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# INFLUENCE OF GRANULAR BED PARAMETERS ON EMULSION FLOW AND ELUTION PROCESS OF OIL-IN-WATER EMULSION

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Key words: porous media, oil-in-water emulsion, elution.

Abstract

Emulsion flow through porous structure is used in many processes in the field of chemical engineering. Good examples of practical applications are Enhanced Oil Recovery (EOR) techniques, soil remediation and treatment of oily wastewater. The emulsion transport in through porous media is not easy to describe, due to rheological behavior of emulsions and porous structure properties. In case of emulsified system flow through porous bed it is possible to observe the retention of oil on porous structure, and reduction of permeability of bed. In the presented study, we tried to investigate the influence of porous bed parameters on oil-in-water emulsion behavior during its flow and elution from porous structure. In experiments we used porous beds with varied particle size range and with different lengths. We measured pressure drop and related permeability changes in time. The turbidimetric and microscopic analysis that we conducted allowed us to check how concentration of emulsion changed during its flow and elution from porous structure.

**Symbols:**

$L_z$ – porous bed length [m]
$\Delta p$ – pressure difference [Pa]
$c_e$ – concentration of base emulsion [%]
$c_w$ – concentration of emulsion during experiment [%]
$\varepsilon$ – porosity of medium
$Q_v$ – volumetric flow rate [m$^3$/s]
$k$ – the permeability coefficient [m$^2$]
$l$ – the distance of flow [m]

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$A$ – surface of cross-section of bed [m$^2$]
$\mu$ – dynamic viscosity of liquid [Pa · s]
$n_{ei}$ – number of droplets with certain diameter
$d_{ei}$ – diameter of emulsion drop

## Introduction

Transport of emulsion in porous media is crucial for such processes as crude oil extraction, soil remediation and wastewater treatment (LANGEVIN et al. 2004 and YOUNG-CHUL 2007). The knowledge in field of emulsion flow in porous structure can help companies to reduce costs associated with oil recovery (CRAWFORD et al. 1997). The phenomena observed during two phase flow in porous medium are also important for geomechanics, hydrogeology and reservoir engineering (WANG 2000). Knowledge of emulsion systems migration in granular structures can contribute to the development of new techniques for obtaining oil from oil sands and oil shales (DULLIEN 1992, ALVARADO et al. 2013, BŁASZCZYK et al. 2016a). For example, in Enhanced Oil Recovery (EOR) techniques, better understanding of emulsion flow through porous media is needed for development and practical improvement of processes based on emulsion flooding mechanisms (MORADI et al. 2014, GUILLEN 2012, BŁASZCZYK et al. 2016c). It is also especially important in heavy oil recovery, where emulsified solvent flooding has been shown to be efficient technique (KUMAR et al. 2012).

Fundamental mathematical explanation of fluid flow through porous structure is given by Darcy's equation. This law can also be used to explain flow through porous bed indicated by a pump. In this case, the driving force will be the pressure difference $\Delta p$ between the pressure produced by pump and the atmospheric pressure (HEINEMANN 2005). Also, for porous structure having certain length and diameter it is possible to determine the permeability coefficient according to the Darcy's law. In equation (1) parameter known as permeability coefficient is introduced. It is a measure of the ability of a porous material to allow fluids to pass through it. The Darcy's law therefore can be described as:

$$Q_v = k \frac{A}{\mu} \frac{\Delta p}{l} \tag{1}$$

Emulsion flow in porous media differs from the independent movement of individual phases, and therefore must be considered separately (IDORENYIN et al. 2012). To describe the nature of such flows, permeability reduction mechanisms and changes in concentration of internal phase must be recognized. This allows to understand and to predict the way in which emulsion systems behave during transport in porous media. This knowledge is useful to obtain a comprehensive picture of multiphase flow in porous media.

