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## **DEVELOPING METHODOLOGY AND EXPERIMENTAL PROCEDURE FOR EXPERIMENTAL MICROFLUIDIC STUDY OF CHEMICAL ENHANCED OIL RECOVERY**

*The concern of this research was to review the different methodology on microfluidic experiments conducted to study chemical enhanced oil recovery methods on micromodel chips. In general, there are several ways to study EOR methods such core flooding and microfluidics. The disadvantage of first method is that the flow processes inside the core sample cannot be imagined. Hence, the second method helps us to fully visualize how fluid flow behaviour occurs through the porous medium of the rocks.*

*The various chemical EOR processes such as surfactant flooding, polymer flooding and ASP flooding were studied on microfluidic chips. These chemicals were injected into micromodels to drive out crude oil. It helps to understand the interactions between crude oil and chemicals, the advancement of front developed between displacing and displaced fluids and the viscous fingering effect. Visual studies enabled us to understand the effectiveness of polymer, surfactant and alkaline separately and as combined.*

*The different experimental methodologies to study the EOR methods are reviewed. Mainly experiments divided into two main groups: methods of geological characterisations influence to fluid transport while others study oil displacement at different condition, such high temperature, high pressure, low or high salinity, highly viscous oil. We reviewed methodologies applied to study an oil displacement by polymer, surfactant-polymer (SP) and alkaline surfactant polymer (ASP) solutions, their interactions and transport in porous media. Based on reviewed article the experimental procedure was developed. Analyses of published materials have helped to design and direct the methodology of research.*

***Keywords:** microfluidics, glass micromodel, ASP flooding, polymer flooding, recovery factor, concentration.*

**Introduction.** Enhanced oil recovery is the last stage of oil and gas production and its main objective is to mobilize the remaining oil through enhancing the oil displacement and volumetric sweep efficiency. For this purpose, different types of chemicals are used such as polymer, surfactant and alkaline. Polymer is added to brine to increase its viscosity, alkaline and surfactant to decrease water-oil interfacial tension. Altogether, injection of chemicals can lead to changes in fractional flow, mobility ratio. As a result, it helps to reduce viscous fingering and improve displacement profile, increase swept area [1]. There are several methods applied to laboratory study of chemical EOR methods such as core flooding and microfluidics.

Core flooding experiments are the classical way of performing oil recovery studies by displacing oil from saturated rock samples using various flooding approaches. An advantage of this method includes the possibility to perform measurements at similar to reservoir conditions. Limitations of the core flooding method include long and not always repeatable measurements, insufficient number of core plugs from the reservoir and time consuming,

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specialized methods to visualize the processes inside the cores. The latter means that information about fluid displacement processes is normally based on indirect measurements. Immiscible fluid–fluid displacements can either be stable or unstable. The unstable displacement can further be categorized into viscous or capillary fingering and are major reasons for inefficiency in subsurface two-phase flow [2]. To assess the displacement processes, it is useful to have thorough understanding of displacement stability and fluid flow pathways [3]. Detailed mapping of fluid displacement in porous media can benefit from easy optical visualization. This, together with auxiliary measurements, can result in improved knowledge in the flow dynamics of fluids in pores and provide better input for numerical simulations, as shown recently by Yiotis et al. 4.

Microfluidic research method allows optical visualization of many physical processes. Microfluidics was first applied in microbiology as a tool for analytical analysis. However, nowadays its popularity dramatically increased, and it found application in many scientific fields, such as medicine, genetics and oil and gas industry. In oil and gas area it mainly applies for enhanced oil recovery (EOR). In EOR microfluidics utilized to study fluid flow patterns that occur inside pores and permeable zones of the sub surface rocks, which are not possible in case of core flooding. EOR micromodel chips help to get real-time flow behaviour by visualising of fluid flow and relevant in-situ phenomena observed during fluid flow in porous chips [5]. It became highly valued mainly because of its size. Commercially it is better because of reduction in the usage of chemicals as well as power consumption. [6]. This a new method for our industry research, it is applying around 15years, and for this time there is no such research done for the local oilfields.

Our aim is to study EOR methods on pre-Caspian basin oilfields and in this article we describe the methodology of conducting such experiment.

