

## A Novel Experimental Setup to Analyze Model Thin Films Representing Cores for an Ultrasonic Radiation Study of Petroleum Reservoirs

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### Abstract

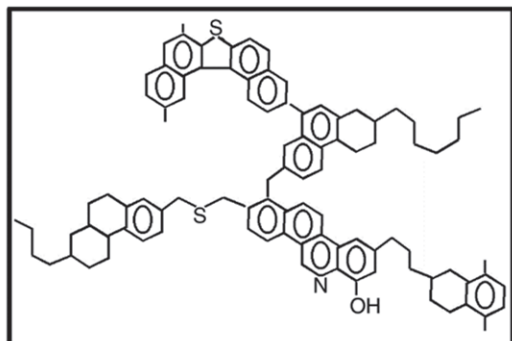
An apparatus was developed for a visual representation of conventional core flooding tests using a Model Thin Film (MTF) setup. The configuration was intended to provide direct visual representation of a flooding process. For our purposes, we investigated asphaltene deposition on a thin-film core sample, by evaluating the oil recovery before and after subjecting rock samples to a sonication process to remove asphaltene deposits, part of an ongoing project. The process involved saturating a specific volume of core sample with an asphaltic crude oil sample and recording flow pressures throughout the process. In order to have a full grade asphaltene deposition on the core sample, an alkane reagent, heptane, was used as a solvent to subsequently flood the rock system. After the formation of the skin and asphaltic sediments, we conducted an oil flood and monitored flow pressures, higher inlet pressures confirmed plugging and asphaltic deposition in the rock matrix. The model thin film setup proved to be a very good demonstrational and experimental apparatus, as it provided excellent visual information relating to the oil flood, and allowed routine experimental pressure, temperature and flow readings to be taken. The prospect of obtaining accurate experimental results from the model thin film is bright. This apparatus is designed to be used for the ultrasonic radiation study of petroleum reservoirs.

**Key words:** Model Thin Film (MTF); Oil recovery; Sonication process; Asphaltene deposits

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### INTRODUCTION

Asphaltenes are molecular substances that are found in crude oil, along with resins, aromatic hydrocarbons, and saturates (i.e. saturated hydrocarbons such as alkanes). The word “asphaltene” was coined by Boussingault in 1837 when he noticed that the distillation residue of some bitumens had asphalt-like properties<sup>[1]</sup>. Asphaltenes are a group of compounds, not a single compound, concentrated in the high-temperature distillation residue of petroleum (> 530 °C). Other components are heavy oils, resins and high-molecular-weight waxes<sup>[2]</sup>. Resins and asphaltenes primarily are a subclass of the aromatics, although some resins may contain only naphthenic rings. They are large molecules consisting primarily of hydrogen and carbon, with one to three sulfur, oxygen, or nitrogen atoms per molecule. The basic structure is composed of rings, mainly aromatics, with three to ten or more rings per molecule<sup>[3]</sup>. Asphaltenes are a solubility class and are usually defined as the fraction of a crude oil that precipitates in aliphatic solvent (typically n-pentane or n-heptane) yet remains soluble in toluene. Asphaltenes are the most aromatic and polar fraction of crude oil and have the largest heteroatom and metal content. They consist of a variety of molecular species with molar masses of at least 1,000 g/mol<sup>[4]</sup>. An example of an asphaltene molecule is given in Figure 1<sup>[5]</sup>.



**Figure 1**  
**Hypothetical/Generic Structure of an Asphaltene**

## 1. ASPHALTENE DEPOSITION

Production of heavy oils and paraffinic crude reserves often results in the deposition of organic solids, typically waxes or asphaltenes. The organic deposits can reduce the productivity of the reservoir as well as foul piping and surface equipment. Current chemical and mechanical methods for treating deposition are only partially effective partly because the deposition process is poorly understood<sup>[4]</sup>. These deposits may plug the wellbore tubing and valves, as well as coat surface safety and process control equipment<sup>[6]</sup>. Asphaltenes can also accumulate in separators and in pipelines causing problems in addition to the plugging of the formation, wellbore and facilities<sup>[7]</sup>. Asphaltene precipitation also occurs frequently during enhanced-oilrecovery by gas injection which seriously impedes hydrocarbon recovery<sup>[8]</sup>.