During flow of emulsion through porous structure oil droplets can be trapped in it, because of the existing retention mechanisms of fluid in porous structure, caused by capillary forces (IRYNA et al. 2016). During the process of elution by washing liquid, these droplets can move again as emulsion systems.

In Enhanced Oil Recovery (EOR) techniques surfactants are added to the washing media, in order to reduce surface tension, and thereby increase the degree of elution. This is an additional factor causing the formation of the emulsion during the elution process (LI, GU 2005). During EOR sometimes emulsions are used to decrease mobility of the injected fluids, and they are called then extrusive liquid. They can be also applied to block highly permeable zones (MANDAL et al. 2010). However, the emulsions may also be formed in the bed by itself. For example crude oil can form dispersed system with water, since it contain naphthalene acid and resins, which exhibit the properties of natural surfactants to facilitate the formation of the emulsion structures. In such case during its extraction or transportation under shear forces emulsification phenomenon occurs. Moreover, the presence of variety of alkali in crude oil causes stabilization of formed systems. Evaluation of how the emulsions move in the granular bed, may also help to increase efficiency of soil treatment from oil-derived substances and reduce the associated financial costs (COBOS et al. 2009).

In practical situation during consideration of O/W emulsion flow process through porous medium it is important whether it can be treated as a homogeneous liquid or not. If the emulsion droplets are very small, compared to the size of the flow channels, it can be considered that the fluid behaves as one-phase medium and neglect the microscopic droplets of the emulsion (CORTIS, GHEZZEHEI 2007). However, in most practical cases, the sizes of the emulsion droplets are not significantly smaller than the pore sizes, or even larger from them, which means that their presence in the bed cannot be ignored. In such situation, it is necessary to examine how the various properties of the emulsion affect the flow through a porous medium (WANG, DONG 2011)

The flow of the emulsion through the porous medium depends on the properties of both: the porous bed and the dispersed system. Properties of obtained two-phase liquid such as: stability, concentration, droplets sizes and interfacial interactions, play here particular role. As regards to the parameters characterizing granular bed, wettability, pore average size and pore size distribution are the most relevant here. Wettability of the porous medium controls the flow, location and distribution of the fluid inside the porous bed.

This parameter affects the capillary pressure, relative permeability, water and oil saturation, and other properties (DULLIEN 1992). Pore sizes and their distribution are directly related to the capture and retention of oil drops. For example in the experiment described by CAI et al. (2012), GUILLEN et al. (2012) and ALVARADO et al. (2011) it was observed that, at the beginning of the process, the concentration of emulsion that leaved the porous bed was lower than the concentration of emulsion at the inlet to porous structure. Although, after some time, the concentration of emulsion leaving the bed increases and reaches the value similar to the one at the entrance to bed. This can be related to phenomenon known as "straining", where the oil phase is retained in porous medium and water flows out. The more precise explanation of this mechanism is as following: when small pores are clogged by oil drops the flow occurs through large paths. In that pores emulsion is not captured and their structure does not change, that is why the concentration of emulsion that leaves bed not varies.

Also, the pore diameter size distribution and emulsion droplet size distribution affects the reduction of permeability (HEINEMANN 2005). It has been observed (ALVARADO et al. 2011, BŁASZCZYK et al. 2016b) that the droplets leaving porous bed at the beginning of process were smaller than the ones initially pumped. However, during the process their size changed and bigger droplets were presented in outflow, and ultimately droplet size distribution was similar to the droplets size distribution in emulsion injected to the bed. The droplet size distribution was also a subject of other experiments (COBOS et al., 2009, MORADI et al. 2014) that showed that when droplet size increased, the retention of emulsion was also higher. It was explained by the fact that the probability that bigger droplets will be trapped inside pore is greater, and they fill pores faster. The emulsions with small diameter of droplets comparing to the pore sizes generally leave bed with the same oil droplet distribution (CORTIS, GHEZZEHEI 2007).

