Separation of mineral oil droplets using polypropylene fibre bed coalescence

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Abstract
This paper investigates the separation possibilities of model emulsion oil-in-water using polypropylene fibre bed coalescence. Experiments were carried out over a wide range of physicochemical characteristics of mineral oils, bed permeability and operating fluid velocities. The aim of this study was to analyze the influence of the dispersed oil phase nature and of the bed geometry on the separation efficiency. From the obtained results, it can be concluded that polypropylene fibers in the broadest studied range of bed permeabilities and fluid velocities, effectively separate oil that is highly polar. On the contrary, for the other two investigated oils at low values of bed permeability a region was detected in which the coalescer is incapable to operate. It has to be emphasized that the polypropylene fibres efficiently separate all three investigated oils at the highest studied bed permeability.

Keywords: oily water separation, bed coalescence, fiber material, polypropylene.

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Fibre bed coalescence is a separation technique that is used in the industry for the phase separation of the emulsion. When the emulsion passes through the filter media, the droplets of the dispersed phase form larger droplets that leave the bed enabling easy separation by gravity settling in the effluent stream. This separation technique has proved to be effective for the separation of oil-in-water emulsions, as well as for water-in-oil emulsion, if the droplets of the dispersed phase have diameters smaller than 100 µm.

For the bed formation of filter media in coalescers, the fibres with high and low surface energy can be used [1-–10]. Glass fibres, stainless steel fibres and ceramic fibres are used as high surface energy materials, whereas polypropylene, polyurethane, polyethylene terephthalate, polybutylene terephthalate, nylon and teflon are most commonly used as materials with low surface energy. For the separation of the dispersed oil, the polymer fibres are most frequently applied. Polypropylene is extensively employed in the industry due to low cost and wide production of fibres with various diameters, which are broadly available as waste. Additional reasons for the application of this material are the highly desired properties, such as: high strength and elasticity, fatigue resistance, as well as chemical stability [11–16].

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Wei *et al*. [17] analyzed the wettability of polypropylene by injecting low-viscosity oil with a micro-injector onto the surface of fibres. They concluded that polypropylene fibres had a hydrophobic surface, while the wetting angle of the investigated oil was less than 20°, indicating the affinity of this surface to oil.

Li and Gu [18] have studied the phenomenon of bed coalescence of the emulsion oil-in-water using polypropylene and nylon. They concluded that the smaller diameter of polypropylene fibres are, the greater the efficiency of the separation is. However, the authors ignored the influence of other properties of fibres, of the bed and the physical-chemical characteristics of the dispersed oil phase.

Clayfield *et al*. [19] have investigated the mechanisms of fibre bed coalescence for polypropylene and other polymeric materials. The experiments were carried out using the vertically upward orientation of the emulsion flow. They concluded that the coalescence efficiency strongly depends on the mechanisms of droplet coalescence on the surface of the fibres. Hughes [20] analyzed the emulsion separation of crude oil and kerosene in the water using the bed formed of pure polypropylene and polypropylene fibres coated with a layer of silane. He concluded that the efficiency of separation dominantly depends on the interaction between drops and fibres.

Kulkarni *et al.* [16] have added polypropylene nanofibres to the bed of glass fibres. They investigated the separation efficiency of water from oil and concluded that the polypropylene fibres due to different values of the wetting angle in relation to the glass fibres consi-

derably influence the separation efficiency of the water droplets. The same authors [3] examined the possibility for separation using the filter media composed of glass, polypropylene and polyester fibres. They concluded that this combination of filter media can provide high coalescence efficiency.

In this study the separation possibilities of oil droplets in water with different dispersed oil phase nature, using polypropylene fibre bed was investigated. In addition to changing the nature of the oil phase, the bed geometry of polypropylene fibres was also varied by increasing its compression. In this way, the flexibility of the polypropylene fibres application in the coalescer was also examined.

