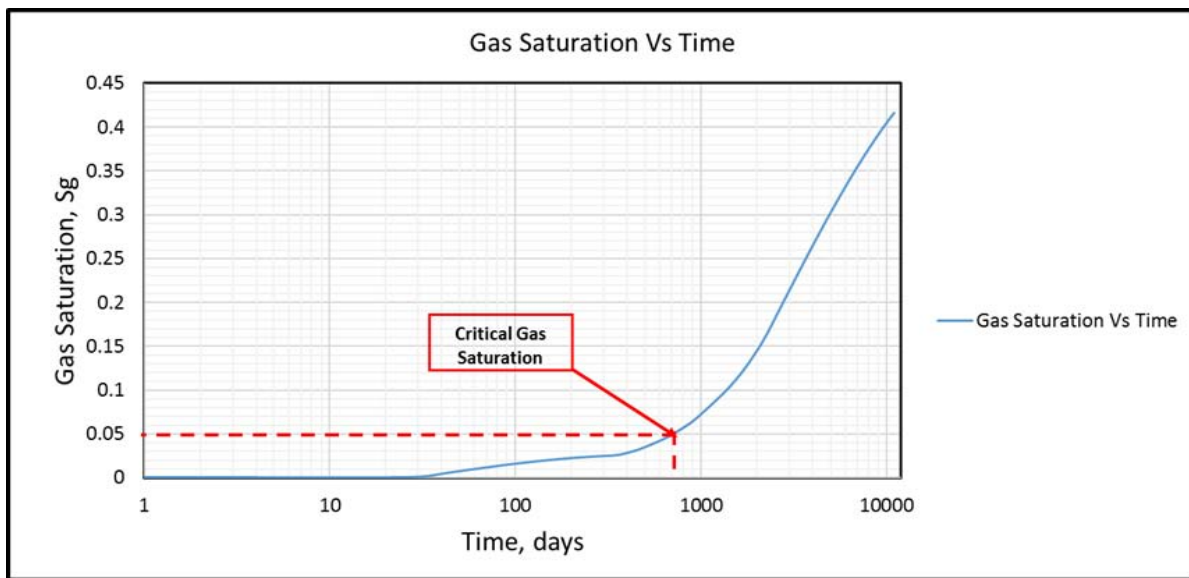


Figure 4: Gas Saturation vs. Time

In Figure 2, it is evident that the reservoir pressure declines rapidly before reaching the bubble point. Beyond the bubble point, the rate of decline slows due to the evolution of gas. The six critical stages of the GOR history of a well in a shale volatile oil reservoir driven by solution gas drive mechanism shown in Figure 3 are briefly explained below:

Reservoir pressure is greater than the saturation pressure (bubble point pressure). Here, no free gas exists in the formation and the producing GOR is approximately equal to the initial solution GOR (i.e., approximately constant GOR);

The gas saturation starts to increase forming a "GOR hill". Though gas is not mobile yet, there is an

increase in the amount of gas released from oil from point 2 to 3 and an increasing gas saturation;

Due to the continuous rapid decline in pressure above the bubble point, gas solubility decreases from point 3 to 4;

The critical gas saturation is reached and gas can flow;

At this point, the reservoir pressure decreases below the bubble point, gas evolution accelerates and producing GOR starts to increase rapidly;

Producing GOR is still increasing after 30 years. For shale oil reservoirs, the producing GOR may continue to increase for even longer due to ultra-low permeability of shales and other contributing factors.

The producing GOR for all the fluid samples (basecases) are compared and shown in Figure 5. They all have a similar trend but generally, the more volatile

the fluid, the higher the producing GOR throughout the production period.

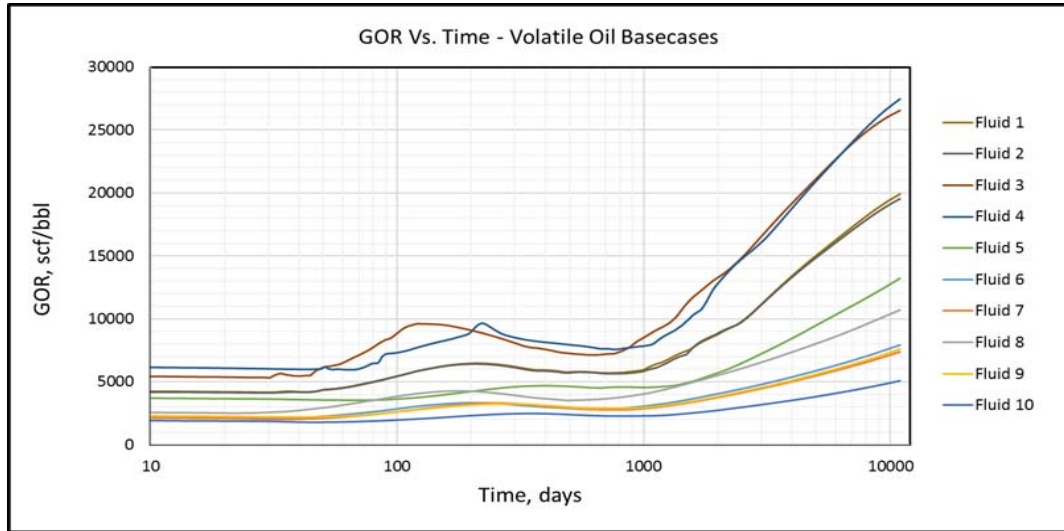


Figure 5: GOR vs. Time – Volatile Oil Basecases

Next, the effects of several factors and parameters on the gas-oil ratio (GOR) behavior of multi-fractured horizontal wells (MFHW) in shale volatile oil reservoirs were examined.

IV. CRITICAL GAS SATURATION

The gas produced when reservoir pressure drops below the saturation pressure in an oil reservoir

remains immobile until it reaches a certain threshold. This threshold is called the critical gas saturation. At and above the critical gas saturation, gas become mobile and begin to flow towards the wellbore. Critical gas saturations of 5% (basecase), 10%, 15% and 20% were considered to determine the impact on the performance of MFHW in shale volatile oil reservoirs.

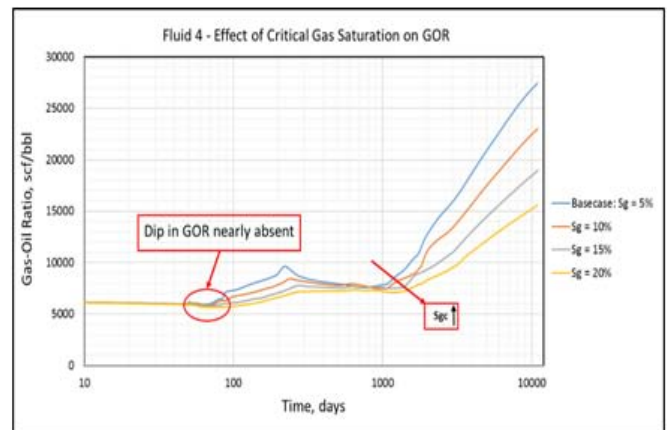
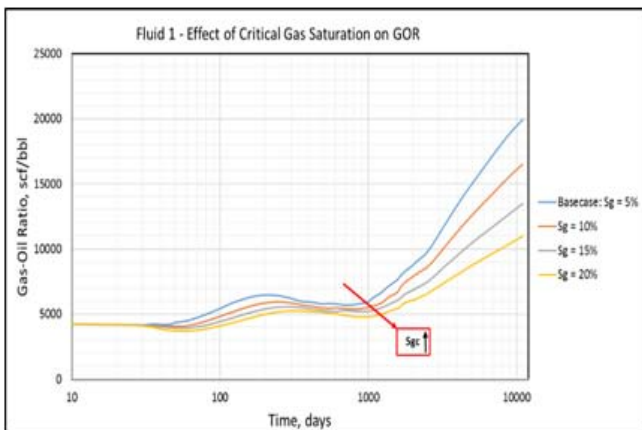


Figure 6: Fluids 1&4 – Effect of Critical Gas Saturation on GOR

Figures 6 and 7 illustrate the impacts of critical gas saturation on producing GOR (semi-log plots) for Fluids 1, 4, 7 and 10. Generally, the higher the critical gas saturation, the lower the producing GOR with time. There is also a delay in the rise of producing GOR with time, as critical gas saturation increases. With increasing critical gas saturation, there is a slight dip in producing GOR after the period of constant GOR. The further away the fluid is from the critical point, the more pronounced the dip is. Fluid 4 is a near-critical fluid, therefore, the dip

in producing GOR after the constant GOR period, is nearly absent in these cases. This can be observed in Figure 6.