The aim of this study was to investigate the effect of particle size fractions on the processes of emulsion flow and elution from the porous bed. For this purpose experiments with the use of glass microspheres, with three different ranges of particle size diameters were carried out. Also the influence of bed length was examined.

## Materials and Methods

For experiments we used equipment presented in Figure 1. The test stand consisted of following elements: container with liquid (*1*), pressure indicator (*3*), signal converter (*2*), peristaltic pump (*4*), exchangeable tubes (*5*) that were packed with glass microspheres and volumetric flow indicator at outflow section (*6*).

The pump that we used was peristaltic type produced by ELPIN-PLUS-model 372c. This device was calibrated before measurements to obtain its characteristics. The signal converter PT-5261M was coupled with pressure indicator MD-5270. The tubes were made from stainless steel and had length of 0.2, 0.3 and 0.5 m and diameterof 0.05 m. For emulsion preparation we used: edible oil with viscosity of 60 mPa · s and density of 865 kg/m$^3$ (in ambient temperature), water and emulsifier Rokacet O7 obtained from PCC Exol S.A. To emulsify the liquids we used high speed handheld homogenizer. The glass microspheres were used as the porous beds. The experiments were carried in the temperature of 22°C.

The emulsion preparation technique was as follows: oil, water and emulsifier were mixed together for three minutes with high speed homogenizer to obtain dispersed system with given concentration. We prepared oil-in-water emulsion with internal phase concentration of 5% and addition of 2% Rokacet O7 as an emulsifier. The obtained system had stability time of more than 24 hours. The viscosity of prepared emulsion was also measured and equaled 1.5 mPa · s.

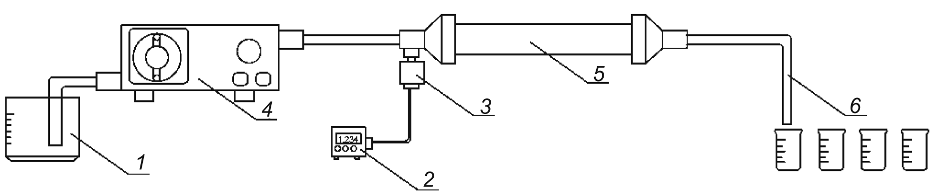


Fig. 1. Equipment used in experiments: *1* – container with liquid, *2* – signal converter, *3* -pressure indicator, *4* – peristaltic pump, *5* – tube with microspheres, *6* – outflow section with flow indicator

Before conducting the main part of the experiment we measured permeability of porous bed for water flow. In order to do so we packed the tubes with porous medium tightly, closed the tubes with nets and started to pump water. When the pressure showed by pressure indicator was stable, we noted it and calculated the permeability according to the equation (1). The results of this experiment are presented in Table 2.

To start the main part of experiment concerning emulsion flow and its elution from porous bed we placed certain amount of model porous bed in pipe. The bed was additionally compacted in order to make sure it is immovable. The experiments were conducted always with the same, previously established mass of porous bed that was also saturated with water. The tubes were equipped with rubber seals and nets preventing the bed from movement.Then we started to pump previously prepared oil-in-water emulsion with known parameters through it. Every 30 seconds we noted pressure difference measured by pressure indicator at the inlet to the bed. The liquid at the outflow was collected in 100 ml bakers in order to conduct microscopic and turbidimetric analysis. The obtained samples allowed us to conduct further analysis of concentration of oil in them. When the steady state was reached, it means when the pressure varied by less than 0.01 bar (equivalent $10^3$ Pa), we started to pump water through bed. At this time the elution process began. At this stage we continued to measure the pressure difference at the inlet and we continued to collect eluted liquid. We stopped pumping of water when the steady state was reached again. The emulsion and water were pumped with the same volumetric flow rate of $6.5 \cdot 10^{-6}$ $m^3/s$ during the entire process.