The tendencies of crudes to deposit asphaltenes do not correlate with the quantity of dissolved asphaltenes present in the reservoir fluid. Some oils with 1% asphaltene or less will form deposits in tubulars, while others with 10% or more asphaltenes will form no deposits. Asphaltene chemistry varies with the field. Asphaltenes contained in oil from a well in the North Sea are chemically different from asphaltenes found in the Venezuela fields, or another North Sea well. The chemistry controlling these depositions is not well defined, underscoring the need to study this further<sup>[4]</sup>. Nevertheless, some generalities are possible, which can aid in the design of prevention and remediation technology for a given well or field<sup>[7]</sup>.

Currently, mechanical and chemical cleaning methods of wellbores are being improvised to maintain production, but these methods are time-consuming and expensive, and fail to address problems with asphaltic deposition in surface flowlines and facilities<sup>[9]</sup>. Generally, cleaning methods involve employing large amounts of cleaning fluids into the wellbore either through coiled tubing or standard pumps. After the cleaning process, it is then attempted to circulate this fluid out of the wellbore, or to minimize formation damage from this fluid. Advanced methods have developed that mitigate fluid-handling problems such as using compressed air or ultrasonic

radiation<sup>[9]</sup>. Thus, our model thin film method was employed to investigate the effectiveness of ultrasonic radiation on asphaltene-removal, which is an ongoing project. Discussion of the results of the ultrasonic investigation is beyond the scope of this paper. The subsequent descriptions of the experimental set up and apparatus intend to present a clear idea of how the model thin film apparatus is constituted into an investigation.

## 2. ROLE OF ULTRASONIC RADIATION

Various authors have proven the usefulness of ultrasonic radiation to remove and/or mobilize asphaltenes, both on man-made equipment and in formations. Najafi and Amani (2011) studied the governing factors behind asphaltene-flocculation prevention under the influence of ultrasonic waves. Shedid et al. (2004) investigated asphaltene behavior post-deposition, stimulated by ultrasonic radiation. The results showed that subjection of the crude oil to ultrasonic radiation decreases the size asphaltene clusters<sup>[10]</sup>. Consequently, this effect increases the suspension of asphaltene in the crude oil and reduces/prevents its tendency to precipitate at 10 minutes or more ultrasonic radiation time<sup>[11]</sup>. To this purpose, an ultrasonic generator and reactor will be used for the sonication of the model thin-film core sample in future experiments. The generator can house both conventional core samples and the model thin film apparatus, with equally effective sonication results achieved.



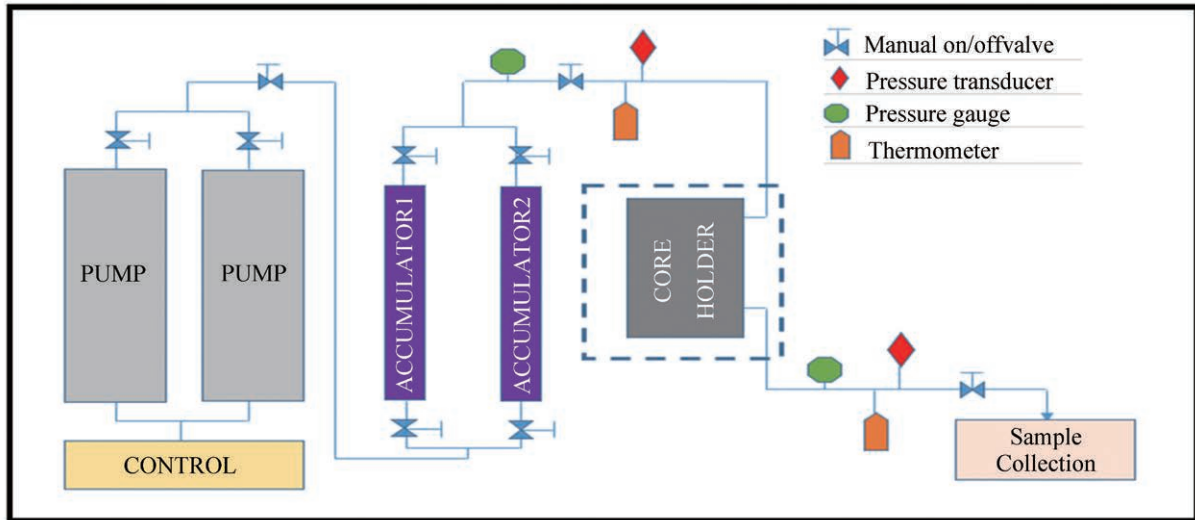
**Figure 2**  
**Ultrasonic Generator and Reactor**

