

EXPERIMENTAL INVESTIGATION OF ASPHALTENE DEPOSITION IN A TRANSPARENT MINI-CHANNEL

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ABSTRACT

One of the most recurring flow assurance problems in oil and gas industry is associated to the formation of organic and inorganic deposits in the wellbores and the near-wellbore regions. In particular, the depositions of asphaltene in wellbores represent both a major obstacle for petroleum engineers and a challenging topic for scientists. This paper focuses on experimental investigation of asphaltene deposition in transparent mini-channel. The working fluid is a mixture of heptane and crude oil. Induced by the addition of n-heptane, the dissolved asphaltene in crude oil precipitates to form asphaltene particles which deposit on the walls of the transparent mini-channel at ambient temperature. The thickness of asphaltene deposition is estimated using a visualization technique based on 3D microscopy. The thickness of the deposition layer is quantified and the two-dimensional profile of the deposition at selected axial section is measured. The obtained experimental results provide new insights into the deposition process in micro-scale and will be used to validate a developed numerical model.

INTRODUCTION

During oil production, transportation and processing, asphaltene deposition may result in blockages. This can occur in reservoir structure, wellbores, separators, pumps, pipelines, heat exchangers, tanks and other equipment leading to equipment downtime, equipment damage and even permanent damage to reservoirs. Asphaltene precipitation is also a very important aspect in understanding the effects of asphaltene on oil production, transportation and processing. Despite extensive study of asphaltene deposition in oil and gas equipment, many questions remain unanswered about the mechanism of asphaltene deposition in mini and micro scales. This paper describes a new approach to measure and quantify an asphaltene deposition distribution and profiles in mini-channels.

Over the last two decades, many experiments to study asphaltene depositions have been reported. These include, but

are not limited to, (i) experiments with stainless steel setups [1–6], (ii) experiments with glass setups [7–9]. These experiments were conducted using capillary tubes [1–4, 7–9] or concentric cylindrical setups [5, 6].

Broseta *et al.* [1] reported asphaltene deposition using flow in a capillary tube. Experiments were conducted using a 15-meter-long stainless steel with a nominal internal radius of 0.25mm. Pressure drops were measured and the deposition depths were back calculated from the increase in pressure drop as a result of asphaltene deposition. Wang *et al.* [2] carried out asphaltene precipitation and deposition experiments using a 0.5mm inner diameter stainless steel capillary tube. To allow depositions, the total length of the capillary of 32 m (100 ft) was employed. Three different oils with three different precipitants were studied. Nobzar *et al.* [3] conducted experiments using much smaller capillary tubes. The inner diameters of their experiments range from 0.065 mm to 0.133 mm with a tube length of 50 cm. Jamialahmadi *et al.* [4] carried out asphaltene deposition experiments using a “large” stainless steel pipe with 23.8 mm in inner diameter and 160 mm in length. This study differs from the earlier studies in two ways namely, (i) smaller axial length-to-diameter ratio and (ii) non-isothermal experiments; wall temperature higher than bulk fluid temperatures. As seen from the above experiments, large axial length-to-diameter ratios are needed to allow for asphaltene depositions. Zougari *et al.* [5] used a concentric cylinders setup to create Taylor-Couette flow. The experiments were performed using a batch of oil subjected to angular rotations. It was observed that deposition levels off with time. Akbarzadeh *et al.* [6] extended the experiments of Zougari *et al.* [5] by allowing for fresh fluid to flow into and out of the concentric cylinders setup. As the above experiments were performed using stainless steel setups, the deposition thicknesses and/or “profiles” were back calculated from available experimental measurements such as pressure drops.

To facilitate visualizations of asphaltene depositions, experiments using glass setups were carried out by Boek *et al.* [7, 8] with 100-mm long, 600 μm \times 150 μm rectangular mini-

channel. Lawal *et al.* [9] conducted asphaltene deposition experiments using 127-mm long cylindrical glass capillary with 0.32 mm inner diameter. The asphaltene deposition thicknesses were deduced from pressure drop measurements. Deposited asphaltene in the glass capillary were showed visually.

