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ANALYSIS OF PROCESS OF EMULSIONS TRANSPORT IN HYDROPHILIC/OLEOPHILIC GRANULAR POROUS MEDIA DRIVEN BY CAPILLARY FORCE

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Abstract

The research focuses on the issues concerning a process of multiphase liquids transport in granular porous media driven by the capillary pressure. The current publication is meant to introduce the results of experimental research conducted to evaluate the kinetics of imbibition and emulsions behavior inside the porous structures. Moreover, the influence of dispersed phase concentration and granular media structure on the mentioned process was considered. The medium imbibition with emulsifier-stabilized emulsions composed of oil as the dispersed phase in concentrations of 10 vol%, 30 vol%, and 50 vol%, was investigated. The porous media consisted of oleophilic/hydrophilic beads with a fraction of 200–300 and 600–800 μ m. The experimental results provided that the emulsions imbibition in such media depended strong on its structure compare to single-phase liquids. The increase of the dispersed phase concentration in a sorptive medium. The concentrations of the imbibed emulsions exceeded their initial values, but reduced with permeants front raise in the granular structures that can be defined as the influential factor for wicking process kinetics.

Symbols

- A_s cross-section area of a porous medium, m²,
- D_f pore fractal dimension, –,
- d'_{b} average diameter of beads in a granular medium, m,
- d_t diameter of a tube, m,
- f_n fragment of a porous medium, m,

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- h_c - height of a liquid front in a capillary, m,
- h_{im} - imbibed liquid height, m,
- h_m - height of a porous medium, m,
- maximal height of an imbibed liquid, m, $h_{\rm max}$
- imbibed liquid mass, kg, m_{im}
- L_s K- distance in the flow direction, m,
- proportionality coefficient, m²,
- $k_{\rm kc}$ Kozerny constant, -,
- average pore radius of the investigated porous medium, m, r_{av}
- radius of a capillary, m, r_c
- hydraulic radius, m, r_h
- r_{max} maximum pore radius, m.
- radius of pore, m,
- $r_p \\ S_{in}$ initial saturation, -,
- S_{im} - medium saturation after imbibition, -,
- time of imbibition, s, t_{im}
- initial dispersed phase concentration, vol%, φ_d
- dispersed phase concentration of an imbibed emulsion, vol%, φ_{im}
- porosity of a medium, -, ε
- density of a wetting liquid, kg/m³ ρ
- pore shape parameters, -, δ
- viscosity of permeant, Pa·s, -, · η
- σ - surface tension, N/m,
- θ - contact angle between a penetrating liquid surface and pore surface, 0°,
- angle formed between an inclined tube and free liquid surface, 0°, W
- average length of a permeant path, m, λ_{ρ}
- tortuosity of the pores in a medium, -,

HLB - hydrophilic-lipophilic balance.

Introduction

A porous medium consists of a matrix as skeletal portion and an interconnected or unconnected network of capillary channels, which are usually characterized by an irregular shape, the different size and structural organization. One type of porous structures is the granular media, which can characterized with the different porosities, dimension and shape of particles (KOWALSKI 2004, STRZELECKI et al. 2008, MASOODI, PILLAI 2010). The representatives of such media can be the void systems of soil, sands and sandstones, sorbent composed of silica gel, concrete specimen composed of vermiculite, etc. (CHATTERJEE, GUPTA 2002, MASOODI, PILLAI 2010, CARBAJO et al. 2015). Over the last decades, a lot of researches have focused on the liquids flow through porous structures. The process of liquids transport driven by the capillary pressure and balanced by viscous drag force and gravy acceleration, is known in literature as the spontaneous imbibition or wicking (KOWALSKI 2004, MASOODI et al. 2007, MASOODI, PILLAI 2010). The suction pressure derives as a result of pore walls wetting with a permeant due to interchanging of their surface energies. The imbibition occurs in case when an adhesion predominates a mutual force of attraction between molecules in a permeating liquid (CHATTERJEE et al. 2002, MASOODI et al. 2007, MASOODI, PILLAI 2010).

The prediction of the imbibition process in porous media is of the practical importance thanks to its relevance as a fundamental phenomenon in numerous industrial technologies and nature. Presently, there has been a continuous discussion concerning the single-phase liquids wicking in the different porous media and its mechanisms (SIEBOLD et al. 2000, ZHMUD et al. 2000, HAMRAOUI, NYLANDER 2002, XUE et al. 2006, MASOODI et al. 2007, MASOODI, PILLAI 2010).