## 3. APPARATUS PREPARATION

The setup consists of two main parts. The first is with the workstation which houses the dosing system for heptane (to aid asphaltene deposition), crude oil (field sample) and brine. The second is with the core flooding assembly. This setup is also meant to be used for conventional core flood experiments and other research experiments. Specifically for the model thin-film experiment, the designed experimental setup allowed a visual representation of the actual core flooding. The overall process workflow

for the experimental setup with the core holder, pumps, accumulators, and gauge points are illustrated in (Figure

3). The core holder can be replaced with the Model Thin Film spill basin setup depending on the required test.



**Figure 3**  
**Model Thin-Film Core Flooding Experimental Set-Up**

### 3.1 Why Model Thin Film?

Core tests have become an industry standard. They are often the basis and preliminary measurements used toward determining essential petrophysical and reservoir parameters such as porosity, permeability, grain-size distribution, rock type and many more. The issue with these core tests is that they are carried out in a sealed cell, often never seeing the light of day from collection to disposal. Managers and engineers who make decisions from data derived from these cores often never even see the cores in question<sup>[12]</sup>. In day to day professional activity, it is easy to lose touch with fundamental concepts, which is even more so a concern for managers and decision-makers who are not trained in the petroleum engineering profession or have strictly a business background. For such purposes, a visual method of displaying a recovery process, a new technology, or the fundamental processes by which rock is saturated with fluids is extremely beneficial. A real, physical and visual representation of what is or will happen in the subsurface can be a very efficient method to help understand, investigate and experiment with these concepts<sup>[13]</sup>. The most natural way to convey information to the human brain is visually, as half of our neurons are dedicated to visualization. Naturally, it follows that decision-makers and scientists should be able to visually and physically observe the processes around which their careers revolve.

Our thin film model is essentially a core test within a transparent cell, that allows us to physically observe the rock being imbibed and drained by a fluid. This process can be photographed or video-recorded in order to visually demonstrate the effectiveness of sonication in

removing asphaltene deposits. The opaque, high pressure cells that are used for conventional core tests are replaced with transparent, acrylic sheets on either side of the Model Thin Film (MTF) rock sample, and are sealed on either side with silicon epoxy (also transparent). The capability of this acrylic to withstand pressure is much lower than a conventional core holder. However, due to the relatively small dimensions of the MTF sample significantly lower pressure thresholds are required. This is because the MTF is effectively a modelled thin slice, or “film” of the rock core, hence the name Model Thin Film. Nevertheless, the acrylic housing was observed to be able to withstand pressures greater than 2,200 psi, which is much greater than most rocks’ pore pressure and sufficient for our model’s scope and purposes. It is important to note here that despite the fact that our MTF setup was used to demonstrate and investigate the effects of sonication on the removal of asphaltene deposits, the setup has a great number of additional functions and processes that it can be used to demonstrate and investigate.