During the experiments we focused on study of the influence of porous bed fraction and its length on the process of emulsion and water flow through porous bed. For the first experiment regarding the different porous bed fraction we prepared three different beds of microspheres with certain diameter ranges and followed the procedure presented in previous paragraph. The properties of porous media used in experiment are shown in Table 1. The second part of experiment was conducted in order to check the influence of bed length on the process of emulsion flow and elution from porous bed The experiments were conducted for the porous bed with the parameters that are shown in Table 1. For all experiments, at the beginning, we used oil-in-water emulsion with 5% concentration.

Table 1

Properties of porous media used in experiments

| Bed type | Particle size range [μm] | Porosity $\varepsilon$ | Mass [kg] | | |
|---|---|---|---|---|---|
| | | | $L_z$ = 0.2 m | $L_z$ = 0.3 m | $L_z$ = 0.5 m |
| Glass microspheres | 90–150 | 0.33 | 0.736 | 1.103 | 1.845 |
| Glass microspheres | 150–250 | 0.34 | – | 1.090 | – |
| Glass microspheres | 200–300 | 0.34 | – | 1.088 | – |

In order to better understand the phenomena of permeability reduction in porous bed we decided to conduct additional experiment concerning pressure drop during flow of tap water through porous bed. The methodology of it was similar to the previous experiments, but in this case through the clean bed only tap water was pumped. In experiment we measured pressure drop, which allowed us to calculate permeability according to the equation (1).

The turbidimetric analysis of collected liquid samples was performed with TurbiscanLab® apparatus delivered by Formulaction Company (France) in order to determine concentration of oil phase. The device scans the samples with near-infrared light. The TurbiscanLab® is equipped with two signal detectors: transmittance receiver that analyzes the light that goes through the sample and detector that receive back scattering light. The obtained results show the percentage of transmitted light (*T*%) and backscattering (BS%) in function of height of sample (*h*). The method of oil concentration determining in the samples of emulsion, which flowed out of the deposit, was developed on the basis of turbidimetric and microscopic tests, presented by LEMARCHAND et al., 2003, SĘK et al. 2011.

Emulsion structure (ie. oil droplets amount and size distribution) was examined with equipment used for analysis of microscopic images The system consisted of optic microscope Alphaphot-2 from Nikon, connected to digital video camera Panasonic GP-KR222 and computer used to analyze and data acquisition. For later droplet diameter analysis we used computer program SigmaScan PRO 5.

## Results and Discussion

### Microscopic analysis of emulsion droplet size and distribution

In order to correctly analyze the influence of porous bed parameters on emulsion flow through it, it is necessary to determine what parameters influence its flow. Especially important is emulsion concentration and diameter and size of droplets in it. Microscopic picture of base emulsion with 5% oil concentration used in experiments is presented in Figure 2. The system presented in Figure 2*a* is stable oil-in-water emulsion, with oil droplet size distribution showed in Figure 2*b*.

As it was mentioned, the computer program for image analysis helped us to determine the distribution of droplet size that were present in emulsified system. It also allowed us to calculate frequency of occurrence of certain oil droplets diameters, such as presented in Figure 2*b*. As it might be noticed the droplets with diameter 3–4 μm were prevailing and counted for about 40% of all drops. The diameter of emulsion droplets generally didn't exceeded 60 μm.

Additionally for calculation of mean droplet size, we used Sauter equation as following (2).

$$d_s = \frac{\Sigma n_{ei} {d_{ei}}^3}{\Sigma n_{ei} {d_{ei}}^2} \tag{2}$$

According to the equation (2) the mean drop size of equalled to 3.58 μm.