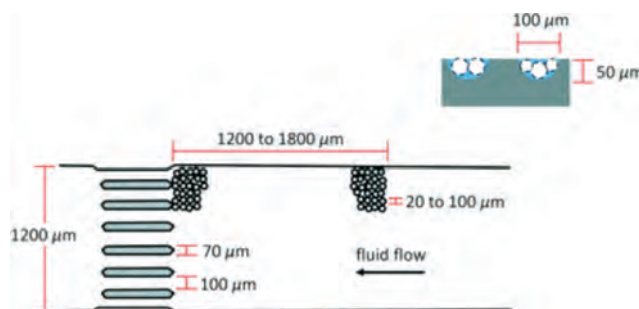
In the present article we demonstrated the study of oil displacement by polymer, SP and ASP solutions in an oil saturated packed bed in a microfluidic channel. For this article we analyzed several works on experimental study of chemical enhanced oil recovery conducted in micromodel set ups. Based on these works we developed our methodology. The methods, experimental set up and materials are described in this article. In the next work the results of the experiment with Pre-Caspian basin crude oil and brine will be described.

**Materials and methods.** In the laboratory model carbonate, sandstone grains from the fields can be applied. If they are not accessible the glass beads can be used to represent sandstone rock. If grains are chosen for work they should be sorted out to fill the micromodel. For this purpose, crushed sandstone are sorted in 45 micron sieves, since the thickness of the chip is 50 microns. However, problems with filling the channel can occur due to not uniform structure of grains and its roughness. The channel can be blocked, in our model the channel became impermeable after 1/3 is filled with grains. To solve the problem the structural sieving can be applied. It was decided to sieve the grains larger than 53 and less than 65, but the problem was not solved. (Shown on figure 1) Packed bed remains impermeable. Therefore, it was decided to use glass beads packed micromodel. Firstly, they were available in the lab and secondly, the beads commonly used to represent sandstone rocks. The main disadvantage is that glass beads have the same size and shape, therefore it creates homogenous pore structure which is not representative of real reservoir structure.

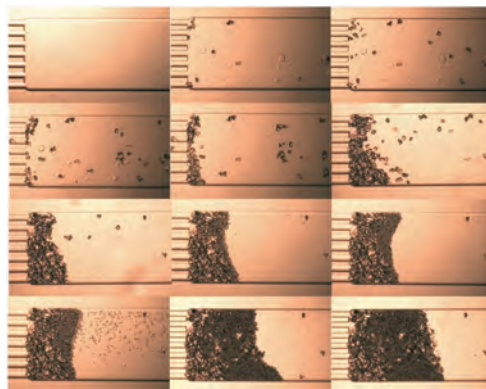


**Figure 1** – Sieving of crushed rock samples on vibratory sieve shaker

In the figure above the sieving process of crushed rock samples on vibratory sieve shaker is shown. In this case the micromodels are unconsolidated, quasi-2.5-dimensional beds of sandstone grains can be packed into micromodel channels.



**Figure 2** – Plan view of the microfluidic channel in which the packed bed is assembled.



**Figure 3** – Assemble of packing process.

Fluid with glass beads/sandstone grains are dispensed from two ports on the right 14 or 18 mm upstream of the gap filter and expelled from a single port on the left (not shown).

Micromodel network can be uniform [4,7, 10] or structured [8, 9]. The channels mainly are 50 μm or 20 μm deep and width can be in wide range from custom made to individually fabricated. The chips can be made of borosilicate or dolomite glass and can hydrophobic or hydrophilic. There are different ways to make micromodel chips. It can be also made PDMS based chips, silicon wafer micromodels, etc. Initial models contained a monolayer of glass beads between two glass plates and done by Chatenever (1952). A plastic sheet was pressurized between beads by Oxall et al. (1952) to avoid bypass of fluids between plates and beads, Tsakiroglou et al. (2013). This model did not require any specific pattern of the porous medium. A micromodel can be reused many number of times compared to a core. The cleaning system is used to clean a fresh micromodel to prepare it for flooding experiments like making it to desirable wettability as well as cleaning it after the experiment is performed.

**Experimental set up.** The basic requirements are similar for different researchers. For the research the microfluidic system has parts which include inlet for fluids, a pump system to move these fluids through the microchips along with sensors to determine the pressure and flow rates at inlet and outlet of the microchips. To visualize pore-scale mechanisms in two dimensional micromodels camera coupled to an optical microscope is used.