EXPERIMENTAL

Experimental device and working conditions

Experiments were carried out using the model oil-in-water emulsion and bed coalescer of polypropylene fibres. Three mineral oils of different physicochemical characteristics were investigated as the dispersed phase. The apparatus which was used for the experimental program is shown in Figure 1. The experimental device consists of a Plexiglas tube 5 cm in diameter (1) a total length of 1.2 m. The length of the polypropylene bed (2) was 5 cm. The emulsion of oil-in-water at the temperature of 20 °C was prepared in two tanks with volume of 80 litres each (3). The oil was dispersed in water by continuous stirring with stainless steel impeller (4) at a rate of 650 rpm for a period of 45 min prior to the experiment, and continuously throughout the whole duration of the experiment. In this way the average droplet diameter of 10 µm was maintained. Transport of fluid through the apparatus was initiated by the membrane dosage pump (5). After leaving the porous bed, merging of smaller droplet to larger ones was established enabling easy separation by gravity settling and collecting in the upper part of the tube, after which oil was discharged discontinuously by a valve (6). Fluid velocity, *v*, was maintained constant for 60 min. In order to measure the effluent oil concentration, a composite sample, consisting of three individual samples, was taken after 45 min at 5-min interval at the exit of the apparatus (7). Each composite sample was stabilized by the addition of concentrated hydrochloric acid. The efficiency of coalescence was monitored *via* the effluent concentration of the dispersed phase, C_i, measured by ThermoNicolet 5700 FTIR spectrophotometer. The sample preparation for FTIR analysis was performed by extraction of the oil phase from the emulsion with carbon tetrachloride. In our previous investigations [8,21,22], if the effluent oil concentration was lower than 15 mg/l, the experiments were resumed at higher fluid velocity.

All experiments were photographed attaining valuable data that simplified the analysis of the obtained results.

Properties of the oil phase

Three mineral oils of different physicochemical characteristics, containing no additives, were investigated as the dispersed phase: naphthenic crude oil (A), naphthenic-base vacuum fraction (A4), and blended petroleum product with a high paraffinic content (P1). For the analysis of physicochemical properties of the dispersed phase numerous methods and techniques were employed in order to determine: density, viscosity, relative molecular mass, interfacial tension, surface tension, pour point, neutralization number, dielectric constant and emulsivity, Table 1. The oil density was determined by SRPS ISO 12185:2004 method, oil viscosity by SRPS ISO 3104:2003 method, relative molecular mass of oil using ASTM D 2502-67, interfacial tension by ASTM D 971 method, the surface tension was determined by stalagmometric method, pour point using SRPS ISO:3016 1997 method, neutralization num-

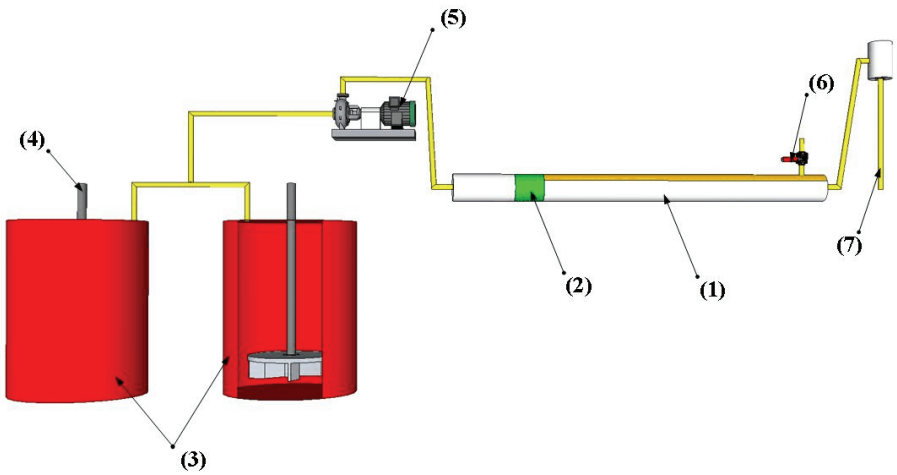

Figure 1. Scheme of experimental apparatus.

ber by SRPS ISO 6619:1994 method, the dielectric constant by EN 60247:2008 method, while emulsivity was determined by the method developed at the Central Laboratory NIS [23].