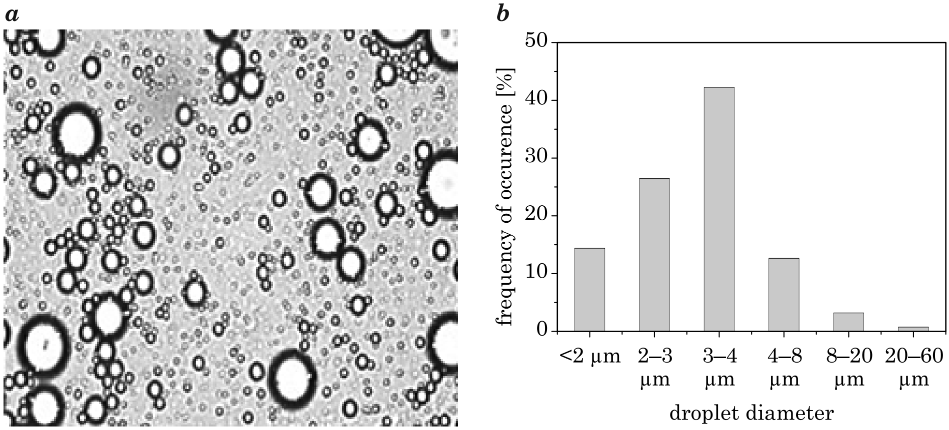


Fig. 2. Oil-in-water emulsion with 5% concentration used in experiments (*a*), droplet size distribution diagram for prepared system (*b*)

## Flow resistance and concentration of emulsion change during the process of emulsion flow and elution

The results of experiments concerning pressure drop and emulsion concentration at outflow are showed in the form of graphs. In Figure 3*a* and 4*a* the inlet pressure $\Delta p$ versus time is presented. In Figure 3*b* and 4*b* the concentrations $c_w$ of emulsion leaving the porous bed versus time are shown for $L_z = 0.3$ m.

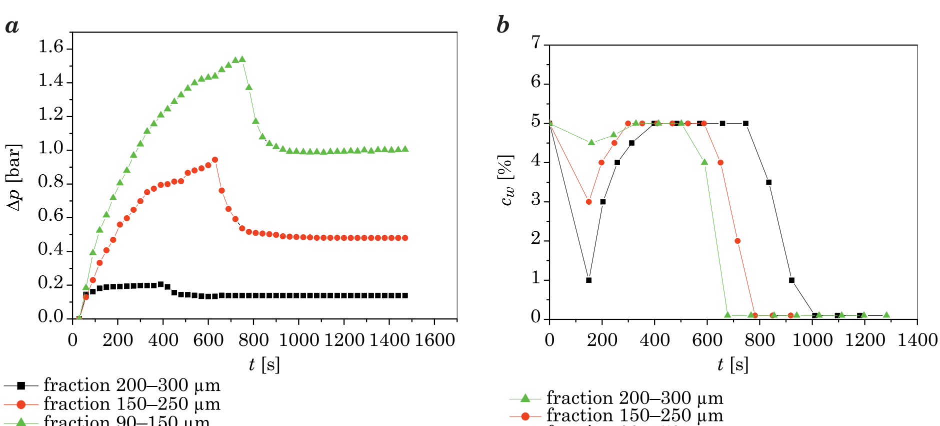


Fig. 3. Pressure difference versus time for different porous bed fractions (*a*), emulsion concentration versus time for different porous bed fractions (*b*)