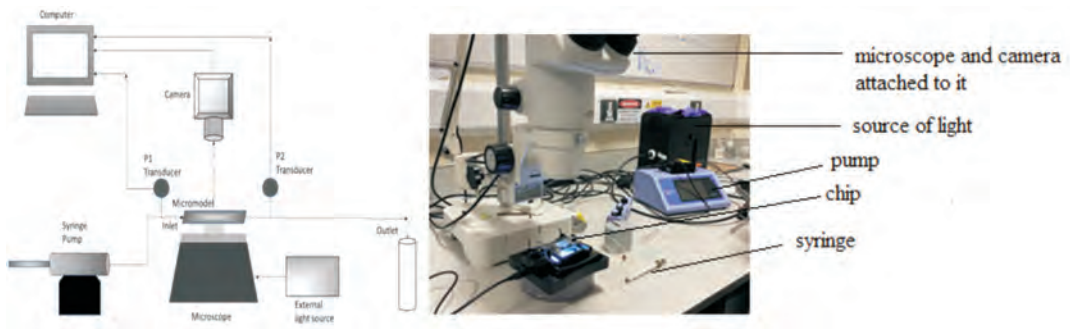


Figure 4 – Schematic diagram of microfluidic set up and picture of set up available

### Injection system

#### Syringe Pump and syringes

The syringe pump such as Harvard apparatus, pump 11 Pico plus elite can be used. The pump is used to push the syringe holder while an emulsion, brine and oil is filled into syringes. The flowrates for the experiments are the following:

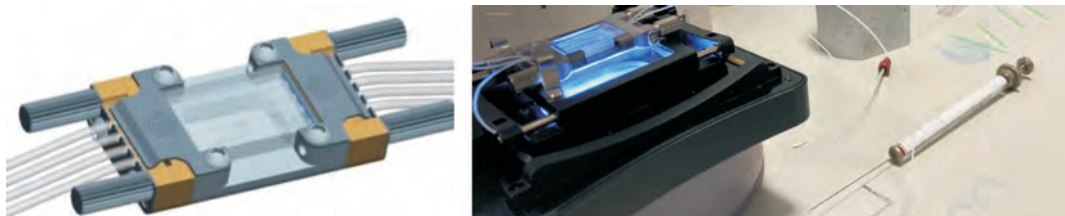
- x The minimum flow rate: 0.0005 μL/min
- x The maximum flow rate: 10.0 μL/min
- x The minimum pressure: 0 psi
- x The maximum pressure: 8 psi

Glass syringes from SGE syringes, in range 100-500 μL were used.

Pressure transducers are utilized to measure pressure differences as well as absolute pressure at a point. The pressure difference is measured between inlet and outlet of the

micromodels through connection into tubing in and out of these models. The systems consist of a pressure transducer, a digital interface box and a display to a computer screen. The pressure is usually measured in bar

**Micromodel chip, chip holder, connection tubes.** Chip as mentioned above can be custom made (Dolomite Centre Ltd.) or fabricated in lab. Custom made chip has 6 channels. In general, only one channel used at a time. It has 2 inlet ports and 1 outlet. It is vital to have second inlet port, to relief air bubbles coming in to prevent 3 phase flow. Glass chip is placed in chip holder, tubes are connected to chip holder to inject and discharge necessary fluids.



*Figure 5* – Microchip image and chip and syringe available at lab

#### Data gathering and visualization system

Microscope Nikon SMZ745T or other with high resolution can be used. It should has high range of zoom from of 0.67x to 5x which provides a broad observation range to visualize pore scale processes. Microscope incorporates an optical path switching lever that enables to switch between eyepiece and camera.

High-speed 24-bit colour camera Pixelink PL-B742F is applied to capture images. It has many features that allows to take pictures each seconds, different colour range, at different resolution and many other.

To mimic the process well it is vital to use variable wavelength light source. Lumen 1600-LED, Prior Scientific is applied in the experiment for this purpose.

**Microscope for packing.** Since during the packing that is not necessary to take picture of the process, for this purpose simple and cheap IQCREW Portable LCD colour digital microscope by Amscope was used.

**Programs used to capture, process, record the data.** Captured images processed by Fiji – imageJ program. ImageJ is a Java-based image processing program developed at the National Institutes of Health and the Laboratory for Optical and Computational Instrumentation (LOCI, University of Wisconsin). ImageJ can display, edit, analyze, process, save, and print 8-bit color and grayscale, 16-bit integer, and 32-bit floating point images. It supports image stacks, it also can calculate area and pixel value statistics of user-defined selections and intensity-thresholded objects. It can measure distances and angles. (wikipedia)

ESI ElveFlow is used to record pressure sensors data.

Pixel link OEM is helped to control and manage the characteristics and properties of camera to have a good quality image.

**Auxiliary equipment.** For the polymer preparation magnetic stir was used. For the proper mixing high speed was chosen.