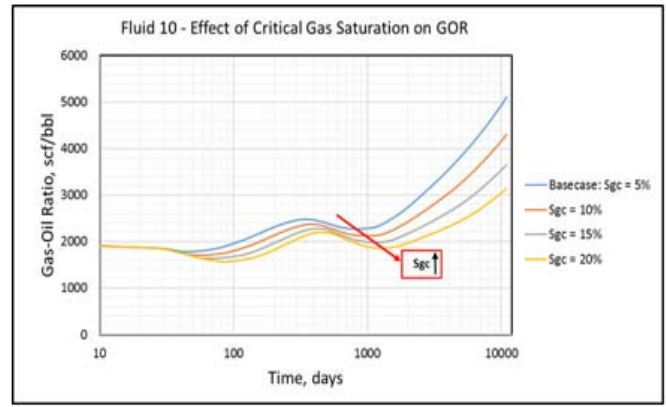
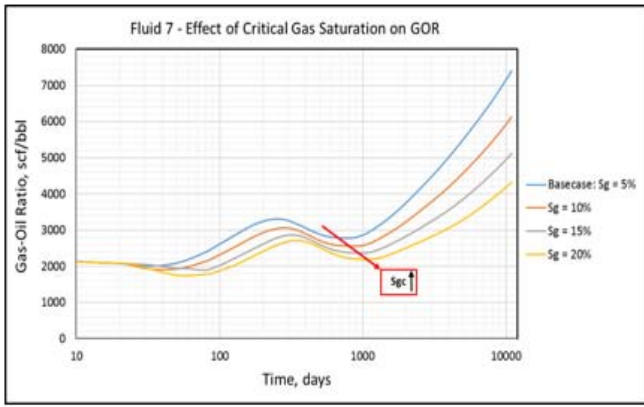


Figure 7: Fluids 7&10 – Effect of Critical Gas Saturation on GOR

V. BOTTOMHOLE PRESSURE (BHP)

The wells under consideration here produce at constant flowing bottomhole pressure (BHP). The lower the BHP below the saturation pressure, the more the drawdown. Cases of different constant flowing BHPs were considered including when the BHP is equal to the bubble point pressure. The basecase is a constant flowing BHP of 1000 psi. The lower the constant flowing BHP, the higher the producing GOR except for the cases of 100 psi and below for the least volatile oil – Fluid 10, 250 psi and below for other moderately volatile oils and from 500 psi and below for highly volatile oils. In these cases, the producing GOR towards the end of the production time decreases with lesser constant flowing

BHP due to the large drawdown which led to the production of gas reaching a peak quickly and declining with time till the end. The more volatile the fluid, the quicker the producing GOR reaches a peak and starts to decline even at higher flowing bottomhole pressures. When the constant flowing BHP is equal to the bubble point pressure, the producing GOR remains constant throughout the production. There is a mild increase in producing GOR with time for the case of BHP equal to 2000 psi (slightly lower than the saturation pressure in most of the cases). Figures 8 and 9 show the effects of bottomhole pressure (BHP) on producing gas-oil ratio (semi-log plots) for Fluids 1, 4, 7 and 10.

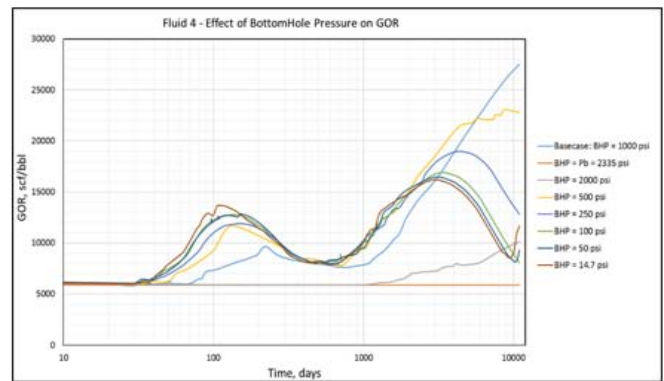
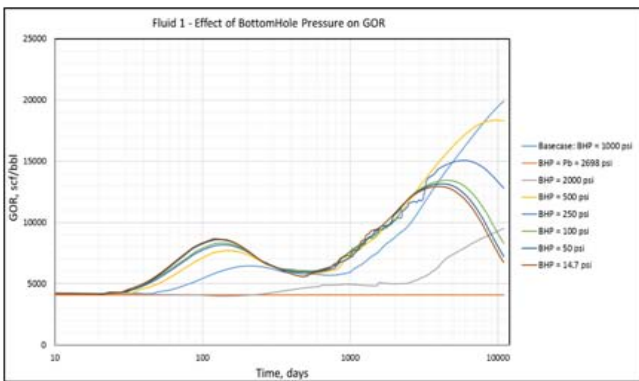


Figure 8: Fluids 1&4 – Effect of Bottomhole Pressure on GOR

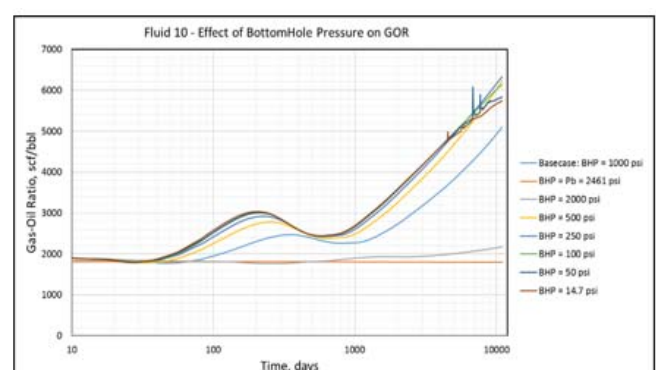
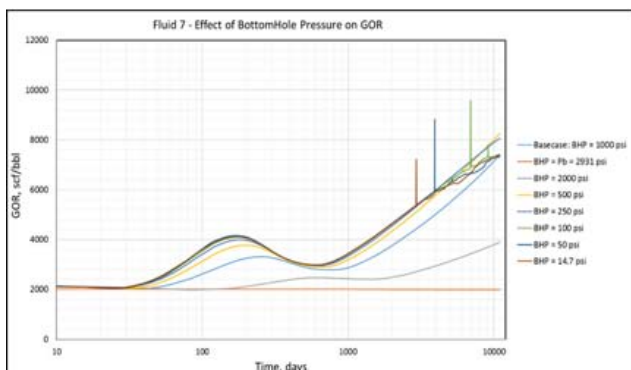


Figure 9: Fluids 7&10 – Effect of Bottomhole Pressure on GOR

VI. DEGREE OF UNDERSATURATION

The degree of undersaturation is the difference between the initial reservoir pressure and the saturation (bubble point) pressure. Cases with initial reservoir pressures of 5000 psi (basecase), 4500 psi, 4000 psi and 3500 psi were studied. The lower the degree of undersaturation, the quicker the reservoir pressure will reach the saturation pressure. Therefore, with decreasing degree of undersaturation, the producing GOR increases with time and vice versa. Correspondingly, there is a delay in the initial rise of producing GOR with increasing degree of

undersaturation and vice versa. Likewise, the higher the degree of undersaturation, the lesser the height of the “GOR hill”. Moreover, the higher the degree of undersaturation, the longer the period (at the start of production) where the producing GOR remains constant i.e., the period where the producing GOR is approximately equal to the initial solution GOR. Figures 10 and 11 show the effects of the degree of undersaturation on the producing GOR (semi-log plots) for Fluids 1, 4, 7 and 10. Generally, the trends are similar in all cases regardless of the volatility of the volatile oil fluid sample considered.

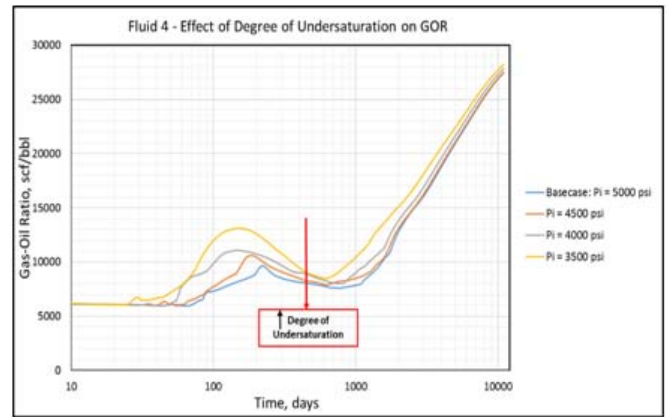
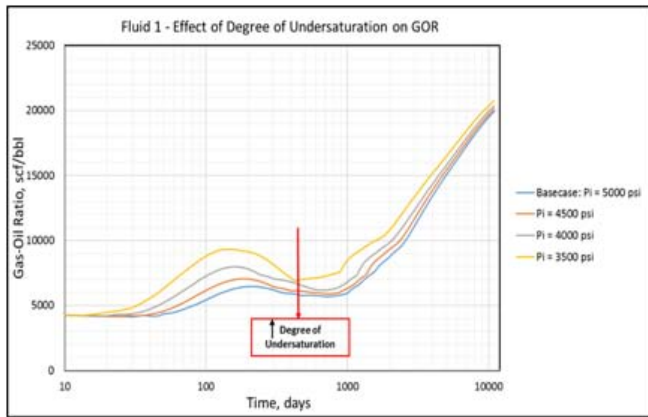


Figure 10: Fluids 1&4 – Effect of Degree of Undersaturation on GOR

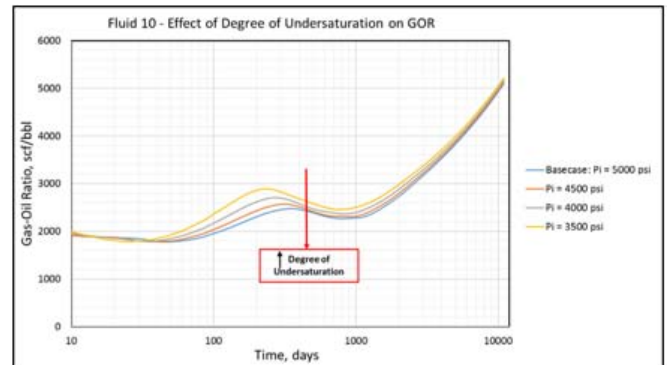
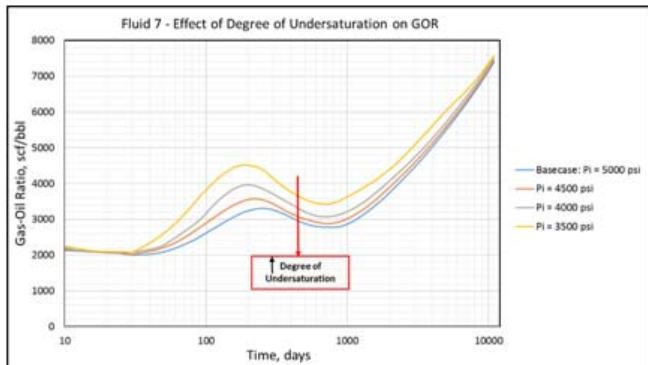


Figure 11: Fluids 7&10 – Effect of Degree of Undersaturation on GOR

VII. DRAINAGE AREA

Drainage area is the reservoir area drained by the well. The drainage area was varied to investigate the effect it has on well performance in shale volatile oil reservoirs. Apart from the basecase (Figure 1) that has

an approximate drainage area of 76 acres, two other different cases were considered – drainage area 1 (approximately 104 acres) and drainage area 2 (approximately 275 acres). Figures 12 and 13 show the pictorial description of drainage areas 1 and 2.