### 3.2 Workstation and Dosing System Setup

The workstation and dosing system setup to be used in the investigation was designed and build by our team is shown in the photo below. The workstation holds the dosing pump and two accumulators that store the crude oil and n-heptane. The pump that is being used in the project is the ISCO Pump which is capable of discharging 1 mL/minute up to 106 mL/minute at a maximum pressure of 2,000 psi. The pump is operated and set at specific pressures by a separate, electronic workstation. Please refer to Figure 3 for the process workflow represented by the photograph in (Figure 4).





**Figure 4**  
**Actual Workstation/Dosing System**

The discharge line from the workstation is connected to the inlet of the model thin-film core flooding system, or the core holder for the conventional core flood experiments. There is a separate, manually operated pump system (not pictured) that provides hydraulic pressure applied to the core. As with all core testing apparatus, the outlets are led to fluid collection containers. The manually operated pump serves to provide overburden pressure to the cells. Note here that this set up may be used to inject surfactant or acid for other investigations.

### 3.3 Model Thin-Film Core Flooding System

As per previous design, the thin-film core flooding housing was made tight so that the excess amount of leakage during the operation is reduced if not eliminated. It was made of 10 mm thick, acrylic bonded with silicon

resin and secured by 4 bolted screws. The chosen acrylic is transparent for visualization purposes. The actual process was operated inside the glass container which serves as a spill basin. On both ends, each pipeline was attached directly to the core and then sealed again by the silicon resin. The actual core flooding system and core housing is shown in the figures below. The system is equipped with real-time pressure and temperature gauges both in the inlet and the outlet of the sample housing. These gauges and their accompanying software can be set to take periodic or continuous analogue readings. For our purposes, we set pressure data points to be taken every 30 seconds. Any number of additional gauges can be installed depending on the application and load of data measurements required.



**Figure 5**  
**Model Thin-Film Core Flooding System (Inlet and Outlet)**



**Figure 6**  
**Model Thin-Film Housing**

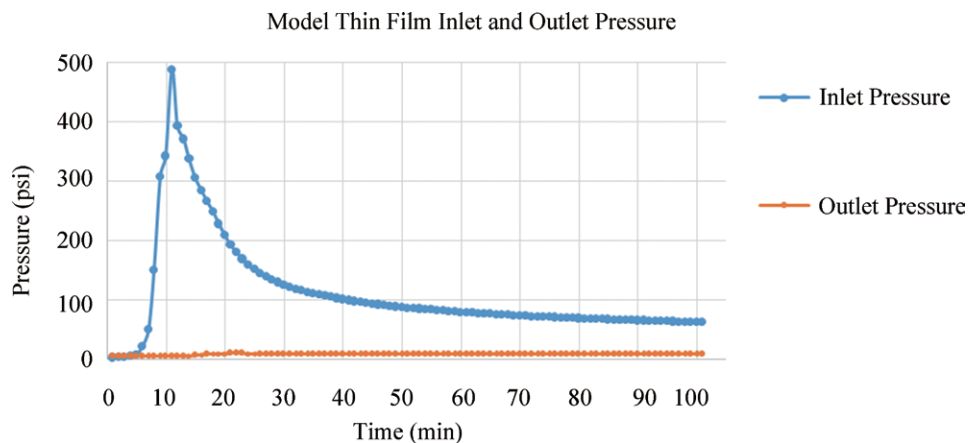
#### 4. MODEL THIN-FILM FLOODING

A trial run was conducted on an existing, previously prepared thin-film core sample. The valves for the oil accumulator were open to the core flooding system and the pump was operated at an injection rate of 1 ml/minute. The inlet pressure built to 1,500 psi, indicating a plugging issue in the flow lines.

Another test was conducted after replacing the core flooding housing inlet tubing. The same procedure as the first trial was applied to the second run. Flooding begins to be visible on the core sample. It takes the accumulator and dosing system 5 minutes to build up sufficient pressure prior to flow within the model thin film set up. After 12 minutes the system pressure drops as leaks begin to develop in the surrounding epoxy. Figure 7 shows the sandstone core sample used in the second model thin film flood. Figure 8 shows the pressure profile of the second trial run.