To measure basic properties such as density and viscosity Anton Paar viscometer was available in the lab. The interfacial tension between oil and water, oil and AS solution were measured by Du -Noüy ring tensiometer Sigma 703d (Dyne Testing Ltd). Ph of the brine was measured by electronic Ph meter.

**Experimental procedure used to study EOR methods.** Experimental procedure can be varied in different authors but mainly consist of the following steps:

Assembly the backed bed or fabrication the own micromodel

Displacement experiments for each flooding methods

Image analyses

For our experiment we develop the following procedure:

➤ Clean the chip and micromodel, change tubes (pipes)

➤ Pack the bed

➤ Saturate micromodel with brine

➤ Calculate absolute permeability

➤ Saturate micromodel with oil

➤ Capture image to determine  $S_{wi}$

➤ Initiate flood experiment

Depending on the flood experiment (Polymer/ASP/SP)

➤ Start the flood with necessary solution or brine

➤ Capture image to determine residual oil saturation and recovery factor

#### **Chip Cleaning.**

i. The chip holder is cleaned with isopropanol then distilled water

ii. Three pairs of tubing is cut using stanley knife. Each pair of tubing is connected to 2 inlets (A1, A2) and 1 outlet A3 through the rubber seal leading to the channel.



*Figure 9* – Schematic diagram of the channel, where bed is packed

iv. The chip is flushed using the isopropanol, then air and distilled water is injected in sequence to remove the solvents through A1 while A2 is blocked with a syringe to prevent a back flow. This is repeated until the channel is completely cleaned (verified by viewing under a microscope).

#### **Packing the bed.**

v. The chip holder seal was detached. By using tubing (A1) suspension of sandstone grains in isopropanol and distilled water mixture is sucked from vial containing it. Care should be taken to ensure that only a small amount was drawn up as too much sand could block the tubing. (what happened time to time anyway)

vi. The chip was re-inserted and held in place within the chip holder. A2 is blocked with a syringe while solution with beads was injected through A1 to push the beads through the channel. This procedure is altered so that A1 is blocked and distilled water is injected through A2. This is used to push the beads/grains deeper to the channel. The entire process should be repeated until grains build up and occupy about 1.5 the channel width.

vii. Once the desired length is achieved, the tubing can be replaced to prevent further grain injection

**Saturate micromodel with brine.**

viii. The three tubing should cut and connected to the inlets and outlet of the channel containing the packed bed. A2 is blocked with a syringe while brine is injected through A1 by using syringe pump at constant flow rate. To fully saturate the model 100microliter of brine is injected. During the injection of the brine absolute permeability will be measured.

ix. To measure absolute permeability pressure sensors should attached to the pumping unit, pressure and flow rate should be recorded. To have correct data at least 7 pressure values have to be measured at different flow rate. To calculate absolute permeability Darcy equation was used.

$$K = \frac{q\mu L}{A\Delta P}$$

where,  $k$  is the permeability of the packed bed in Darcy;  $A$  the cross-sectional area of the micromodel  $\mu m^2$ ;  $\Delta P$  press drop across the channel;  $\mu$  the dynamic viscosity of the injected fluid, brine for absolute permeability, oil for relative permeability;  $L$  is the length of the packed bed;

After that to create reservoir condition oil was injected to the micromodel.

**Oil injection.**

x. While observing under an optical microscope, the packed bed is flooded with crude oil by injecting through A1 while A2 is blocked with a syringe. This should be continued until the packed bed is saturated with the oil as evidenced by oil flowing out from the outlet tubing. The image will be captured to calculate initial oil and water saturation. Images will be processed on ImageJ tool. Based on pixel count initial oil saturation and water saturation can be calculated. (Image processing is time consuming process, therefore could not be performed during the internship. All image processing work will be start soon)

Next steps is flood experiments. This stage, we divided to the following parts:

- Flow rate screening to find suitable flow rate to mimic process behavior and relation flow rate and injection fluid interaction;
- Perform polymer flood at different concentration to observe front evaluation and understand relation between viscous fingering and polymer concentration;
- Alkaline-surfactant flooding then polymer flood to compare with ASP flood;
- To calculate IOR, oil production sequence is designed. Polymer or ASP flood is designed after waterflood.

1 parameter was changed at a time.