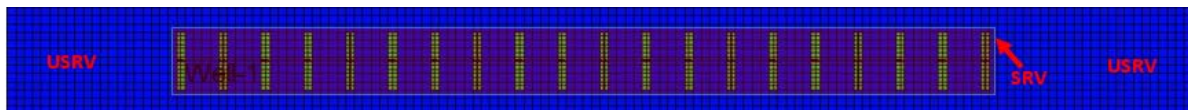


Figure 12: Drainage Area 1 (Approx. 104 acres)

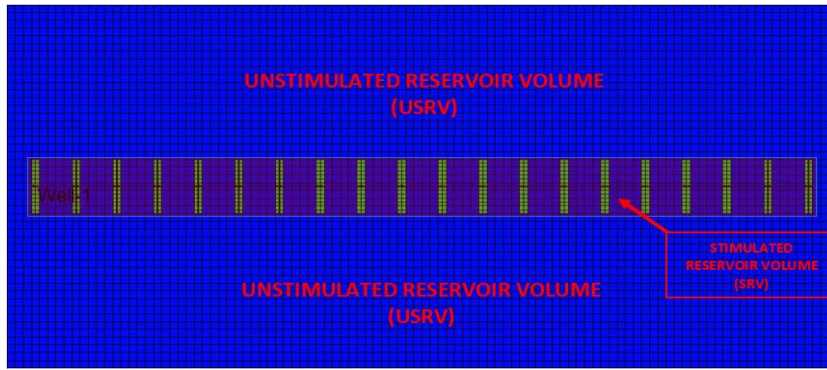


Figure 13: Drainage Area 2 (Approx. 275 acres)

For the case with drainage area of approximately 275 acres (drainage area 2 – Figure 13), boundary-dominated flow (BDF) is not reached in some instances due to low permeability and the relatively large unstimulated reservoir volume (USRV). This is the situation especially when moderately volatile oil reservoir fluids are present. For highly volatile oils, BDF is observed because of higher oil mobility (less viscosity in comparison to less volatile oils) towards the regions close to the stimulated reservoir volume (SRV). This BDF is followed by a late linear (or compound linear) flow when production from the unstimulated reservoir volume (USRV) dominates. The trend of producing GOR is generally the same till boundary-dominated flow (as observed on the rate-time diagnostic plots) is reached. According to Jones Jr. (2016), for multi-fractured horizontal wells (MFHW), producing GOR rises during BDF because of declining pressures at the midpoint

between fractures and corresponding increase in average gas saturation in the drainage area. This phenomenon is observable in our results. After boundary-dominated flow, there is a steeper rise in producing GOR with reducing reservoir drainage area. With increasing reservoir drainage area, it takes longer to reach boundary-dominated flow (BDF is not even observed in some cases depending on the volatility of the reservoir fluid). Therefore, the larger the reservoir area, the milder the rise in producing GOR with time. Due to the higher mobility of highly volatile oils, production may later be dominated by the regions beyond the SRV (for larger reservoir drainage areas), leading to the decline of producing GOR towards the end of the production period (30 years in our cases). Figures 14 to 17 show the impacts of drainage area on rate-time diagnostic plots and producing GOR (semi-log plots) for Fluids 1, 4, 7 and 10.

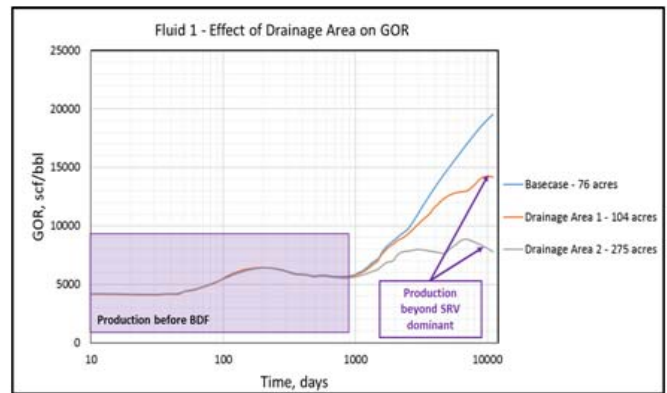
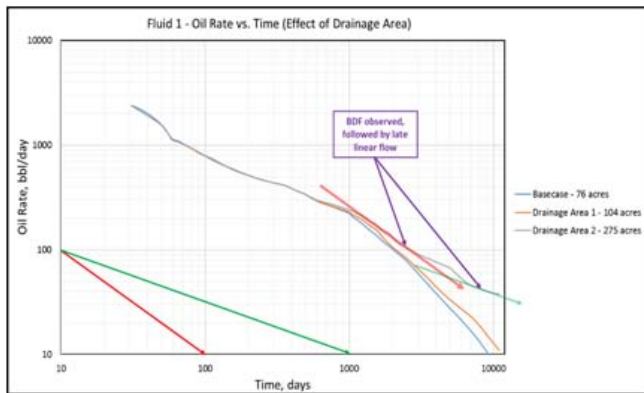


Figure 14: Fluid 1 – Effect of Drainage Area on GOR and Rate-Time Diagnostic Plots

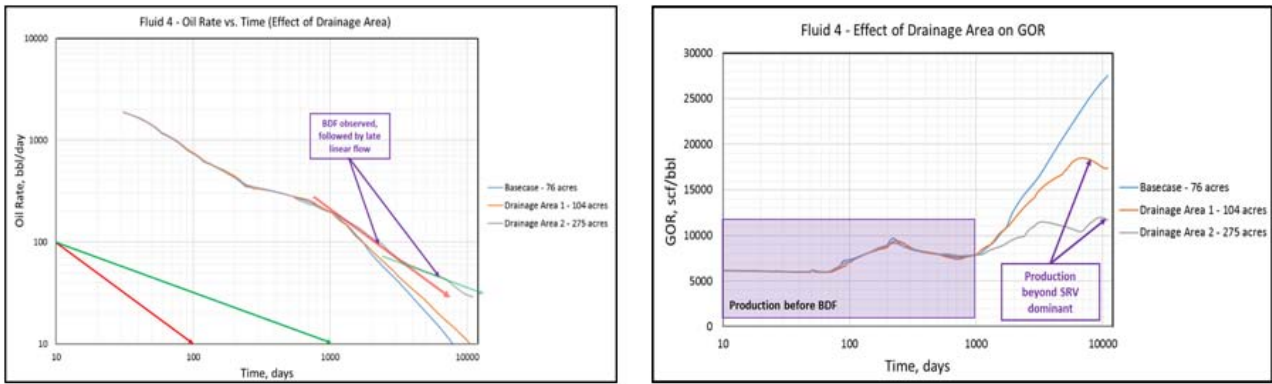


Figure 15: Fluid 4 – Effect of Drainage Area on GOR and Rate-Time Diagnostic Plots

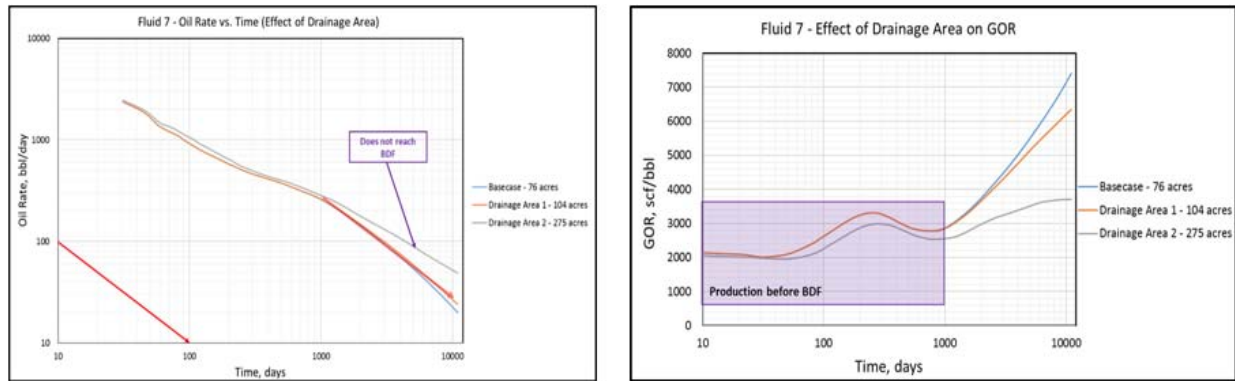


Figure 16: Fluid 7 – Effect of Drainage Area on GOR and Rate-Time Diagnostic Plots

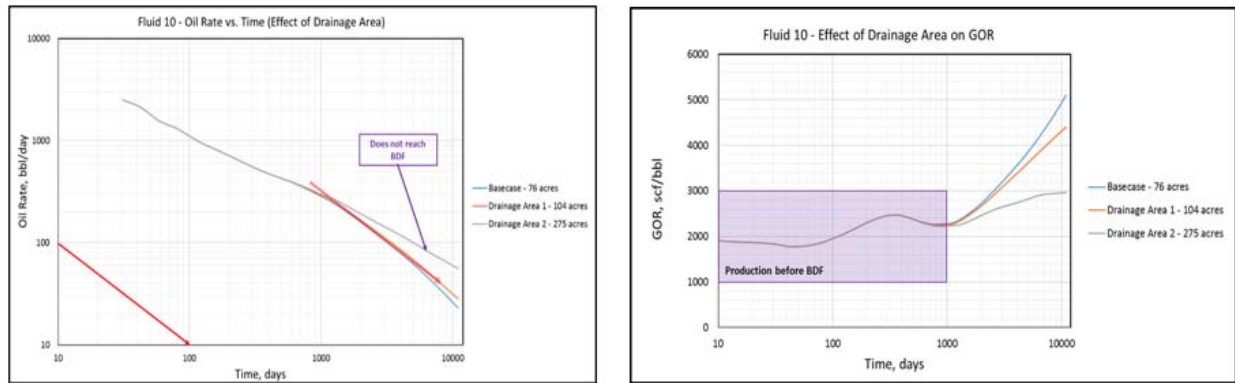


Figure 17: Fluid 10 – Effect of Drainage Area on GOR and Rate-Time Diagnostic Plots

VIII. FRACTURE HALF-LENGTH

Fracture half-length is the distance from the wellbore to the outer tip of a fracture propagated from the well by hydraulic fracturing or penetrated by the well. It is an important completion parameter for shale reservoirs. For these analyses, fracture half-lengths of 50 ft, 100 ft, 150 ft (basecase), 200 ft, 250 ft, 300 ft and

two other cases where the fracture half-lengths are of different lengths, i.e. uneven configuration of fracture lengths were considered. These two special cases were compared separately to the basecase to determine their impact on production performance. Figures 18 to 24 show the pictorial representations of each case apart from the basecase (already shown in Figure 1).