Table 1. Physicochemical properties of dispersed oil

Property	Sample		
	A	A4	P1
Density at 15 °C, kg/m^3	916.7	923.3	883.3
Density at 20 °C, kg/m^3	915.5	918.9	879.0
Viscosity at 40 °C, mPa s	43.350	168.904	10.316
Neutralization number mg KOH/l	1.42	1.71	0.13
Pour point, °C	–42	–3	+3
Interfacial tension, mN/m	18.8	30.5	32.4
Surface tension, mN/m	26.56	27.72	30.16
Emulsivity, vol.%	99.92	70.00	54.17
Dielectric constant	0.1612	0.1905	0.0645
Relative molecular mass kg/kmol	410	520	300
Distillation range, °C	150–550	315–550	320–415

Properties of the filter material

Density and the melting point were measured for the filter media, Table 2, using the pycnometer method for the density and the DSC method for the melting point.

Bed permeability, K_0, was experimentally determined using the Darcy test and presented in Table 3 [24].

Table 2. Properties of filter media

Material	Density, kg/m^3	Melting point, °C
Polypropylene	900	168.60

Table 3. Polypropylene fibre bed permeability (10^{-9} m^2)

Material	Bed permeability				
Polypropylene	K_{01}	K_{02}	K_{03}	K_{04}	K_{05}
	5.389	2.426	1.128	0.380	0.180

RESULTS AND DISCUSSION

Analysis of the results from the coalescence filtration was performed based on the 3D plots representing the interdependence of the effluent oil concentration, bed permeability and the fluid velocity.

The dependence of the effluent concentration and fluid velocity was investigated at five different permeabilities for all three oils. Since the experiments were performed in a wide range of fluid velocity, the effluent oil concentration dependence on velocity was exponential for all operating conditions.

For the investigation of oil coalescence using the polypropylene fibre bed, mineral oils with a wide range of physicochemical characteristics were selected. The experiments were carried out also in a broad range of the following parameters: viscosity (10.316–168.904 mPa s), density (879–918.9 kg/m^3), neutralization number (0.13–1.71 mg KOH/l), interfacial tension (18.8–32.4 mN/m), surface tension (26.56–30.16 mN/m), emulsivity (54.17–99.92 vol.%) and relative molecular mass (300–520 kg/kmol).

During the separation of oil A, low values of the effluent concentration were observed at fluid velocities below 50 m/h and in the whole range of bed permeability, Figure 2. The region where the effluent concentration is lower than 15 mg/l is wide. Figures 3 and 4 present the dependence of the effluent concentration on the fluid velocity and bed permeability for oils A and P1. The plots illustrate the region of fluid velocity and bed permeability corresponding to the effluent concentration above 15 mg/l. It should be noticed that this region corresponds to the lowest bed permeability and low fluid velocity, contributing thus to a drastic increase in the effluent concentration. In our previously published papers [8,21,22,25] this region is defined as the critical bed permeability and is the region unsuitable for the coalescer. The absence of the critical permeability region was only observed for oil A. Therefore, it is necessary to give an explanation of why the effluent concentration does not change during the separation of oil A at low bed permeability values.

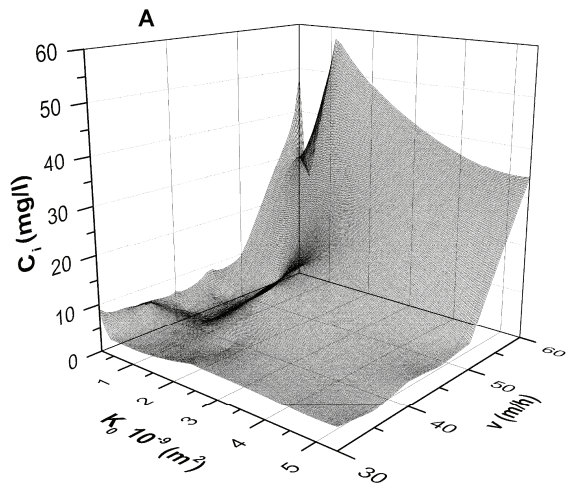

Figure 2. 3D dependence of effluent oil concentration on velocity and fibre bed permeability for oil A.