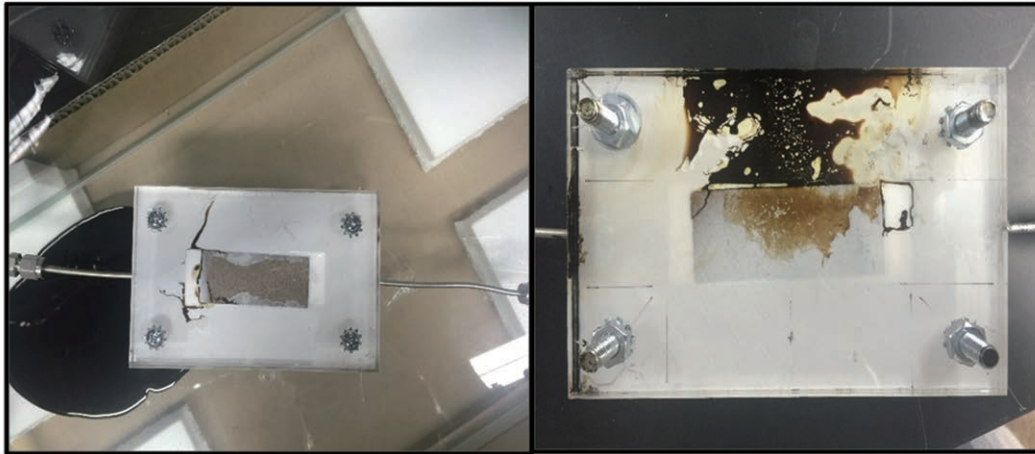
**Figure 7**  
**Trial Run Flooded Core Sample**



**Figure 8**  
**Pressure Profile of Model Thin Film Run**

During the second run, leaks developed due to sealing issues with the surrounding epoxy. As can be seen from Chart 1, a leak developed at approximately 500 psi, which is far below the fracture pressure of the sandstone. This underscores the importance of the need to select the right epoxy; the epoxy used in the second run was of

standard mechanical strength. The subsequent tests were run with industrial strength epoxy, effectively remedying the leaking issues. Figure 9 below shows the leak for the top and bottom of the core flooding housing. Inspections revealed issues with the epoxy sealant, as the pressurized oil chooses pathways of least resistance out of the cell.

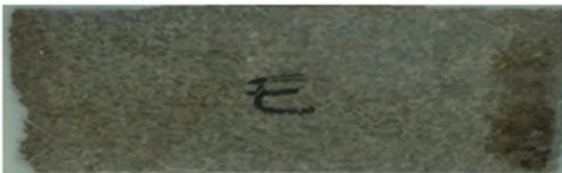
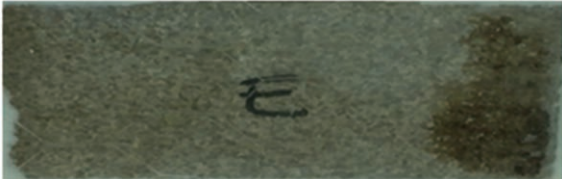
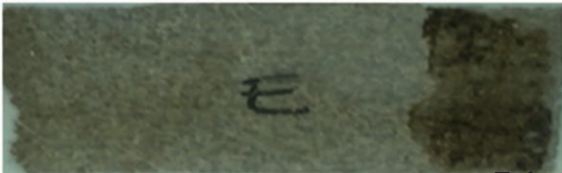