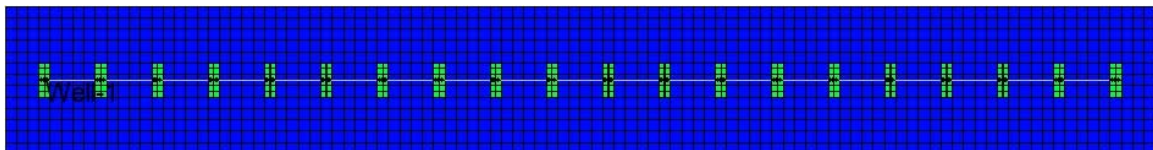


Figure 18: Reservoir Model – 50 ft Fracture Half-Lengths

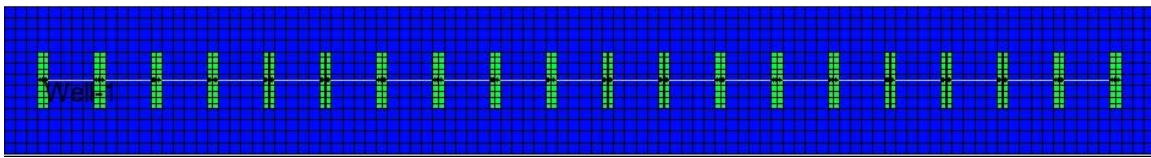


Figure 19: Reservoir Model – 100 ft Fracture Half-Lengths

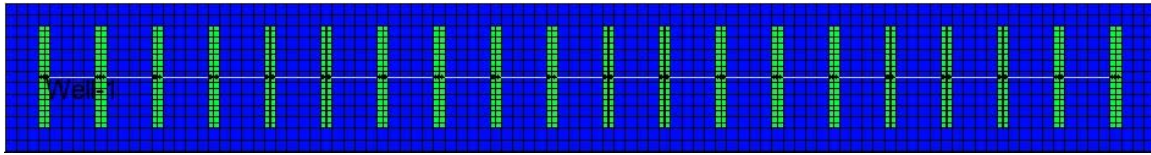


Figure 20: Reservoir Model – 200 ft Fracture Half-Lengths

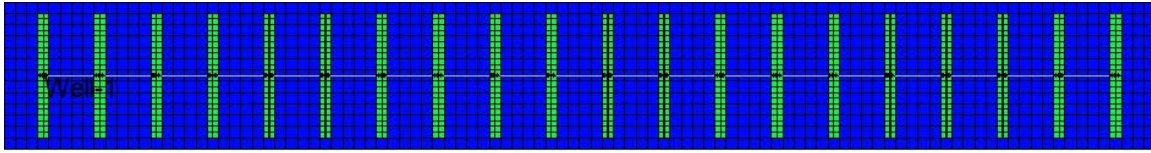


Figure 21: Reservoir Model – 250 ft Fracture Half-Lengths

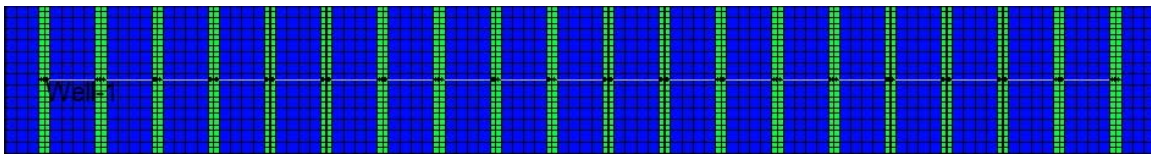


Figure 22: Reservoir Model – 300 ft Fracture Half-Lengths

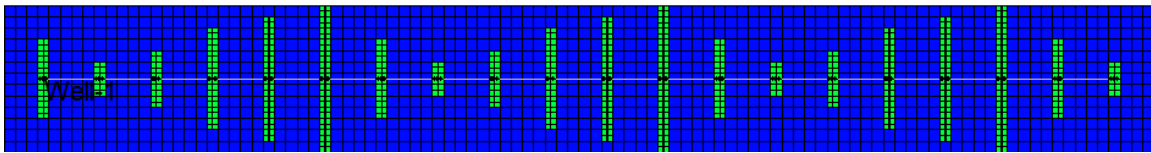


Figure 23: Reservoir Model – Uneven Configuration 1 (Fracture Half-Lengths)

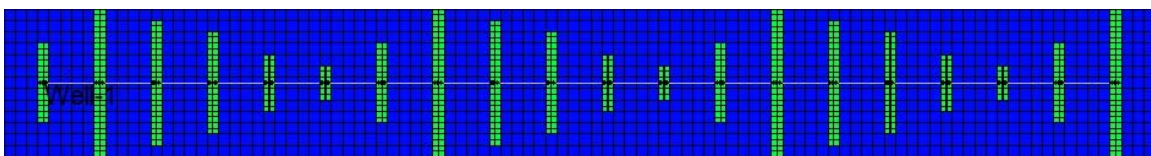


Figure 24: Reservoir Model – Uneven Configuration 2 (Fracture Half-Lengths)

There is a delay in the rise of producing GOR with reducing fracture half-lengths. The shorter the fracture half-length, the lesser the gas saturation at the fracture faces. Also, the further away the bubble point of the volatile oil is from the initial reservoir pressure (degree of undersaturation), the lower the height of the “GOR hill”. This is more noticeable for cases with highly volatile oils. Therefore, the higher the degree of undersaturation and the shorter the fracture half-lengths, the lower the height of the “GOR hill”. The highly volatile oils are closer to the critical point (two fluids are near-critical), therefore in most of these instances, the “GOR hill” is very low or absent during the production period.

Reservoir model with uneven configuration 1 has three of its fractures with half-lengths of 300 ft whereas the reservoir model with uneven configuration 2

has four of its fractures with half-lengths of 300ft. Therefore, the well with uneven configuration 2 generally produce more oil than the well with uneven configuration 1. They both produce more oil than the well with the basecase configuration (uniform fracture half-lengths of 150 ft). The producing GOR generally follows the same trend as already discussed in the previous paragraph. Figures 25 and 26 illustrate the effects of fracture half-lengths on producing GOR (semi-log plots) with time for Fluids 1 and 10.

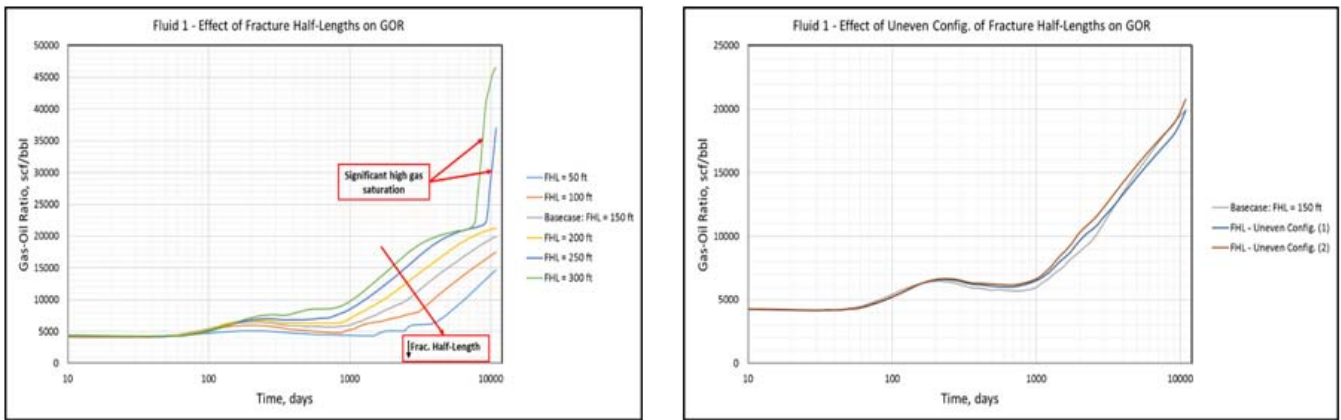


Figure 25: Fluid 1 – Effect of Fracture Half-Lengths on GOR

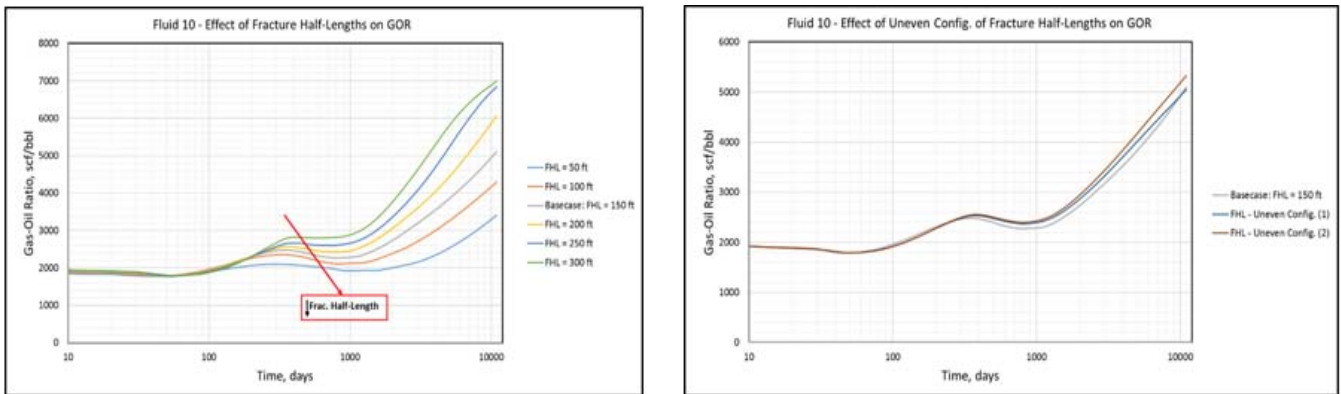


Figure 26: Fluid 10 – Effect of Fracture Half-Lengths on GOR

IX. FRACTURE PERMEABILITY

Fracture permeability is a measure of the ease with which fluids flow through the connecting pore spaces of fractured rocks. In other words, it is a measure of the ability of fractured rocks to transmit fluids. Fracture permeability is directly proportional to the dimensionless fracture conductivity, as seen in Equation 1 below.