The equation firstly suggested the analytical explanation of a liquid rise in the capillary is known as the Lucas–Washburn equation, but its relevance is extended to describe a process of the porous media imbibition. However, the model application for such a reason has a set of limitations connected with disregard of the influential factors (ZHMUD et al. 2000, FRIES et al. 2008, MAGGI et al. 2012). On the other hand, there are the numerous efforts to modified the mentioned equation due to capturing the constitutive issues such as a mean pore radius (HAMRAOUI, NYLANDER 2002, BENAVENTE et al. 2002, MASOODI et al. 2007), tortuosity and shape of pores (BENAVENTE et al. 2002, YU 2008, ZHAO, LI 2009, CAI et al. 2012, 2014), changes of contact angle (HAMRAOUI, NYLANDER 2002, SIEBOLD et al. 2000, CHEBBI 2007, DIGILOV 2008), roughness of pores wall (MARTIC 2003, LIU 2014), surface tension (HAMRAOUI, NYLANDER 2002), and a permeant viscosity (SIEBOLD et al. 2000, XUE et al. 2006, DIGILOV 2008).

The influence of the dynamic contact angel was studied in the work of SIEBOLD et al. (2000) by means of model experiments with capillary rise of the organic single-phase liquids in a glass cylinder tube and in columns filled with powders.

The experiments concerning water and ethanol rise in the vertical glass capillaries differed with a radius (0.4–1 mm) was performed and analysed in the publication of HAMRAOUI and NYLANDER (2002). The obtained results allowed to represent a model, which considered the energy dissipation caused mainly by gravity and surface tension during a liquid rise. To account these influential factors, the introduction of a retardation coefficient was proposed (HAMRAOUI, NYLANDER 2002).

Another model for description of the single-phase liquid penetration rising in a capillary considered the hydrostatic effects (XUE et al. 2006). The short-time and long-time analytical solutions for the dynamics of a fluid penetration in a capillary was also discussed (CHEBBI 2007). MASSODI et al. (2007) compared the adequacy of capillary and energy balance models with Washburn equation using independently measured parameters. The effect of a permeant viscosity and dynamic contact angle on the liquid rise dynamics in a vertical capillary was investigated experimentally and debated in the work of DIGILOV (2008). To conclude, a majority of the reviewed previously models considered mostly interaction on the border of solid-liquid and gas-liquid phases. The model initially based of LWE and modified by the correction factors relating to porous media structure such as the pore shape, i.e. roundness, its effective radius, and tortuosity was used to predict the weight rate (BENAVENTE et al. 2002). There is another approach related to the spontaneous imbibition of a wetting permeant into gas-saturated porous media consisting of a complex structure of pores based on their fractal geometry (YU 2008, ZHAO, LI 2009, CAI et al. 2012, 2014). The mentioned models allowed to characterize the imbibition process in a variety of the porous media, i.e. rocks, soil, fibrous material and bio-structures, etc. (BENAVENTE et al. 2002, YU 2008, ZHAO, LI 2009, CAI et al. 2012, 2014).

To conclude spontaneous imbibition in porous media is a complex physical process, which depends on numerous parameters and in the recent publications there is a great deal of the mathematical models to predict this phenomenon. However, the issue regarding the porous structure imbibition with liquids composed with several phases has not yet been fully investigated experimentally and described by theoretical models.

Thus, the present work focuses on a study of the kinetics of granular structures imbibition with two-phase liquids such as oil-in-water emulsions. In the current publication, there is a discussion of the hypothesis that the porous media imbibition with the multiphase permeants differs considerably from the process with single-phase liquids, and its validation using the obtained experimental results. Such a deviation can relate to the obstruction of penetration path by the dispersed phase. The dependence of imbibition rate on the emulsions properties, i.e. the fractional composition, viscosity, and structure of the granular porous media was also currently considered.

Materials and methods

In these experiments, the object of investigation was a granular medium consisting of spherical hydrophilic/oleophilic grains. The beads were produced and obtained from "Alumetal-Technik" (Lodz, Poland). The porous structures differed by a size of the particles diameter, which was in a range of 200-300 and $600-800 \mu$ m. The bulk density was equal to 1621 ± 5.2 and $1728\pm1.2 \text{ kg/m}^3$. The porosity of prepared media was 0.36 ± 0.011 and 0.37 ± 0.013 , respectively.