**Waterflooding.**

xi. The syringe containing brine is fitted to the pump and connected to a long tubing, which is then connected to A1 with A2 blocked to prevent oil flowing out. Brine is slowly injected in small amounts through A1 to clean the flow lines of oil from tubing A1 and A2. A2 is then blocked with a syringe and the pump is started. Brine was pumped into the packed bed at a constant flow rate 0.0008  $\mu L/min$  using the Harvard apparatus.

xii. Images should be captured just before the brine injection commences and throughout the duration of injection. The residual oil saturation is calculated through images.

**Polymer /surfactant polymer/ASP flooding.**

xiii. While being observed under an optical microscope and images continuously captures, 100microliter of prepared chemical solution (polymer/surfactant-polymer or ASP) is pumped into the packed bed trough A1 while A2 is blocked with a syringe.

xv. Images should be captured throughout the duration of chemical injection. Front evaluation, swept area, oil displacement will be calculated from images on ImageJ.

**Cleaning.**

xvi. The chip was flushed with polymer and Decon 90 solutions to remove the remained oil. After that, isopropanol/distilled water is injected in sequence to mechanically remove the grains/beads.

After cheap is clean above procedure is repeated for the next experiment.

Experimental procedure for the microfluidic was described above. Additionally, the following experiments will conducted:

- Polymer preparation
- Measurement of basic physical properties of oil, polymer, brine
- Measurement of interfacial tension for brine-oil, AS-oil

**Polymer Mixing.**

To prepare the polymer solution mechanical stirrer, scale and a beaker is needed. For each experiment different concentration and, therefore, different amounts of polymer is used. For these experiments, brine is used to mix the solution. Solution should be prepared carefully to avoid any precipitation and polymer sticking. For an appropriate mixing, the stirrer had to be set up at a speed fast enough to build a very strong vortex. Then, the weighted polymer will be added slowly by sprinkling it into the wall of the vortex. After 30 minutes of stirring, the RPM was increased to approximately 400 -500 RPM and was stirred for 3-4 hours. The mechanical stirrer heated up the solution, which also helped to improve the dissolution process. However, to prevent any blocking of the tubing and channel that can occur by polymer molecules stacked together or with undissolved particles, the polymer solution was filtered with filter paper in size of 15micron

**Conclusion.** Substantial amount of microfluidics works in EOR study has been reviewed. Many published materials have helped shed light into the advantages and disadvantages of microfluidics in oil and gas industries. By analyzing these works method of research has been developed to conduct the laboratory experiment on pre-Caspian basin oilfield. The main reason of applying microfluidic research is that wide range of study can be performed in experimental set up. The following studies such as study of flow behaviour at different conditions, effect of different patterns on fluid flow, interfacial interactions between oil, water and solutions, wettability effect on recovery factor, relative permeability studies can be evaluated and visualized. However, because of time limitation only a few of them can be performed. The study of flow behaviour at different concentration, front evolution at different flow rate and polymer, SP and ASP flood and their effectiveness and recovery factors were chosen for further study.

The micromodels are based on many raw materials. Among them the popular ones are the use of glass micromodels and PDMS micromodels. Based on availability of the



laboratory the dolomite glass model were chosen. At first, sandstone grains were preferred to pack the bed, however due to blocking of the channel the glass beads will be applied. It is possible to design a range of heterogeneity, pore structure and flow patterns.

We will study the displacement mechanism at different combinations and concentrations of polymer, surfactant and alkaline used for EOR processes. For this purposes polymer of the SNF company Flopaam 5205 is chosen. As a surfactant a Sodium C14-16 Olefin Sulfonate, as an alkaline Sodium hydroxide will be used. Different concentration of polymer and Alkaline-surfactant solutions will be prepared.

For the result analysis images processing tools are required and Image J image processing software were selected for this work. The experiment and its results will be described in the next article.

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## ХИМИЯЛЫҚ ӘДІСТЕРМЕН МҰНАЙ ӨНДІРУДІ АРТТЫРУДЫ ЭКСПЕРИМЕНТТІК МИКРОФЛЮИДТІ ЗЕРТТЕУЛЕР ЖҮРГІЗУ ӘДІСТЕМЕСІ МЕН ӘДІСТЕРІН ӘЗІРЛЕУ

Бұл зерттеудің мақсаты микромодель чиптерінде химиялық әдістермен мұнай өндіруді арттыру әдістемесін зерттеу үшін жүргізілген микро-сұйықтық эксперименттерінің әртүрлі әдістерін қарастыру болды. Жалпы алғанда, мұнай өндіруді арттырудың бірнеше әдісі бар, мысалы, суландыру және микрофлюидты. Бірінші әдістің кемшілігі – негізгі үлгінің ішіндегі ағым үдерістерін бейнесін бере алмауы. Сонымен, екінші әдіс тау жыныстарының кеуекті ортасы арқылы сұйық ағынының әрекетін толығымен бейнелеуге мүмкіндік береді.