$$F_{CD} = \frac{k_f w_f}{k x_f}, \quad (1)$$

where FCD is the dimensionless fracture conductivity, k_f is the fracture permeability, w_f is the fracture width, k is the formation permeability and x_f is the fracture half-length. In the analyses of the impacts of fracture permeability on well performance, fracture permeabilities of 5 md, 10 md, 20 md, 60 md, 80 md and the basecase – 41.65 md were considered. With reducing fracture permeability, there is a delay in the increase of gas saturation at the fracture faces. Consequently, there is a delay in the formation of the “GOR hill” (delay in the initial rise of producing GOR) and longer period of constant GOR. The reverse is the case with increasing fracture permeability. Figures 27 and 28 show the impacts of fracture permeability on

producing GOR (semi-log plots) for Fluids 1, 4, 7 and 10.

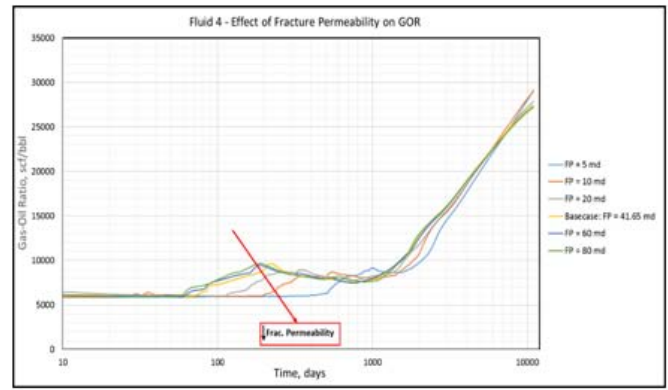
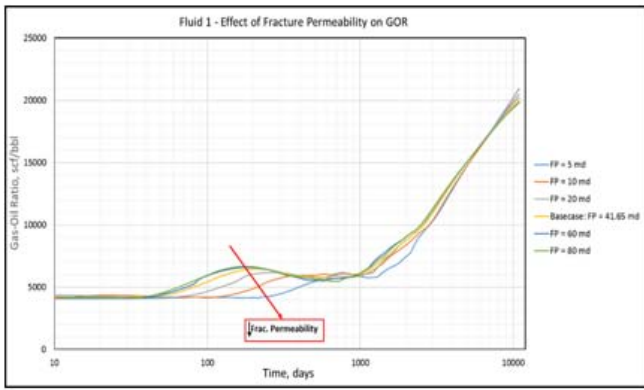


Figure 27: Fluids 1&4 – Effect of Fracture Permeability on GOR

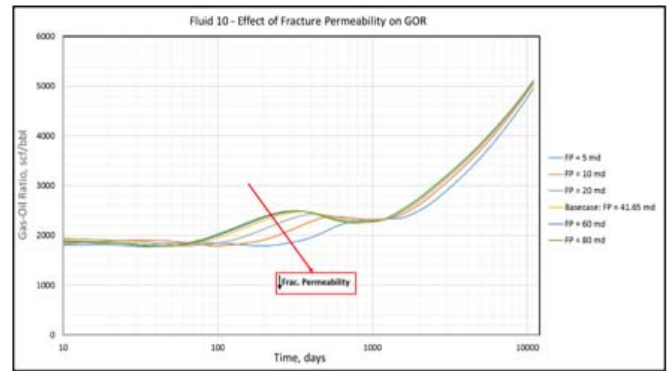
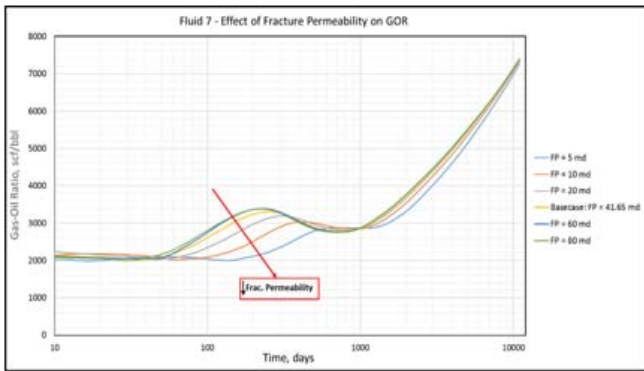


Figure 28: Fluids 7&10 – Effect of Fracture Permeability on GOR

X. FRACTURE SPACING

Fracture spacing is a vital parameter to consider during well completions. It is possible for reservoir engineers to generate different scenarios that can help completion engineers find the optimum spacing necessary for their operations. Apart from the basecase – fracture spacing of 250 ft (20 fracture stages), four other cases were considered in this study –

100 ft (50 fracture stages), 500 ft (10 fracture stages) and two different scenarios with uneven fracture spacing. The cases with uneven fracture spacing are compared to the basecase separately to investigate the impact of non-uniform fracture spacing on shale volatile oil well production performance. Figures 29 to 32 show the reservoir models for the different instances. The basecase reservoir model is already shown in Figure 1.

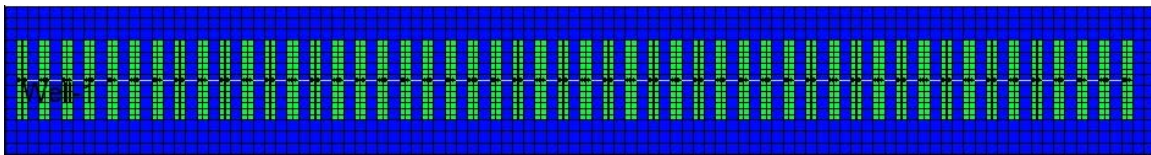


Figure 29: Reservoir Model – 100 ft Fracture Spacing (50 Fracture Stages)

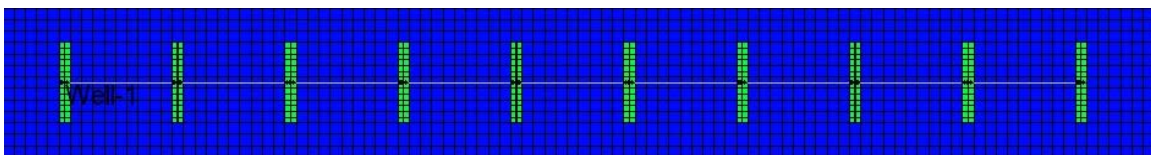


Figure 30: Reservoir Model – 500 ft Fracture Spacing (10 Fracture Stages)

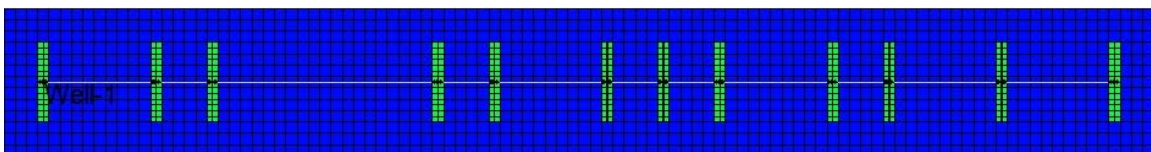


Figure 31: Reservoir Model – Uneven Configuration 1 (Fracture Spacing)

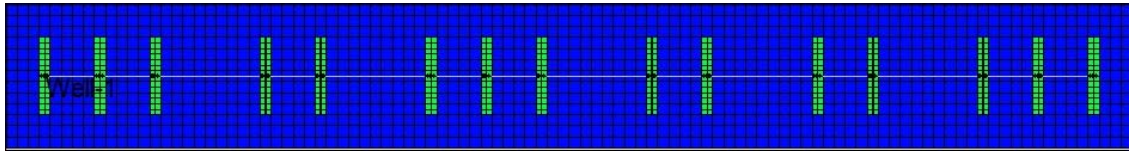


Figure 32: Reservoir Model – Uneven Configuration 2 (Fracture Spacing)

Even though closer fracture spacing (more fracture stages) requires a higher completion cost per well, it eventually means better drainage of the SRV within a shorter period (Makinde, 2014). The closer the fracture spacing, the larger the cumulative oil production. For highly volatile oils, cumulative oil production starts to reduce with closer fracture spacing later during production because of high gas saturation.

The effect of fracture spacing on producing GOR is quite significant. The closer the fracture spacing, the more rapid the critical gas saturation is reached. This therefore results in higher producing GOR with time as fracture spacing reduces. For highly volatile oils, high gas saturation can result in very high producing GOR towards the end of the production period.

For the special cases with uneven fracture spacing, the well with uneven configuration 2 (15

fracture stages) has closer fracture spacing in comparison with the well with uneven configuration 1 (12 fracture stages). This can be observed in Figures 31 and 32. Therefore, though fracture spacing is non-uniform and since the well with uneven configuration 2 generally has closer fracture spacing than that with uneven configuration 1, it produces more oil (larger cumulative oil production). Oil produced in both cases is lower than the oil produced from the well with basecase configuration (Figure 1). This is because they both have lesser fracture stages than the basecase (20 fracture stages). The impact on producing GOR is like earlier discussed scenarios. The closer the fracture spacing, the higher the producing GOR with time. Figures 33 and 34 show the effects of fracture spacing on producing GOR (semi-log plots) for Fluids 1 and 10.