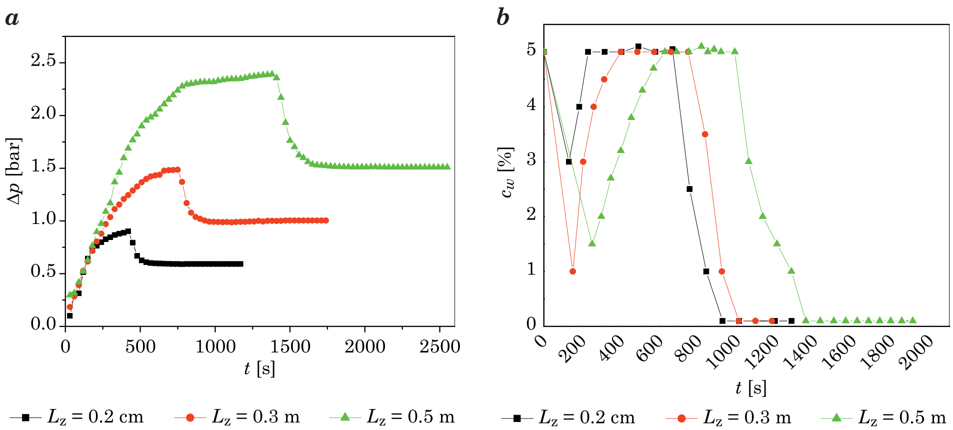


Fig. 4. Pressure difference versus time for different porous bed length (*a*), emulsion concentration versus time for different porous bed fractions (*b*)

The time of switch from emulsion flow to elution with water differ depended on which porous bed size was used. For bed with 90–150 μm – it was 710 s, while for 150–250 μm – 584 s and for 200–300 μm – 392 s. The time of change was also different for various bed lengths. Accordingly for 0.2 m it was 410 s, for 0.3 m the time equaled to 710 s and for 0.5 m – 1331 s.

The fraction size has direct impact on the size of free pores spaces. Therefore the emulsion flow is greatly influenced by this parameter, as it can be observed in Figure 3*a*. In case of bed with larger microspheres size, the flow resistance was smaller. Therefore, as it is presented in Figure 3*b* the significant drop of emulsion concentration was not observed in the first phase of experiment. Contrary, in case of porous bed with smaller diameter the situation was different. In this situation the flow resistance was higher, as can be seen in Figure 3*a*, and the retention mechanism was clearly observed, which can be concluded from concentration of emulsion drop observed in Figure 3*b*. From Figure 3*a* it can be also noted that the steady state was reached faster for the porous bed with larger fraction. It is true both for emulsion flow and its elution with water. This was caused by the fact that porous media with bigger fraction had less pores with sizes that were similar to the diameter of oil droplets in prepared emulsion. Therefore fewer spaces could be blocked by oil droplets.

The process of emulsion retention in porous structure depends also on the length of porous bed. To show how this parameter influences the emulsion flow and elution from porous media we conducted experiments with three tubes with different lengths. The results of experiment are presented in Figure 4*a* and Figure 4*b* for particle size range 90–150 μm.

In Figure 4*a* it is possible to observe that for the longer bed was used, the bigger flow resistance was noted. Time that was needed to reach steady state was also dependent from this parameter. For every tube the time until steady state was reached varied. In case of shorter tubes, it means 0.2 m and 0.3 m the steady state was reached in shorter time, than for 0.5 m length bed. It was caused by the fact that in longer pipes there are more free pores that can be blocked by oil phase. Therefore the time of pores clogging is also longer, as can be seen from emulsion concentration drop in Figure 4*b* – for 0.5 m length the drop in emulsion concentration is observed after 300 s, while for 0.2 m after 150 s. During the process of elution with water the time until the steady state was reached was also dependent from porous bed length.

## The analysis of porous bed permeability change during emulsion flow and elution

Knowing the pressure changes during the flow as well as certain parameters such as flow rate, porous bed length and its diameter, it was possible to determine permeability changes of bed in time of process of emulsion flow and elution. The graphs in Figure 5 present the changes in permeability of bed calculated according to the equation (1).