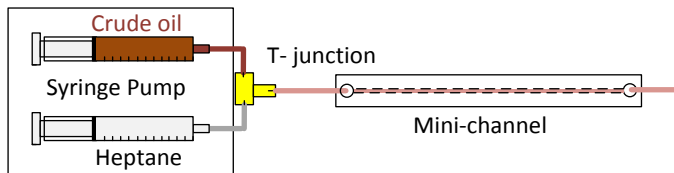
The objective of this article is to present a new approach to measure asphaltene deposition profiles in a rectangular Plexiglas mini-channel.

In what follows, the experimental setup, details of crude oil preparation and 3D microscopy technique are described in the second section. The results of this study are presented in third section which contains the 3D distribution of the asphaltene deposition with its corresponding vertical profile at a selected horizontal position. Finally, some conclusions and the use of this measurement technique for future work are discussed.

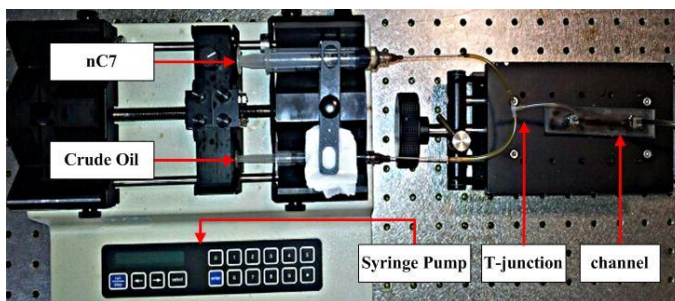
EXPERIMENTAL CONDITIONS AND MEASUREMENT TECHNIQUES

Experimental setup

The experimental setup consists of a rectangular horizontal mini-channel of size 1 mm × 2 mm × 53 mm (height × width × length) made of transparent Plexiglas as shown in Figure 1. The mini-channel was designed and fabricated using a CNC machine. The machined side of the Plexiglas represents the bottom plate and a non-machined transparent sheet of Plexiglas is deposited on the top side of the channel and glued using chloroform. Chloroform allows the Plexiglas material to stick to each other semi-permanently. For the entrance and exit, miniature barbed Polypropylene fittings are attached with epoxy glue. The channel is filled with a mixture of two miscible liquids using a Cole-Parmer double syringe pump (having a maximum flow rates of 190.8 mm/min), which operates with two independent syringes.



(a) Schematic of experimental set-up



(b) Top view photography

Figure 1: Experimental setup

A T-junction is used at the entrance of the mini-channel to inject and mix n-Heptane (nC7) and crude oil with a constant mixture flow rate of 0.005 ml/min. The properties of the

working fluids are reported in Table 1. The volume of syringes (BD Luer-Lok™) used for injecting crude oil and n-Heptane were 3ml and 10ml respectively. The calculated ratio of crude oil to n-Heptane for our experiments was 28:72, respectively. The obtained liquid flow rate ratio represents an optimal mixture for generating asphaltene particles in the system. The temperature of the working fluids is 24°C (room temperature). The experiments were carried out over 12 hours. The fluid mixture at the outlet was collected in a disposal container.

Crude oil preparation

The oil samples used for these study was found to be highly unstable (i.e., asphaltene had already precipitated in the oil) at the laboratory conditions. These precipitated particles are removed by centrifuging the crude oil for 20 minutes at an angular speed of 5000 rpm. The absence of major suspended particles was further confirmed by examining the supernatant oil obtained after centrifugation using a Hiox KH7700 digital microscope. The oil sample was then titrated under ambient conditions with an asphaltene precipitant such as n-Heptane. In order to obtain the best ratio of oil to asphaltene precipitant, the oil sample was titrated with n-Heptane in the ratios of 0:100 to 100:0 over an aging period of 5 minutes. The ratio of 28:72, oil to n-Heptane was found to be the most suitable ratio for the deposition study of asphaltene over an aging period of 5 mins in the micro-channels. The ratio of oil to n-Heptane can be well maintained by using different sized syringes in combination with the syringe pump, as the volume (or internal diameter) of the syringe determines the flow rate for each syringe.