**Figure 9**  
**Top and Bottom View of the Core Flooding Housing**

More trials were performed on the model thin-film setup until a proper visual representation of the flooding was achieved. Table 1 shows the progression of the flooding along the plane of the MTF cell. The flooding was achieved at a flow rate of 1 ml/min and a constant pressure of 400 psi. We can clearly observe the oil front progress through the rock medium, the video (from which these images are acquired for time-lapse) would show very clearly and accurately the progression of the flooding phase. This clear visual exemplifies how effectively fluids and saturation processes can be observed in the model thin film setup. The implications of this clarity are even more profound in applications where surfactant, water

or carbon dioxide floods are being investigated and employed. Observing the fluid in question visually will allow very accurate determination of flow characteristics, simulation inputs, and further porous media investigations such as relative permeability and saturation end-points<sup>[14]</sup>. Transparent liquids can be stained with simple non-reactive dyes to be able to visually observe the front progression. Educational settings also have a very impactful application, young engineers who can visually understand the flooding and saturation process from an early point in their education will no doubt have an easier time with further studies of detailed petrophysical concepts and fundamentals.

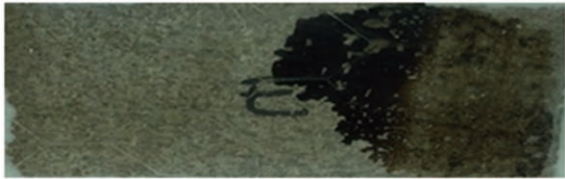
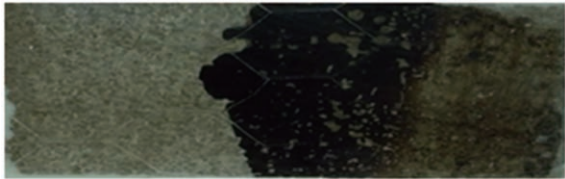
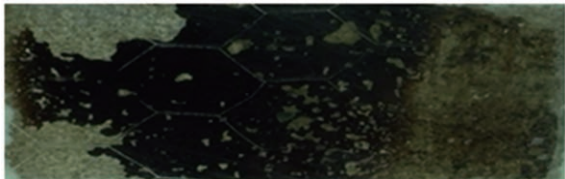
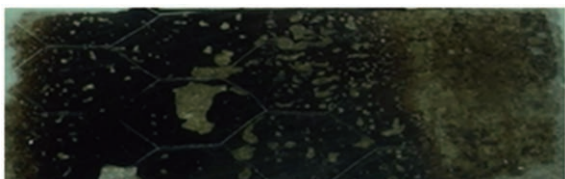
**Table 1**  
**Flooding Sequence of First Model Thin Film Run**

Sequence	Time ( minutes )	Appearance
1	1	
2	3	
3	5	

To be continued



Continued

Sequence	Time ( minutes )	Appearance
4	7	
5	9	
6	11	
7	13	

## CONCLUSION

The model thin film approach has provided excellent visual and experimental results so far in the investigation. Compared to conventional core tests that are conducted in sealed cells, the model thin film design can be an excellent alternative. Not only does it provide visual confirmation of the fundamental flooding and saturation processes upon which such a large portion of the hydrocarbon industry is based, it provides comparable levels of experimental functionality, in terms of flow rates and pressures. Ample pressure and flow readings were taken, allowing further petrophysical properties to be easily calculated and determined in ways exactly identical to conventional core tests. Visually observing an oil flood in the rock gives an idea of the process that can hardly be rivalled by numerical modeling or simulation.

The visual clarity also has applications beyond the scope of the authors' specific asphaltene project, such as in the education of young engineers. Apparatus such as the model thin film setup are excellent examples of safe, low-cost educational instruments that allows the visual observation of such an essential process. The model thin film coupled with magnifying lens, lighting and/or

recording equipment can also give rise to highly detailed study of the flow behavior of a range of fluids through the rock matrix. If a reactive brine is injected in a carbonate model thin film apparatus, the dissolution process can be studied in great detail, shedding more light on worm holing processes in acid stimulation, also allowing recording of the process and to be replayed as many times as required and at any number of playback speeds.