The wicking liquids were oil, distilled water and their stabilized emulsions with the different dispersed phase concentrations, i.e. 10 vol%, 30 vol%, and 50 vol%. The oil-in-water emulsions were prepared by means of mechanical stirring during 600 s. The vegetable oil was produced by EOL Polska Sp. z.o.o., Poland. The used non-ionic emulsifier was Rokacet O7 (PCC Exol SA, Poland) composed of ethoxylated oleic acid. The fraction of the emulsifier equalled 2 vol%. The emulsions components properties are given in Table 1.

Physicochemical characteristics of the basic liquids $(1=23\pm1^{\circ}\text{C})$					
Component	Viscosity [mPa·s]	Surface tension [mN/m]	Density [kg/m ³]	HLB* [–]	Contact angel [degs]
Dispersed phase	53.2 ± 0.4	32 ± 1.7	922 ± 1.6	-	22.4±2.1
Emulsifier	50.2 ± 0.6	36 ± 1.8	908 ± 2.7	10.6	24.3±2.7

Table 1

* HLB – hydrophilic-lipophilic balance

The density of liquids was measured using the pycnometric method. The prepared dispersions differed from each other by density, thus, for 10% emulsion it was 990.1 ± 1.31 kg/m³, in case of 30% one, it was 973.9 ± 1.43 kg/m³. For emulsions with the dispersed phase concentration of 50%, density equaled 959.1 ± 2.18 kg/m³. The surface tension was defined by means of a tensiometer KRÜSS K100 (KRÜSS GmbH, Germany). The viscosity of permeating liquids was measured by a shear rheometer Bohlin CVO-120 (Malvern Instruments, UK).

The kinetics of the imbibition process evaluated as changes of the imbibed emulsion mass and the height of its front penetration versus time. They were investigated using the classical wicking test, during which the porous medium was directly submerged in a liquid. The used experimental stand is shown schematically in Figure 1.



Fig. 1. Scheme of the experimental stand: I – reservoir with a liquid, 2 – tube with a diameter d_t , 3 – dry granular bed in a tube, 4 – balance, 5 – fixator of a tube

The process was observed and followed till the equilibrium state achieving when the mass changes became negligible small, i.e. less than 0.001 g per 300 s. After the imbibition test, in the obtained samples the concentrations of the imbibed emulsions were defined at certain heights of liquids penetration, e.g. $f_1, f_2 \dots f_n$, as in Figure 1. The nephelometrical method was used to measure such concentrations by means of the optical analyser TurbiscanTM LAB (Formulaction, France). The mentioned methods have been reported in details elsewhere (SEK et al. 2015, SHTYKA et al. 2016).

All experiments were performed at the temperature of $23\pm1^{\circ}$ C and atmospheric pressure. Three independent replications were conducted for each type of liquids and their mean value was considered as the obtained result.

Results and Discussion

Changes of imbibition kinetics versus time

The kinetics of imbibition process as mentioned previously, was characterized as the changes of wicked emulsion mass m_{im} and height of its penetration h_{im} as a function of time t_{im} . The obtained results on the mass variation are represented in Figure 2. The highest mass of the imbibed liquid was observed for water, i.e. 25.2 10^{-3} kg. Among the investigated two-phase liquids it was obtained in case of emulsions with the lowest dispersed phase concentration, i.e. $\varphi_d = 10$ vol% for both granular media (Figs. 2a and 2b). In contrast, the lowest one was derived for oil, i.e. ~9.5 10^{-3} kg (Fig. 2). The tendency of imbibition was the similar for both investigated granular media, but higher values of the mass were obtained in case of a bed with particles diameters of 200–300 µm. Consequently, the increase of beads size caused the decrease of imbibed mass. Thus, for emulsions the mass value reduced twice, for water by about 30% and only for oil such difference was negligibly small (Fig. 2).

To summarize, the emulsions imbibition in the granular media considerably differed from the process of their pure components wicking, and depended strongly on the hydraulic radius of pores. The difference between the masses of imbibed emulsions was lower than 1.1 g, thus, the initial concentration of the dispersed phase is considered as a less significant factor.