Table 1: Properties of the working fluids

	Density (g/cm ³)	Kinematic Viscosity (cSt)
Crude oil	0.838	6.521
n-Heptane	0.684	0.647

3D Microscopy technique

After 12 hours of continuous mixture flow, the syringe pump is stopped and connectors were removed from the mini-channel. The top cover of the channel was removed using acetone to facilitate measurements of asphaltene deposit in the channel. The high resolution 3D depth profile of asphaltene was observed using a Hiox KH 7700 digital microscope (Table 2) with a maximum magnification lens of ×3500. As shown in Figure 2, the 3D microscope was installed perpendicular to the horizontal channel and records digital image at different vertical positions.

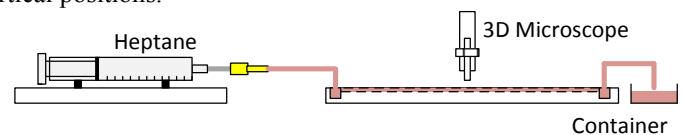


Figure 2: 3D Microscopy technique

The microscope starts from the very bottom surface of the sample and moves upwards at a constant interval distance until it reaches the very top surface of the sample. The bottom and

top surfaces are defined by the user before performing a 3D scan of the sample. Interval distance is determined relative to the distance between the bottom surface and the top surface. Every image taken by the microscope has different zones with fully in and out of focus. The height of each picture is recorded to construct the 3D structure of the deposition. A maximum of 128 images can be taken by the microscope between the bottom and top surfaces. The smallest interval increment between two images is 0.25 μm .

Table 2: Properties of the 3D digital microscope

Production Name	Hirox KH-7700
Use Lens Name	MX(G)-5040SZ: OL - 350 II
Magnification	350 - 3500 \times
Depth of Field (Optical)	762 μm to 90 μm

RESULTS

The asphaltene layer thickness is related to the amount of asphaltene particles deposited on the walls of the mini-channel. The 3D image of asphaltene layer thickness deposited on the bottom wall of the mini-channel is presented in Figure 3. This figure represents a reconstructed image of 128 singles images taken between the bottom and top surfaces; each image focused at different vertical positions of the channel. Due to the high magnification of the lens, only half region of the micro-channel is visible. The black area represents the deposited layer of asphaltene particles. The white area of the image represents the clean (no asphaltene deposition) zone corresponding to boundary of the channel which is the bottom surface in this case. As it can be observed in the figure, the entire vertical wall of the mini-channel is also covered by a thin layer of asphaltene.

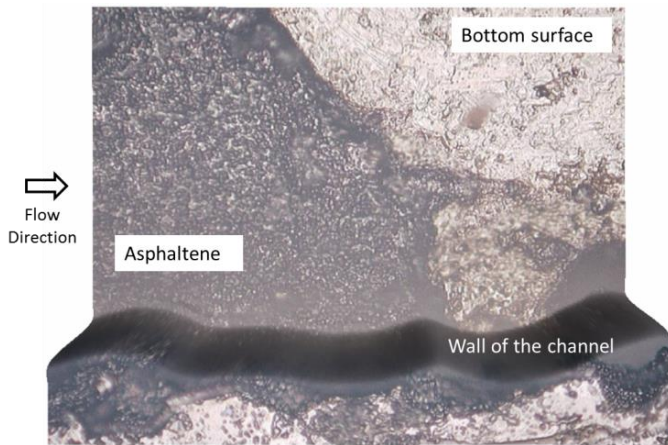


Figure 3: 3D view of the reconstructed image of the asphaltene layer thickness in a mini-channel (the size of the 2D image is 1mm \times 1.2mm; the magnification is \times 350)

In order to visualize the 3D dimension effect of the image, Figure 4 shows the constructed image of the asphaltene layer thickness using an isometric view. The thin layer of asphaltene on the bottom surface of the channel is clearly illustrated. An approach to measure precisely the thickness of asphaltene

deposition, and provides a 3 dimensional profile of the deposition using 3D microscope is described next.