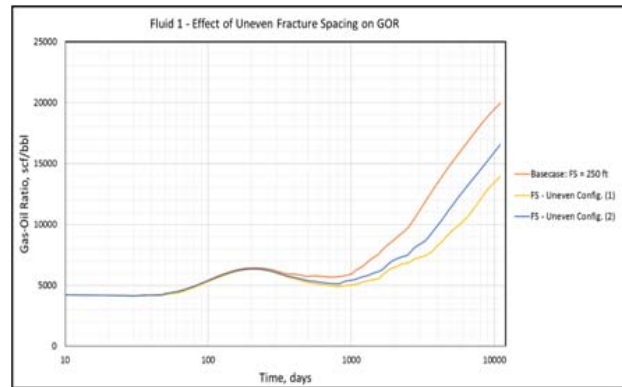
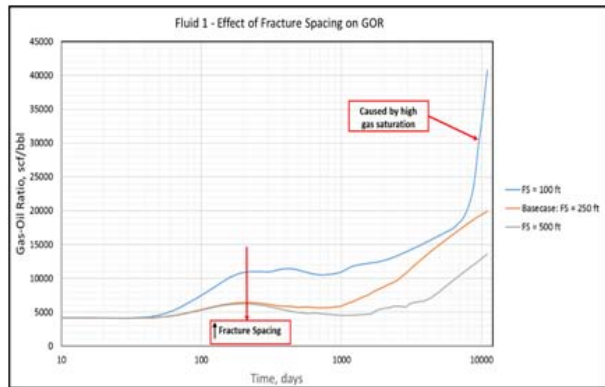


Figure 33: Fluid 1 – Effect of Fracture Spacing on GOR

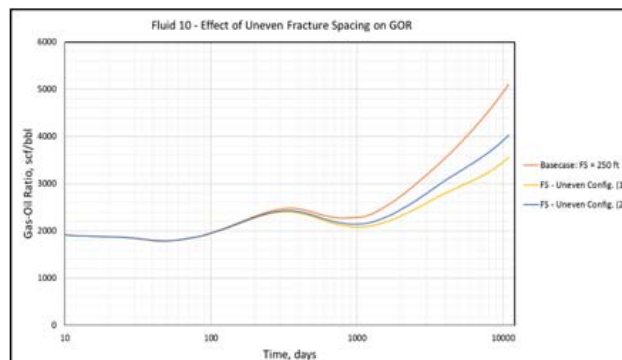
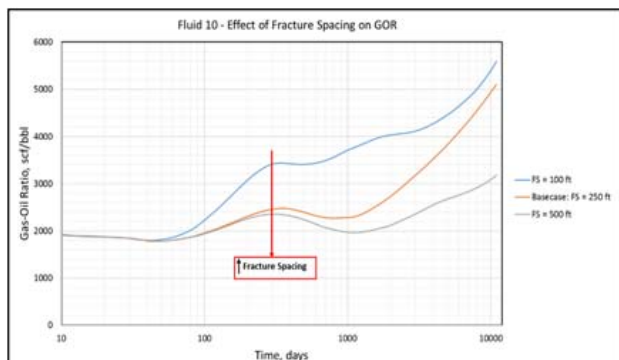


Figure 34: Fluid 10 – Effect of Fracture Spacing on GOR

XI. ROCK COMPRESSIBILITY

Rock compressibility is the isothermal change in rock volume per unit volume per unit change in pressure. For these analyses, rock compressibility with

the following values – 2×10^{-6} psi⁻¹, 6×10^{-6} psi⁻¹, 8×10^{-6} psi⁻¹ and 4×10^{-6} psi⁻¹ (basecase) were considered. The depletion of reservoir pressure during production causes the volume of reservoir rocks to expand. Therefore, the higher the rock compressibility, the larger

the cumulative oil production and vice versa. At much higher rock compressibility values, there is a possibility that high gas saturation may impede oil production later during production, especially for highly volatile oils.

The impact of rock compressibility on producing GOR (for the values considered) is not significant. The trends are generally similar and the

higher the rock compressibility, the lower the producing GOR with time. It is likely that the impact of high gas saturation may alter the pattern of producing GOR at much higher rock compressibility values. Figures 35 and 36 show the effects of rock compressibility on producing GOR (semi-log plots) for Fluids 1, 4, 7 and 10.

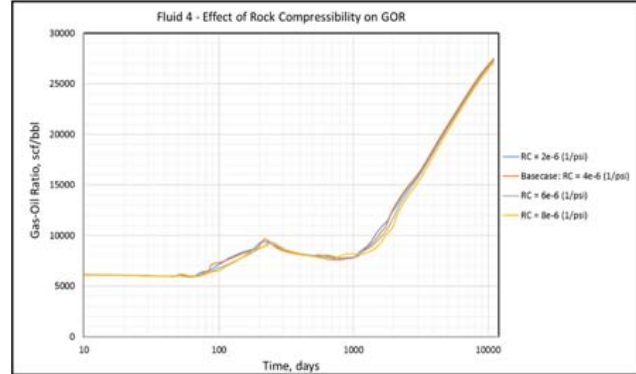
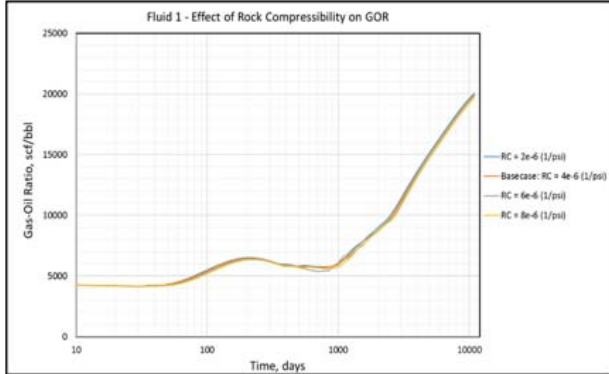


Figure 35: Fluids 1&4 – Effect of Rock Compressibility on GOR

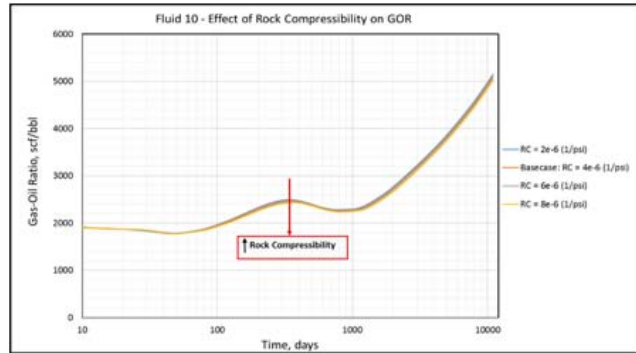
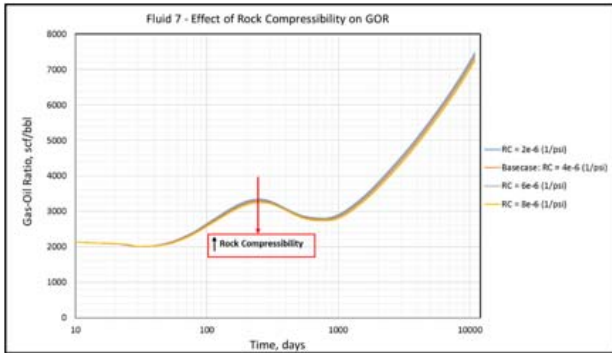


Figure 36: Fluids 7&10 – Effects of Rock Compressibility on GOR

XII. COMPACTION

Compaction is the consolidation of sediments that results in the cementing or packing together of grains. Compaction may lead to subsidence i.e. sediment loading and removal of fluids from the reservoir. It can have a tremendous impact on well performance. For the basecase reservoir model, compaction was not included. However, here, the

effects of compaction on shale volatile oil well production performance were investigated. Cases of weak compaction (constant rock compressibility of 4×10^{-6} psi⁻¹), mild compaction (constant rock compressibility of 20×10^{-6} psi⁻¹) and strong compaction (with the use of pressure-dependent compaction table shown in Table 3) were examined in the reservoir model. All the results were compared together with the basecase (no compaction) results.

Table 3: Pressure-Dependent Compaction Table

Reservoir Pressure, psi	Porosity Multiplier	Horizontal Permeability Multiplier	Vertical Permeability Multiplier
1,000	0.2	0.2	0.2
1,500	0.2	0.2	0.2
2,000	0.2	0.2	0.2
2,500	0.2	0.2	0.2
3,000	0.4	0.4	0.4
3,500	0.6	0.6	0.6
4,000	0.8	0.8	0.8
4,500	0.9	0.9	0.9
5,000	1.0	1.0	1.0

As reservoir pressure depletion occurs during production, compaction increases the pressure on the rocks (net confining pressure) due to the weight of the overlying sediments (overburden) and the pore fluid pressure decreases. This increase in net confining pressure can lead to collapse of pore spaces and thus, efficient expulsion of hydrocarbons can take place. Though compaction leads to reduction of porosity and permeability, strong compaction can enhance oil recovery significantly. The stronger the compaction, the larger the cumulative oil production. Weak compaction may lead to slight reduction in cumulative oil production (slightly smaller oil production than the basecases). This is because the slight reduction in porosity and permeability caused by weak compaction overrides the major compaction effect in this instance. Mild compaction leads to more oil production than the basecases and strong compaction results in the largest cumulative oil production. A similar result was obtained

by Khoshghadam et al. (2015) in their study of the impact of confined pore spaces on liquid-rich shale reservoir performance.

Weak compaction has little or no effect on producing GOR with time. For most cases, it is approximately identical to the basecases (no compaction). Mild compaction results in the reduction of producing GOR with time as more oil is produced in this case. For the cases with strong compaction, producing GOR remains approximately constant throughout the production period. This is because strong compaction keeps the average reservoir pressure so high that it never depletes beyond the saturation pressure. Also, large quantities of oil were produced due to strong compaction. Figures 37 to 40 portray the impacts of compaction on producing GOR (semi-log plots) and cumulative oil production for Fluids 1, 4, 7 and 10.