The region favourable for efficient separation for oil A4 is narrower (K_0 = (0.380–5.389)×10^{-9} m^2 and v = 30–50 m/h) compared to the region of the oil A (K_0 = (0.180–5.389)×10^{-9} m^2 and v = 30–50 m/h). However, this region is significantly wider compared to the region of oil P1 (K_0 = (2.426.180–5.389)×10^{-9} m^2 and v = 19–40

m/h). Remarkably, polypropylene fibres have the lowest separation efficiency for oil P1, since the operating range is extremely narrow. Therefore, it is also necessary to explain why this phenomenon occurs just with oil P1. From the results, it can be concluded that polypropylene fibres show significant sensitivity to changes in the nature of the dispersed oil phase. This is a disadvantage for the coalescer bed material.

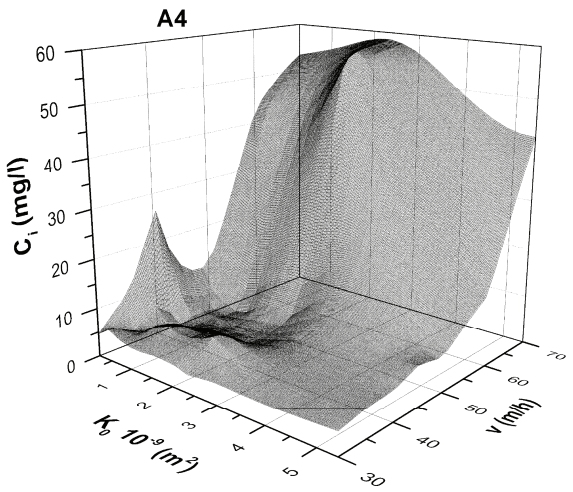

Figure 3. 3D dependence of effluent oil concentration on velocity and fibre bed permeability for oil A4.

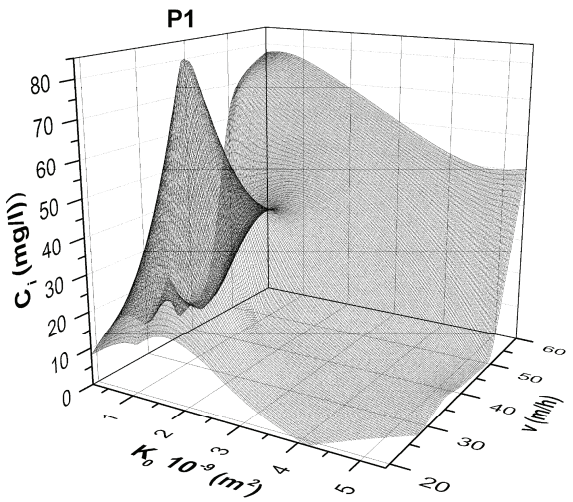

Figure 4. 3D dependence of effluent oil concentration on velocity and fibre bed permeability for oil P1.

During the passing of the emulsion through the fibre bed several coalescence mechanisms simultaneously take place: coalescence between the droplets in the bed pores, droplets coalescence on the surface of the fibres and droplets coalescence into the surface of the capillary-conducted oil phase. In addition coalescence of the droplets takes place after leaving the bed forming the oil continuous phase [26]. It is safe to assume that all the mentioned mechanisms always occur during the separation of any oil, but their rate varies. Which of these coalescence mechanisms will predominate depends significantly on the properties of the oil phase, on the nature of the fibres, as well as on their mutual influence and bed permeability.