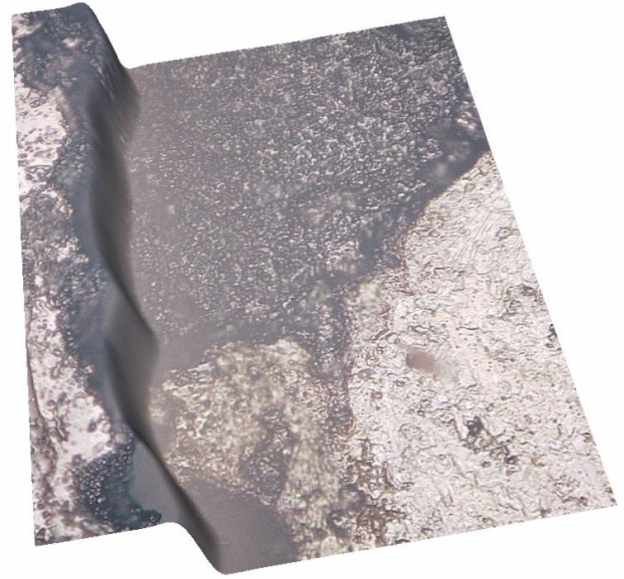


Figure 4: 3D Isometric view of the reconstructed image of the asphaltene layer thickness in a mini-channel

In Figure 5, the depth of each layer of the asphaltene deposited on the bottom wall is measured and presented using a palette of colors. The legend is given in the lower bar with dimension in μm . Blue and red represent the lowest and the highest vertical positions in the reconstructed image which corresponds to the bottom and top surfaces of the channel, respectively. In this image, the maximum thickness of the deposited layer is approximately 10 times smaller than the entire height of the channel. Therefore higher magnification is necessary to obtain better resolution and better estimation of asphaltene deposition thickness.

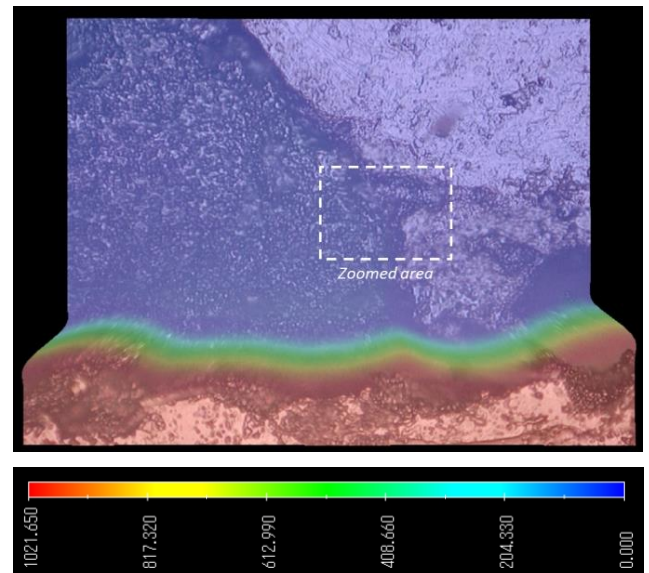


Figure 5: 3D distribution depth of the asphaltene deposition layer

In Figure 6, a selected area from Figure 5 is zoomed with $\times 1050$ magnification. The 3D profile of asphaltene including the elevation at each point is shown using colors. The highest and the lowest positions of the asphaltene deposit are at $62\mu\text{m}$ and $8\mu\text{m}$, respectively, with an error of $\pm 4\mu\text{m}$.

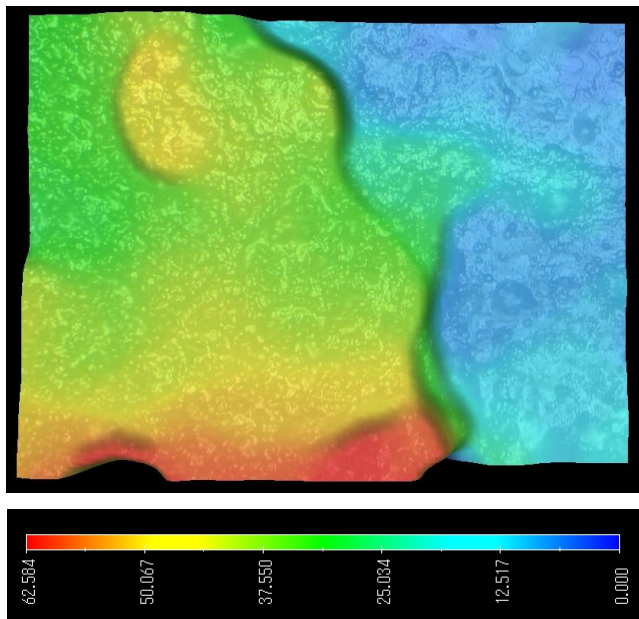


Figure 6: Zoomed area from Figure 5 showing a 3D distribution depth of the asphaltene deposition layer (the size of the 2D image is $220\mu\text{m}\times 280\mu\text{m}$; the magnification is $\times 1050$)