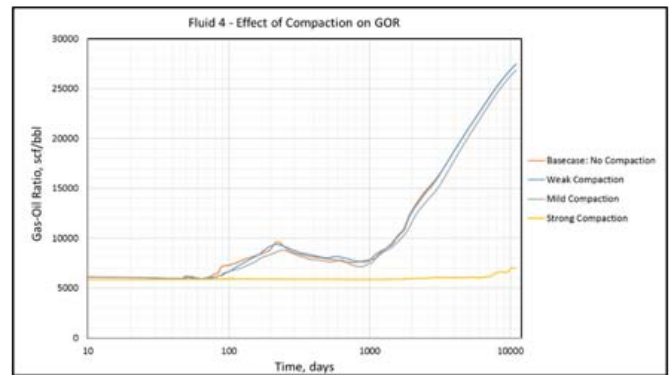
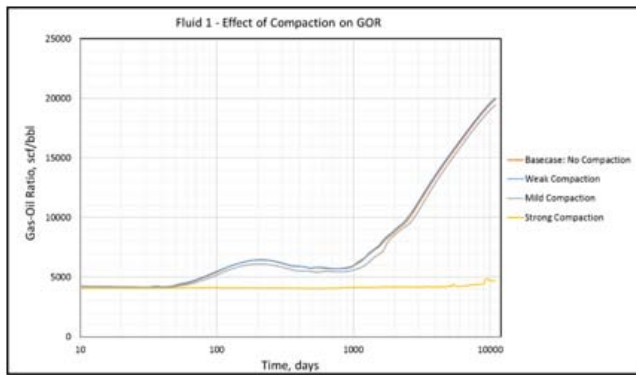


Figure 37: Fluids 1&4 – Effect of Compaction on GOR

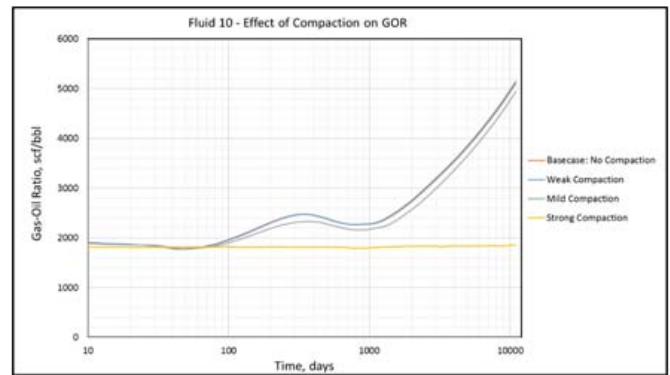
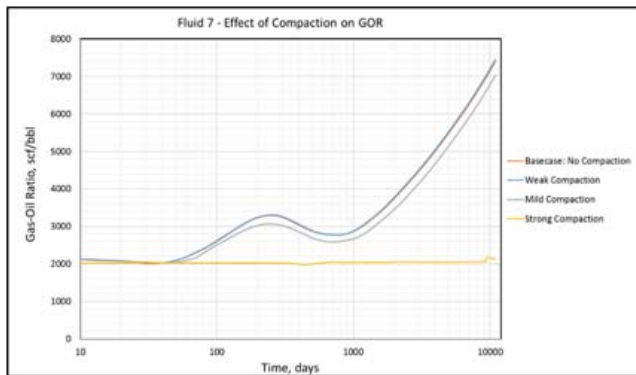


Figure 38: Fluids 7&10 – Effect of Compaction on GOR

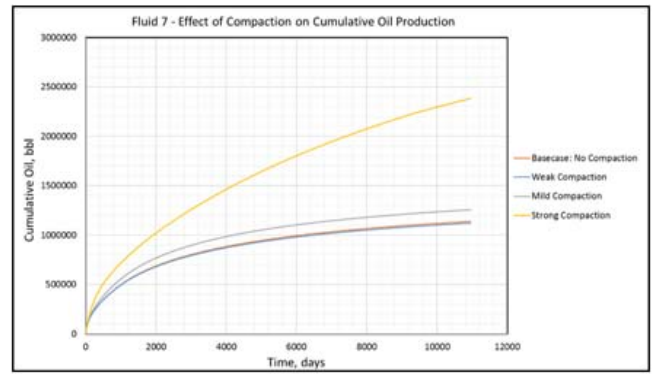
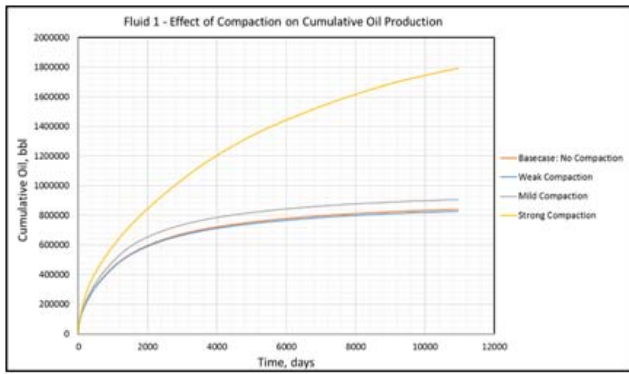


Figure 39: Fluids 1&7 – Effect of Compaction on Cumulative Oil Production

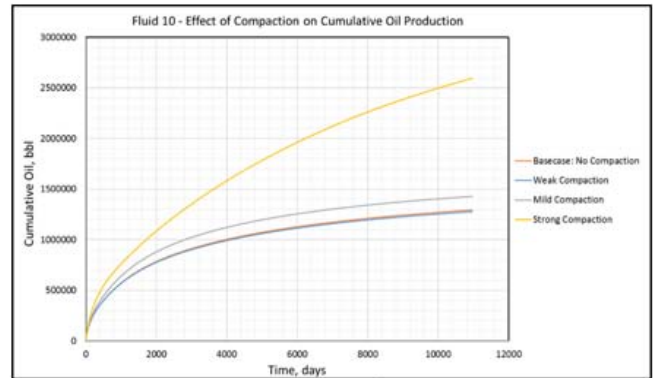
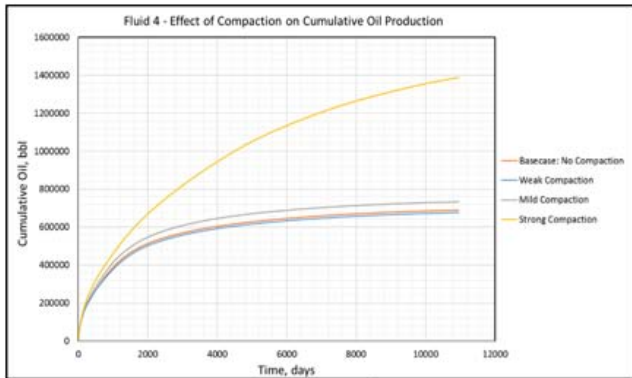


Figure 40: Fluids 4&10 – Effect of Compaction on Cumulative Oil Production

XIII. CONCLUSIONS

1. Production mechanisms of liquid-rich shale (LRS) reservoirs may be influenced by factors or combination of factors that include reservoir and fluid characteristics, as well as vital completion parameters;
2. Six critical stages in the GOR history of shale volatile oil reservoirs producing by solution gas drive mechanism were identified;
3. The degree of volatility of volatile oils as well as gas saturation may have substantial impact on the production performance and behavior of producing gas-oil ratio in shale volatile oil reservoirs;
4. Sensitivity analyses have proven that the flowing bottomhole pressure, rock compressibility, fracture half-length, fracture spacing and fracture permeability are important parameters that affect GOR behavior liquid-rich shale reservoirs;
5. The degree of undersaturation of shale volatile oil reservoirs is a major factor that influences the GOR behavior of LRS reservoirs. With decreasing degree of undersaturation, the producing GOR increases with time and vice versa.
6. Production performance of multi-fractured horizontal wells in a LRS reservoir is hugely influenced by the drainage area. The nature of the rate-time diagnostic plots are dependent on the geometry of

the drainage area. This, in turn, affects the GOR behavior of LRS reservoirs.

7. The presence of compaction can play a significant role in the production mechanisms of LRS reservoirs. Strong compaction may lead to considerably enhanced oil recovery;
8. The following factors or combination of these factors may lead to constant GOR or prolonged periods of constant GOR during production of LRS reservoirs:
 - High degree of undersaturation;
 - Flowing bottomhole pressure equal to or approximately equal to the fluid saturation pressure;
 - The presence of strong compaction.

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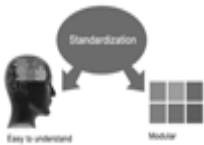
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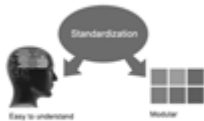
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30. Think and then print: When you will go to print your paper, notice that tables are not be split, headings are not detached from their descriptions, and page sequence is maintained.

31. Adding unnecessary information: Do not add unnecessary information, like, I have used MS Excel to draw graph. Do not add irrelevant and inappropriate material. These all will create superfluous. Foreign terminology and phrases are not apropos. One should NEVER take a broad view. Analogy in script is like feathers on a snake. Not at all use a large word when a very small one would be sufficient. Use words properly, regardless of how others use them. Remove quotations. Puns are for kids, not grunt readers. Amplification is a billion times of inferior quality than sarcasm.

32. Never oversimplify everything: To add material in your research paper, never go for oversimplification. This will definitely irritate the evaluator. Be more or less specific. Also too, by no means, ever use rhythmic redundancies. Contractions aren't essential and shouldn't be there used. Comparisons are as terrible as clichés. Give up ampersands and abbreviations, and so on. Remove commas, that are, not necessary. Parenthetical words however should be together with this in commas. Understatement is all the time the complete best way to put onward earth-shaking thoughts. Give a detailed literary review.

33. Report concluded results: Use concluded results. From raw data, filter the results and then conclude your studies based on measurements and observations taken. Significant figures and appropriate number of decimal places should be used. Parenthetical remarks are prohibitive. Proofread carefully at final stage. In the end give outline to your arguments. Spot out perspectives of further study of this subject. Justify your conclusion by at the bottom of them with sufficient justifications and examples.