The good functionality of the model thin film apparatus has provided the authors with a bright prospect. The ongoing project concerning sonication and removal of asphaltene deposits can be sufficiently investigated using the model thin film apparatus, as so far it has handled oil and heptane flood well. Sealing issues and flow obstacles have been removed and the model is now ready for sonication and further investigations in terms of asphaltene removal. The authors aim to use the model thin film set up instead of conventional core tests to demonstrate the effectiveness of sonication on asphaltene removal, with the hope that the same apparatus can be used to investigate other recovery and stimulation methods such as surfactant, carbon dioxide, acid and waterflood processes in the future.

## REFERENCES

- [1] Kokal, S. L., & Sayegh, S. G. (1995, March). *Asphaltenes: The cholesterol of petroleum*. Paper presented at Middle East Oil Show, Bahrain.
- [2] Speight, J. G., Yen, T. F., & Chilingarian, G. V. (1994). Asphaltenes and asphalts, 1. *Developments in Petroleum Science*, 40, 7.
- [3] Ellison, B. T., Gallagher, C. T., Frostman, L. M., & Lorimer, S. E. (2000, May). *The physical chemistry of wax, hydrates, and asphaltene*. Paper presented at Offshore Technology Conference, Houston, Texas.
- [4] Yarranton, H. (2000). *Asphaltene deposition*. Paper presented at Canadian International Petroleum Conference.
- [5] Cimimo, R., Corra, S., Del Bianco, A., & Lockhart, T. P. (1995). Solubility and phase behavior of asphaltenes in hydrocarbon media. In *Asphaltenes: Fundamentals and Applications* (pp.97-130). New York, USA: Plenum Press.
- [6] Hunt, M. J. (1996). *Petroleum Geochemistry and Geology*. New York, USA: W. Freeman and Co.
- [7] Thawer, R., Nicoll, D. C. A., & Dick, G. (1990). Asphaltene deposition in production facilities. *Society of Petroleum Engineers*, 5(4), 475-480.
- [8] Nghiem, L. X., Hassam, M. S., Nutakki, R., & George, A. E. D. (1993, October). *Efficient modelling of asphaltene precipitation*. Paper presented at SPE Annual Technical Conference and Exhibition, Houston, Texas.
- [9] Nabati-Shoghl, F., Saasen, A., Berg, E., & Jensen, B. (2000, June). *Ingenious solutions for well cleaning applying environmental engineering*. Paper presented at SPE International Conference on Health, Safety and Environment in Oil and Gas Exploration and Production, Stavanger, Norway.
- [10] Hamed Rad, M., Tavakolian, M., Najafi, I., Ghazanfari, M., Taghikhani, V., & Amani, M. (2013, June). Modeling the kinetics of asphaltene flocculation in toluene-pentane systems for the case of sonicated crude oils. *Scientific Iranica, Transaction C, Chemistry and Chemical Engineering*, 20(3), 611-616.
- [11] Shedid, S. A., & Attallah, S. R. (2004, February). *Influences of ultrasonic radiation on asphaltene behavior with and without solvent effects*. Paper presented at SPE International Symposium and Exhibition on Formation Damage Control, Lafayette, Louisiana.
- [12] Kidd, G. D. (1999). Fundamentals of 3D seismic volume visualization. *Leading Edge*, 18(6), 702-709.
- [13] Seller, D. (1995, June). *Borehole visualization in 3-D*. Paper presented at SPWLA 36th Annual Logging Symposium, Paris, France.
- [14] Dacy, J. M. (2010, September). *Core tests for relative permeability of unconventional gas reservoirs*. Paper presented at SPE Annual Technical Conference and Exhibition, Florence, Italy.
- [15] Nwagu, C., Awobadejo, T., & Gaskin, K. (2014, August). Application of mechanical cleaning device: Hole cleaning tubulars, to improve hole cleaning. Paper presented at SPE Nigeria Annual International Conference and Exhibition, Lagos, Nigeria.
- [16] Najafi, I., & Amani, M. (2011). A asphaltene flocculation inhibition with ultrasonic wave radiation: A detailed experimental study of the governing mechanisms. *Journal of Advances in Petroleum Exploration and Development*, 2(2), 32-36.