Мұнай өндіруді арттырудың беттік-белсенді заттармен суландыру, полимерлермен суландыру және ASP суландыру тәрізді әртүрлі химиялық үдерістері микро-сұйықтық чиптерде зерттелді. Аталған химикаттар ишкі мұнайды ығыстыру үшін микромодельдерге енгізілді. Бұл ишкі мұнай мен химиялық заттардың өзара әрекеттесуін, ығыстыратын және ығыстырылатын сұйықтықтар арасындағы шекаралық фронттың алға жылжуын және тұтқыр саусақтардың әсерін түсінуге көмектеседі. Көрнекі зерттеулер суландыру жағдайында қолданылатын полимердің, беттік-белсенді заттың және сілтінің тиімділігін, жеке-жеке химикаттар үшін және олардың әртүрлі қоспалары үшін, түсінуге мүмкіндік берді.

Мұнай өндіруді арттыру әдістерін зерттеудің әртүрлі эксперименттік әдістемесі қарастырылған. Тәжірибелер негізінен екі басты топқа бөлінеді: бірінші топқа геологиялық сипаттамалардың сұйықтықтың берілуіне әсер ету әдістері, ал екінші топқа жоғары температура, жоғары қысым, төмен немесе жоғары тұздылық, жоғары тұтқыр май сияқты әртүрлі жағдайларда мұнайдың ығысуын зерттейтін эксперименттер жатады. Мұнайды полимерлер, беттік-полимерлер (PP) және сілтілі беттік-полимерлер (ASP) ерітінділерімен ығыстыруды, олардың кеуекті ортадағы әрекеттесуі мен тасымалдануын зерттеу үшін қолданылатын әдістер қарастырылған.

**Түйін сөздер:** микрофлюид, микромодель, АЭК суландыру, полимерлі суландыру, Мөк, концентрация.

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### РАЗРАБОТКА МЕТОДОЛОГИИ И МЕТОДИКИ ПРОВЕДЕНИЯ ЭКСПЕРИМЕНТАЛЬНЫХ МИКРОФЛЮИДНЫХ ИССЛЕДОВАНИЙ ХИМИЧЕСКОГО МЕТОДА ПОВЫШЕНИЯ НЕФТЕОТДАЧИ

Цель этого исследования заключалась в рассмотрении различных методологий микрожидкостных экспериментов, проведенных для изучения методов повышения нефтеотдачи химическими методами на чипах микромоделей. В целом, существует несколько способов изучения методов повышения нефтеотдачи, такие как заводнение керна и микрофлюидное исследование. Недостатком первого метода является невозможность представить процессы течения внутри образца керна. Таким образом, второй метод помогает нам полностью визуализировать поведение потока жидкости через пористую среду в породе.

*Различные химические процессы повышения нефтеотдачи, такие как заводнение поверхностно-активными веществами, заводнение полимерами и заводнение полимер/ПА В/щелочь, были изучены на микрожидкостных чипах. Данные растворы вводили в микромодели для вытеснения сырой нефти, что впоследствии помогает понять взаимодействие между сырой нефтью и химическими веществами, продвижение фронта между вытесняющими и вытесняемыми флюидами и эффект вязкого пальцеобразования. Визуальные исследования позволили нам понять, какова эффективность заводнения полимером, поверхностно-активным веществом и щелочью по отдельности и в сочетании.*

*Рассмотрены различные экспериментальные методики изучения методов увеличения нефтеотдачи. В основном эксперименты делятся на две главные группы: первая группа охватывает методы влияния геологических характеристик на перенос жидкости, во вторую группу относятся эксперименты, изучающие вытеснение нефти при различных условиях: таких как высокая температура, высокое давление, низкая или высокая соленость, высоковязкая нефть. Рассмотрены методики, применяемые для изучения вытеснения нефти растворами полимеров, ПАВ-полимеров (ПП) и щелочных ПАВ-полимеров (АСП), их взаимодействия и переноса в пористых средах.*

*Проанализировав несколько методов проведения эксперимента и взяв их за основу и учитывая возможности лаборатории, имеющихся расходных материалов, была разработана методика проведения эксперимента.*

**Ключевые слова:** микрофлюид, микромодель, АСП заводнение, полимерное заводнение, КИН, концентрация.