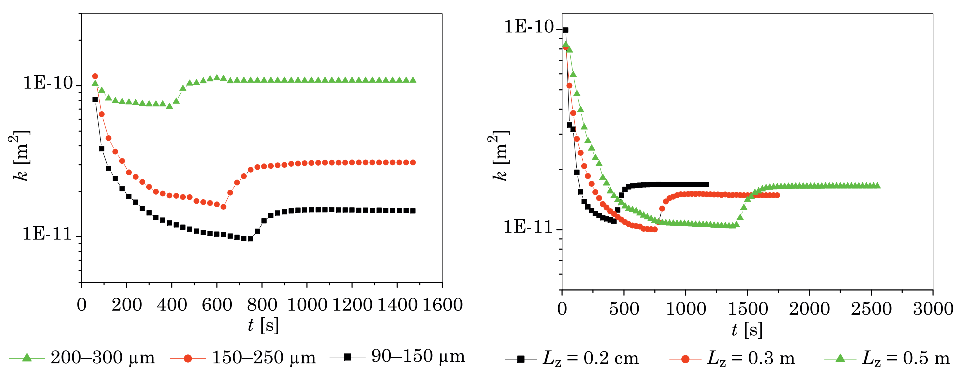


Fig. 5. Permeability change versus time for different porous bed fractions during experiment ($Lz$ = 0.3 m) (*a*), permeability change versus time for different porous bed lengths – particle size range 90–150 μm (*b*)

As it is possible to observe from Figures 5*a* and 5*b* the permeability of porous bed decreases with time. This drop is dependent on the particle size range used in experiments. The smaller particle size were used the lower value of permeability is observed. This confirms the phenomena of oil droplets retention that is more intense in porous bed with smaller microspheres. However, for porous bed with different length the values of permeability at the end of emulsion flow experiments are similar. The difference in this case is observed in terms of time that is needed to reach the steady state-as shown in Figure 5*b*.

During analysis of elution process it is possible to note the increase in permeability factor for all cases. Eventually during elution with water the constant value of permeability is reached. In order to better understand and extend the capability of understanding the phenomenon that occurred during experiments we decide to conduct experiments concerning the flow of water only through the porous bed with three particle size fractions. Then we calculate the value of permeability for the flow of water. The results are presented in Table 2.

Table 2

Permeability of porous bed used in experiments

| Particle size [μm] | Permeability [m$^2$] | | |
|---|---|---|---|
| | $L_z$ = 0.2 m | $L_z$ = 0.3 m | $L_z$ = 0.5 m |
| 90–150 | $2.15 \cdot 10^{-11}$ | $2.02 \cdot 10^{-11}$ | $2.27 \cdot 10^{-11}$ |
| 150–250 | – | $4.58 \cdot 10^{-11}$ | – |
| 200–300 | – | $1.22 \cdot 10^{-10}$ | – |

Considering the data from Table 2 as well as Figure 5 it can be observed that permeability for water flow are higher than values of permeability for bed that was previously saturated with emulsion. This is true for every analyzed case in our experiment. Therefore it is possible to state that the phenomena of trapping of oil in porous bed occurred. In case of porous media with smaller microspheres, permeability for bed length of 0.3 m was smaller by 27% when comparing to permeability in experiment only with water (from value $2.02 \cdot 10^{-11}$ m$^2$ to $1.48 \cdot 10^{-11}$ m$^2$). Meanwhile for porous bed particle size range 200–300 μm the value of permeability decreased by only 11% (from $1.22 \cdot \cdot 10^{-10}$ m$^2$ to $1.08 \cdot 10^{-10}$ m$^2$). Regarding the influence of length of porous bed on oil retention mechanism it is possible to state that permeability of clean bed was 25% higher comparing to bed saturated previously with emulsion.

## Conclusion

The conducted work delivered valuable experimental data that concern influence of parameters of porous bed on process of emulsion flow and elution. The work quantitatively determines the flow resistance during flow and concentration changes of fluid at the outlet from bed. The analysis of change in concentration in permeability of bed indicates the gradual change of permeability that suggests the phenomena of oil retention inside the porous structure. After comparison of permeability for water flow that was previously saturated with emulsion, with the permeability for water flow through clean bed it was possible to determine the retention rate of oil phase in porous structure. The analysis of changes in emulsion concentration that leaved the bed showed that in the first phase of process the concentration of emulsion rapidly decreases. It was caused by retention of big droplets in porous structure. When all spaces where droplets can be trapped are filled, the flow takes place through the main paths of flow. This is the reason that concentration of effluent emulsion increase and ultimately reaches the original value.

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