Figure 3 represents the comparison of the obtained results related to the height changes versus time. In this case, the beads and consequently, pores radius were proved to be the influential parameter. As shown on the graph, the highest values were observed for a bed with smaller particles diameter, i.e. $200-300 \mu m$ (Fig. 3). The maximal value of experimental data error was equal to 3.8% for a medium with beads diameter of $200-300 \mu m$, and up to 10% for another type.



Fig. 2. Changes of wicked liquid mass m_{im} versus time t_{im} during emulsions imbibition in granular media with beads diameter of: $a-200{-}300~\mu{\rm m},~b-600{-}800~\mu{\rm m}$

The maximal height of emulsions penetration depended on the initial dispersed phase concentration in case of granular diameter of $200-300 \ \mu m \ (d_1 \ in \ Fig. 3)$. Thus, for 10% emulsion such value was the highest and equalled 0.083 m, for 30% it was about 11% lower, i.e. 0.074 m, and 0.069 m in case of 50% emulsion. The data also showed that for another porous medium with a particles fraction of 600–800 μm , such difference between the heights of emulsions fronts was



Fig. 3. Changes of an imbibed liquid height h_{im} versus time t_{im} during emulsions imbibition in granular media with beads diameter of: $d_1-200-300~\mu\text{m},\,d_2-600-800~\mu\text{m}$

insignificant, and the imbibition process achieved more quickly the equilibrium state (d_2 in Fig. 3). Analysing the data presented in Figures 2 and 3, it is possible to conclude that the structure of a granular medium can be admitted as one of the main factor defining a trend of the multiphase liquids imbibition.

Approximation of the experimental data on the emulsion imbibition

The accuracy of the proposed in literature models to describe the liquids wicking in porous structure and possibility of their application to predict the dispersions penetration were also discussed in the current work. The obtained experimental data on emulsions imbibition in the granular media were approximated by equations mentioned previously in introduction.

The classical Lucas-Washburn equation is frequently used to describe the capillary rise in one cylinder or their bundle, but its application is extended to characterize imbibition process in porous structures (KOWALSKI 2004, MASOODI et al. 2007, FRIES, DREYER 2008, MASOODI, PILLAI 2010). In case when the gravity force is small enough to be negligible in comparison with the capillary force ($p_c >> \rho g h_c$), the equation has the following form (KOWALSKI 2004, MASOODI et al. 2007):

$$h_c^2 = \frac{r_c \sigma \cos\theta}{2\eta} t \tag{1}$$

where:

- h_c the height of a liquid front in a capillary,
- t the time of rise,
- η the viscosity of a permeant and σ is its surface tension,
- r_c the radius of this capillary,
- $\theta~-$ the contact angle between a penetrating liquid surface and pores surface.

The expression for a liquid height h_f rise for the polymer wicks proposed in the publication of MASSODI et al. (2007). This equation considers such peculiarity of a porous medium as the tortuosity of pores in the investigated medium τ , and it can be written as:

$$h_f^2 = \frac{r_h \sigma \cos\theta}{2\eta \tau^2} t \tag{2}$$

where:

 r_h – the hydraulic radius.

The hydraulic radius is calculated according to Kozeny-Carman theory. Thus, the hydraulic radius can be calculated as (KOWALSKI 2004):

$$r_h = \frac{1}{6} d_b \left(\frac{\varepsilon}{1 - \varepsilon} \right), \tag{3}$$

where:

 d_b – the average diameter of beads in a granular medium.

The tortuosity can be calculated as a ratio of the average length of a permeant path λ_e to the distance in flow direction L_s (DUDA et al. 2011):

$$\tau = \frac{\lambda_e}{L_s} \tag{4}$$

In the discussed publication, the capillary model was also presented to predict a permeant front height rise considering the porosity ε , the proportionality coefficient *K*, known also as the permeability or hydraulic conductivity parameter. The represented model neglects the gravity effect, and has the following form as (MASOODI et al. 2007):

$$h_f^2 = \frac{4K\sigma\cos\theta}{\varepsilon\eta r_p}t$$
(5)

where:

 r_p – the mean pore radius.