34. After conclusion: Once you have concluded your research, the next most important step is to present your findings. Presentation is extremely important as it is the definite medium through which your research is going to be in print to the rest of the crowd. Care should be taken to categorize your thoughts well and present them in a logical and neat manner. A good quality research paper format is essential because it serves to highlight your research paper and bring to light all necessary aspects in your research.

INFORMAL GUIDELINES OF RESEARCH PAPER WRITING

Key points to remember:

- Submit all work in its final form.
- Write your paper in the form, which is presented in the guidelines using the template.
- Please note the criterion for grading the final paper by peer-reviewers.

Final Points:

A purpose of organizing a research paper is to let people to interpret your effort selectively. The journal requires the following sections, submitted in the order listed, each section to start on a new page.

The introduction will be compiled from reference matter and will reflect the design processes or outline of basis that direct you to make study. As you will carry out the process of study, the method and process section will be constructed as like that. The result segment will show related statistics in nearly sequential order and will direct the reviewers next to the similar intellectual paths throughout the data that you took to carry out your study. The discussion section will provide understanding of the data and projections as to the implication of the results. The use of good quality references all through the paper will give the effort trustworthiness by representing an alertness of prior workings.



Writing a research paper is not an easy job no matter how trouble-free the actual research or concept. Practice, excellent preparation, and controlled record keeping are the only means to make straightforward the progression.

General style:

Specific editorial column necessities for compliance of a manuscript will always take over from directions in these general guidelines.

To make a paper clear

- Adhere to recommended page limits

Mistakes to evade

- Insertion a title at the foot of a page with the subsequent text on the next page
- Separating a table/chart or figure - impound each figure/table to a single page
- Submitting a manuscript with pages out of sequence

In every sections of your document

- Use standard writing style including articles ("a", "the," etc.)
- Keep on paying attention on the research topic of the paper
- Use paragraphs to split each significant point (excluding for the abstract)
- Align the primary line of each section
- Present your points in sound order
- Use present tense to report well accepted
- Use past tense to describe specific results
- Shun familiar wording, don't address the reviewer directly, and don't use slang, slang language, or superlatives
- Shun use of extra pictures - include only those figures essential to presenting results

Title Page:

Choose a revealing title. It should be short. It should not have non-standard acronyms or abbreviations. It should not exceed two printed lines. It should include the name(s) and address (es) of all authors.



Abstract:

The summary should be two hundred words or less. It should briefly and clearly explain the key findings reported in the manuscript-- must have precise statistics. It should not have abnormal acronyms or abbreviations. It should be logical in itself. Shun citing references at this point.

An abstract is a brief distinct paragraph summary of finished work or work in development. In a minute or less a reviewer can be taught the foundation behind the study, common approach to the problem, relevant results, and significant conclusions or new questions.

Write your summary when your paper is completed because how can you write the summary of anything which is not yet written? Wealth of terminology is very essential in abstract. Yet, use comprehensive sentences and do not let go readability for brevity. You can maintain it succinct by phrasing sentences so that they provide more than lone rationale. The author can at this moment go straight to shortening the outcome. Sum up the study, with the subsequent elements in any summary. Try to maintain the initial two items to no more than one ruling each.

- Reason of the study - theory, overall issue, purpose
- Fundamental goal
- To the point depiction of the research
- Consequences, including definite statistics - if the consequences are quantitative in nature, account quantitative data; results of any numerical analysis should be reported
- Significant conclusions or questions that track from the research(es)

Approach:

- Single section, and succinct
- As an outline of job done, it is always written in past tense
- A conceptual should situate on its own, and not submit to any other part of the paper such as a form or table
- Center on shortening results - bound background information to a verdict or two, if completely necessary
- What you account in an abstract must be regular with what you reported in the manuscript
- Exact spelling, clearness of sentences and phrases, and appropriate reporting of quantities (proper units, important statistics) are just as significant in an abstract as they are anywhere else

Introduction:

The **Introduction** should "introduce" the manuscript. The reviewer should be presented with sufficient background information to be capable to comprehend and calculate the purpose of your study without having to submit to other works. The basis for the study should be offered. Give most important references but shun difficult to make a comprehensive appraisal of the topic. In the introduction, describe the problem visibly. If the problem is not acknowledged in a logical, reasonable way, the reviewer will have no attention in your result. Speak in common terms about techniques used to explain the problem, if needed, but do not present any particulars about the protocols here. Following approach can create a valuable beginning:

- Explain the value (significance) of the study
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- Present a justification. State your particular theory (es) or aim(s), and describe the logic that led you to choose them.
- Very for a short time explain the tentative propose and how it skilled the declared objectives.

Approach:

- Use past tense except for when referring to recognized facts. After all, the manuscript will be submitted after the entire job is done.
- Sort out your thoughts; manufacture one key point with every section. If you make the four points listed above, you will need a least of four paragraphs.



- Present surroundings information only as desirable in order hold up a situation. The reviewer does not desire to read the whole thing you know about a topic.
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Materials:

- Explain materials individually only if the study is so complex that it saves liberty this way.
- Embrace particular materials, and any tools or provisions that are not frequently found in laboratories.
- Do not take in frequently found.
- If use of a definite type of tools.
- Materials may be reported in a part section or else they may be recognized along with your measures.

Methods:

- Report the method (not particulars of each process that engaged the same methodology)
- Describe the method entirely
- To be succinct, present methods under headings dedicated to specific dealings or groups of measures
- Simplify - details how procedures were completed not how they were exclusively performed on a particular day.
- If well known procedures were used, account the procedure by name, possibly with reference, and that's all.

Approach:

- It is embarrassed or not possible to use vigorous voice when documenting methods with no using first person, which would focus the reviewer's interest on the researcher rather than the job. As a result when script up the methods most authors use third person passive voice.
- Use standard style in this and in every other part of the paper - avoid familiar lists, and use full sentences.

What to keep away from

- Resources and methods are not a set of information.
- Skip all descriptive information and surroundings - save it for the argument.
- Leave out information that is immaterial to a third party.

Results:

The principle of a results segment is to present and demonstrate your conclusion. Create this part a entirely objective details of the outcome, and save all understanding for the discussion.

The page length of this segment is set by the sum and types of data to be reported. Carry on to be to the point, by means of statistics and tables, if suitable, to present consequences most efficiently. You must obviously differentiate material that would usually be incorporated in a study editorial from any unprocessed data or additional appendix matter that would not be available. In fact, such matter should not be submitted at all except requested by the instructor.



Content

- Sum up your conclusion in text and demonstrate them, if suitable, with figures and tables.
- In manuscript, explain each of your consequences, point the reader to remarks that are most appropriate.
- Present a background, such as by describing the question that was addressed by creation an exacting study.
- Explain results of control experiments and comprise remarks that are not accessible in a prescribed figure or table, if appropriate.
- Examine your data, then prepare the analyzed (transformed) data in the form of a figure (graph), table, or in manuscript form.

What to stay away from

- Do not discuss or infer your outcome, report surroundings information, or try to explain anything.
- Not at all, take in raw data or intermediate calculations in a research manuscript.
- Do not present the similar data more than once.
- Manuscript should complement any figures or tables, not duplicate the identical information.
- Never confuse figures with tables - there is a difference.

Approach

- As forever, use past tense when you submit to your results, and put the whole thing in a reasonable order.
- Put figures and tables, appropriately numbered, in order at the end of the report
- If you desire, you may place your figures and tables properly within the text of your results part.

Figures and tables

- If you put figures and tables at the end of the details, make certain that they are visibly distinguished from any attach appendix materials, such as raw facts
- Despite of position, each figure must be numbered one after the other and complete with subtitle
- In spite of position, each table must be titled, numbered one after the other and complete with heading
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The Discussion is expected the trickiest segment to write and describe. A lot of papers submitted for journal are discarded based on problems with the Discussion. There is no head of state for how long a argument should be. Position your understanding of the outcome visibly to lead the reviewer through your conclusions, and then finish the paper with a summing up of the implication of the study. The purpose here is to offer an understanding of your results and hold up for all of your conclusions, using facts from your research and generally accepted information, if suitable. The implication of result should be visibly described. Infer your data in the conversation in suitable depth. This means that when you clarify an observable fact you must explain mechanisms that may account for the observation. If your results vary from your prospect, make clear why that may have happened. If your results agree, then explain the theory that the proof supported. It is never suitable to just state that the data approved with prospect, and let it drop at that.

- Make a decision if each premise is supported, discarded, or if you cannot make a conclusion with assurance. Do not just dismiss a study or part of a study as "uncertain."
- Research papers are not acknowledged if the work is imperfect. Draw what conclusions you can based upon the results that you have, and take care of the study as a finished work
- You may propose future guidelines, such as how the experiment might be personalized to accomplish a new idea.
- Give details all of your remarks as much as possible, focus on mechanisms.
- Make a decision if the tentative design sufficiently addressed the theory, and whether or not it was correctly restricted.
- Try to present substitute explanations if sensible alternatives be present.
- One research will not counter an overall question, so maintain the large picture in mind, where do you go next? The best studies unlock new avenues of study. What questions remain?
- Recommendations for detailed papers will offer supplementary suggestions.

Approach:

- When you refer to information, differentiate data generated by your own studies from available information
- Submit to work done by specific persons (including you) in past tense.
- Submit to generally acknowledged facts and main beliefs in present tense.



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<i>Introduction</i>	Containing all background details with clear goal and appropriate details, flow specification, no grammar and spelling mistake, well organized sentence and paragraph, reference cited	Unclear and confusing data, appropriate format, grammar and spelling errors with unorganized matter	Out of place depth and content, hazy format
<i>Methods and Procedures</i>	Clear and to the point with well arranged paragraph, precision and accuracy of facts and figures, well organized subheads	Difficult to comprehend with embarrassed text, too much explanation but completed	Incorrect and unorganized structure with hazy meaning
<i>Result</i>	Well organized, Clear and specific, Correct units with precision, correct data, well structuring of paragraph, no grammar and spelling mistake	Complete and embarrassed text, difficult to comprehend	Irregular format with wrong facts and figures
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<i>References</i>	Complete and correct format, well organized	Beside the point, Incomplete	Wrong format and structuring



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