From the reconstructed 3D image, a 2D distribution of the asphaltene deposition can be obtained. The position of the 2D distribution is highlighted in the isometric view of the zoomed area in Figure 7. A selected vertical rectangular area (in red) perpendicular to the reconstructed image where asphaltene deposition profile is being sought-after is shown in Figure 7. The corresponding 2D profile of the asphaltene is presented in Figure 8 where the horizontal component represents the axial coordinate (x) along the channel and the vertical component represents the height of the asphaltene deposition (h).

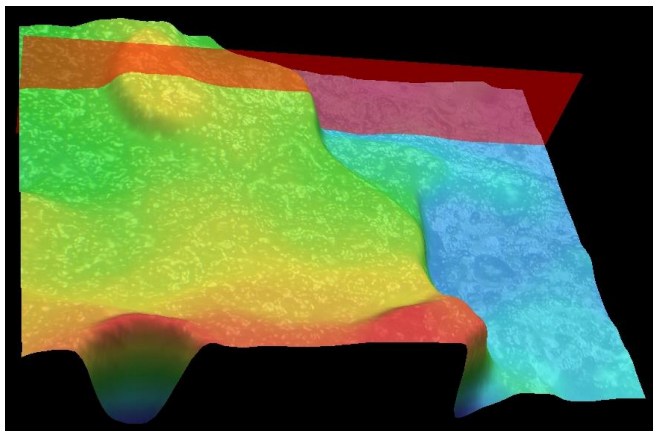


Figure 7: 3D Isometric view of the zoomed reconstructed image of the asphaltene layer thickness in a mini-channel

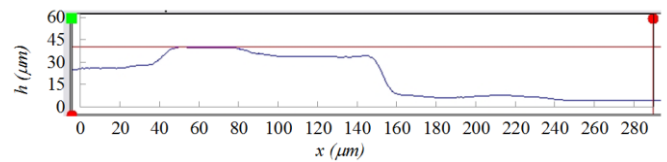


Figure 8: 2D profile of the asphaltene deposition at the selected position from Figure 7

In Figure 8, the maximum height of the asphaltene deposition layer is $42\mu\text{m}$ and is located in the region of $50\mu\text{m} < x < 80\mu\text{m}$. A sudden decrease of the deposition is also observed at $x = 160\mu\text{m}$ corresponding to the position where a small amount of asphaltene was removed after the experiment to identify the bottom surface of the channel. Although not shown, a “complete” deposition profile over the whole width of the mini-channel can be obtained by measuring the 2D deposition profiles, as the one shown in Figure 8, from one side of the mini-channel to the opposite side of the same channel.

Using this 3D measurements technique, it is possible to quantify the amount of asphaltene deposited on the bottom wall of a mini-channel. In this study only 2 magnifications (i) $\times 350$ and (ii) $\times 1050$, have been used to quantify the thickness of the deposition layer. This study can be performed at lower scale and new experiments are focused on the measurements of the asphaltene deposition in micro-scale. The influence of an obstacle on asphaltene particle deposition is also under analysis in order to apply this method of measurements in case of asphaltene deposition in micro-porous media.

CONCLUSION

A new method of measurements using a 3D microscopy system is presented. A reconstructed image is produced to visualize the topology of the 3D asphaltene deposition layer. The thickness of the deposition layer is estimated and two-dimensional profile of the deposition is measured. This new approach will permit quantification of deposition layer and study the mechanism of asphaltene deposition. Further experiment is under analysis to expand this measurement into micro-scale level. This measurement technique will be used to validate new numerical models in order to predict the asphaltene deposition in oil and gas equipment.

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