The permeability regarded in this equation can be calculated using Kaviany model which considers the porosity of investigated bed and average diameter of beads. Thus, this parameter is expressed as (KAVIANY 1995):

$$K = \frac{\varepsilon^3}{36k_{kc}(1-\varepsilon)^2} d_b^2 \tag{6}$$

where:

 k_{kc} – the Kozerny constant, which is equal to 5 for a bed packed with beads (KAVIANY 1995, KOWALSKI 2004).

The permeability of a granular medium can also be determined according to the following equation (RUMPF, GUPTE 1975):

$$K = \frac{\varepsilon^{5.5}}{5.6} d_b^2 \tag{7}$$

Fries and Dreyer analyzed the capillary rise of a wetting liquid in a cylinder or porous medium considering the gravity term. The equation has the following form (FRIES, DREYER 2008):

$$h(t) = \frac{a}{b} \left(1 - e^{-(b^2 t/a)} \right)$$
(8)

and related to the spontaneous imbibition in porous structures by means of coefficients, which are expressed as:

$$a = \frac{\sigma r_p \cos \theta}{4\eta} \tag{9}$$

$$b = \frac{\rho g r_p^2 \sin \psi}{8\eta} \tag{10}$$

where:

 ψ – the angle formed between an inclined tube and free liquid surface.

There is a group of the mathematical models that describe the kinetics of spontaneous imbibition in term of mass changes with time (BENAVENTE et al. 2002, LI, HORNE 2004, MASSODI et al. 2007, CAI et al. 2012, 2014). The increase of an imbibed mass can be represented in relation to the height of liquid front h_f according to the equation (MASSODI et al. 2007):

$$m = \varepsilon \rho \pi r_p^2 h_f \tag{11}$$

BENAVENTE et al. (2002) derived a model that considers the imbibition weight related to the structural parameters of porous medium, such as tortuosity τ ,

pore shape parameters, denoted as δ , and the average pore radius r_{av} of the investigated porous bed. The equation can be represented as:

$$m^{2} = \frac{A_{s}^{2}\varepsilon^{2}\rho^{2}r_{av}\delta\sigma\cos\theta}{2\eta\tau}t$$
(12)

where:

 $A_{\rm s}-{\rm the\ cross-section\ area of\ a \ porous\ medium\ (Benavente et al.\ 2002).}$

The tortuosity of a granular medium can be calculated analytically using Du Plessis-Maslivah equation (DU PLESSISS, MASLIVAH 1991):

$$\tau = \varepsilon / \left[1 - (1 - \varepsilon)^{2/3} \right]$$
(13)

Thus, the tortuosity was equal to 1.4 that was also confirmed experimentally due to microscopic images analysis of porous structure.

The influence of the investigated porous media saturation with a wetting liquid was taken into consideration in Li-Horne equation (LI, HORNE 2004). It is introduced as the difference between the initial saturation S_{in} and the medium saturation after imbibition, S_{im} . The equation can be written as follows:

$$m^{2} = \frac{2A_{s}^{2}\rho^{2}K\varepsilon(S_{im} - S_{in})}{\eta}\frac{\sigma\cos\theta}{r_{p}}t$$
(14)

The porous bed might be not fully saturated by a wetting permeant during the spontaneous wicking due to gas phase entrapment or the partial connectivity of pore voids, i.e. in rocks, soil, building materials, concrete, etc.

CAI et al. (2012, 2014) proposed an approach to predict the spontaneous imbibition of a wetting liquid on the base of fractal geometry. The mass changes versus time is expressed in a form of:

$$m^{2} = \frac{A_{s}^{2} \rho^{2} \varepsilon^{2} r_{\max}}{1 - \varepsilon} \frac{\sigma \cos \theta}{2\eta \tau^{2}} \frac{2 - D_{f}}{3 - D_{f}} t$$
(15)

where:

 $r_{\rm max}$ – the maximum pore radius, which can be calculated as a ratio (YU 2005, CAI et al. 2014):

$$r_{\max} = \frac{r_{av}}{\left(D_f / (4 - D_f)\right)^{1/4}}$$
(16)

where:

 D_f- the pore fractal dimension (XU, YU 2008, CAI et al. 2012, 2014).

The graphs plotted in Figure 4 introduce the comparisons between the experimental data and the predictions by equations presented in the current work. It can be noted that Equation 1 describes more precisely experimental data on the height changes than others, but till $t_{im} = 600$ s. Further, a trend towards lower than the equation prediction (Fig. 4*a*).



Fig. 4. Comparisons of the emulsion imbibition process predicted by the models and experimental data: a – height h_{im} changes versus time $t_{im},$ b – mass m_{im} changes versus time t_{im} . The parameters used are A_s = 9.62 cm², ρ = 0.959 g/cm³, σ = 36 mN/m, r_h = 22.9 μ m, η = 87.35 mPa·s, and θ =30°

The data in Figure 4*b* were obtained both experimentally and analytically, and represent the imbibed mass changes versus time. More accurate approximation was observed in case of Equation 12, but till $t_{im} = 1,800$ s. Thereafter, the mass increase slowed down and the imbibition process achieved the equilibrium state (Fig. 4*b*). To summarize, the trends of the experimental data differed from those obtained analytically by mean of models.

Peculiarities of imbibition process in case of emulsions

The results on viscosity changes with increase of the dispersed phase concentration are shown in Figure 5. Its increase caused changes of emulsion viscosity and its behavior at the different shear rates. The investigated disperses behaved as the Newtonian liquids and flow curves are approximated by a straight line if the dispersed phase concentration is less than 30%. Whereas other emulsions with higher concentrations behaved as the non-Newtonian liquids (Fig. 5).



Fig. 5. Viscosity of the investigated emulsions at the different shear rates γ

The results of emulsions concentration changes versus the height of their front penetration in the porous media are plotted in Figure 6.

According to the experimental data, the imbibed emulsions revealed a tendency to decrease concentration with the increase of their penetration height in both granular beds. To compare, the highest dispersed phase concentrations were observed for porous media with beads diameter of $600-800 \mu m$, whereas the height of penetration in this case was the lowest one, i.e. less than 0.04 m (Fig. 6). It related to pores structure, i.e. hydraulic radius, which was wider compared to another type of medium, but it was still enough to induce the capillary forces.

At $h_{im} \leq 0.02$ m, the concentration of imbibed emulsions was recognized as maximal in both experiments. Furthermore, the concentration of imbibed



Fig. 6. The dispersed phase concentration φ_{im} changes with the height h_{im} for emulsions penetration in granular media with beads diameter of: $a - 200-300 \mu$ m, $b - 600-800 \mu$ m

emulsions exceeded their initial values φ_d , the exception was observed for 50% emulsions at 0.03 m $\leq h_{im}$. As shown in Figure 6, the obtained concentrations were lower for emulsions with φ_d of 10 vol%.

Conclusions

The imbibition of multiphase liquids in granular media differs significantly from the same process with single-phase liquids. There was a strong influence of the pores structure on wicking process that revealed the increase of the imbibed mass and penetration height with the decrease of particles diameter in a granular bed. Though such a factor was of less significance for the investigated single-phase liquids, especially for oil.

The highest mass of imbibed emulsion and penetration height were obtained for emulsions with the lowest dispersed phase concentration and consequently, viscosity. The increase of oil content in an emulsion was a reason of the insignificant reduction of the imbibed mass in both investigated media. The difference between the heights of permeant penetration was derived only in case of porous media with beads diameter of $200-300 \mu m$.

The comparisons between the obtained experimental data and those predicted by the equations shown that they disallow to describe the emulsions imbibition in porous structures with the sufficient accuracy. The limitations might be related to the several aspects: i) emulsions are recognized as heterogeneous multiphase systems, consisting of immiscible components with different physicochemical properties, and ii) their composition changes during wicking process.

On the one hand, the concentrations of imbibed emulsions exceeded their initial value at $h_{im} \leq 0.02$ m. On the other hand, there was a general tendency of concentrations decrease with front raise in both granular media. The highest value of the dispersed phase was observed for a bed composed of 600–800 µm beads. Consequently, higher radius of pores allows to prevent the penetration paths obstruction with emulsion droplets. The effect of permeants concentration increase was the viscosity changes, and as a result, the lowest height of penetration was observed.

The experimental data on the spontaneous imbibition discussed in this publication, can facilitate understanding of two-phase liquids behaviour inside the granular media during their transport by the capillary force. The obtained results can be appropriate to predict and describe the imbibition of multiphase liquids in porous media with different particles sizes, for example sorbents and such structures as soils, sand, rocks, and others.

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