Based on presented 3D plots for all three oils it can be observed that at the highest bed permeability the maximum fluid velocity not exceeding the effluent concentration of 15 mg/l is achieved. At the highest bed permeability the pore diameters, as well as the total bed porosity, is the highest, and therefore consequently the interstitial velocity in such pores is the lowest. Also, due to the high porosity, a significant amount of capillary-conducted phase is formed. In such cases the oil droplets coalescence on the surface of the capillary-conducted phase is favoured becoming the dominant mechanism of coalescence. Spielman and Goren concluded that the oil amount in the form of the capillary-conducted phase and the presence of high porosity bed in a steady state ranges from 30 to 40% of the total pore volume [27]. Flowing of the emulsion through the bed and the distribution of the capillary-conducted phase at the cross-section of the device in the horizontal fluid flow orientation is predominantly influenced by the density and viscosity of the dispersed phase, the bed geometry and the intensity of the hydrodynamic forces [9]. When the bed permeability is low, the intensity of the hydrodynamic forces is the highest because the pore diameter is the lowest and the bed is highly compressed. In these conditions there is a minimum amount of the capillary-conducted phase in the bed leading to the increase of effluent concentration for the oils A4 and P1. As already mentioned, the region of the critical bed permeability for the oil A does not exist. This could be explained by the domination of the following coalescence mechanisms: coalescence of the droplets on the fibres surface, and/or the coalescence of the adjacent droplets in the pore space. The release of enlarged droplets from the downstream side of the fibre bed was photographed in all experiments. Furthermore, the efficiency of fibre bed coalescence was evaluated using image analysis. The appearance, the shape, the amount and the colour of the oil formed after leaving the fibre bed differs for all three oils. For the oil P1 after leaving the polypropylene bed, the phase inversion occurs and water droplets wrapped with a thin layer of oil were detected leading to the formation of the double emulsion, Figure 5. From the downstream side of the polypropylene fibre bed, these droplets of different diameters were retarded at the continuous oil/water interface. It is important to note that the formation of the dual emulsion was not observed for the other two oils. The agglomeration of water droplets wrapped with oil P1 was detected. Also, stratification of droplets that leave the bed occurs leading to the formation of the hexagonal structure of

agglomerated drops. The largest agglomerates are retained at the interface, while smaller droplets leaving the bed are located the farthest from the phase interface. It can be assumed that the coalescence of released droplets from the downstream side of the bed is not efficient. Furthermore, it may be the main reason for the limited success of oil P1 coalescence. The cause may be due to the poor separation efficiency of droplets for oil P1 with polypropylene fibres. It is interesting to note that when membrane pump was turned off, quick destruction of agglomerated droplets occurred and separation of oil in the continuous oil phase was observed. This phenomenon can be explained by the cracking of the oil film around the water droplets when the effect of hydrodynamic forces is eliminated.

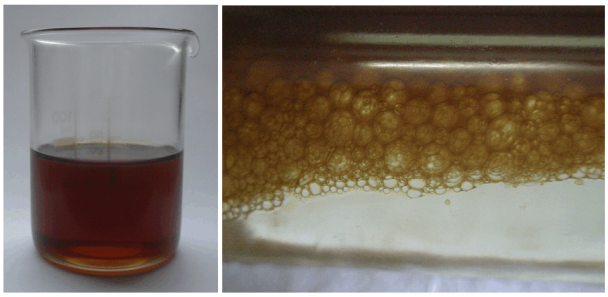

Figure 5. Mineral oil P1 before experiment and after separation of emulsion using polypropylene fibre bed.

In order to form a continuous oil phase from the downstream side of the bed, phase inversion is needed. It is considered that the conditions for the occurrence of the inversion phase are reached when the concentration of dispersed droplets is around 75%. This would imply that the phase inversion during the separation process occurs for all three oils, but is observed only for the oil P1. It is clear that the coalescence of the oil P1 is considerably slower than A and A1. Figure 6 shows the layer of oil at the outlet of the bed. This layer is formed of the capillary-conducted phase dislocated from the fibre bed. From this layer of oil, larger droplets were detached and formed a continuous oil phase in the settling zone. This coalescence mechanism was also observed by other authors [27]. The polypropylene fibres most efficiently separate oil A over a wide range of bed permeabilities. Therefore, it can be assumed that the mentioned mechanism contributes to higher coalescence efficiency.

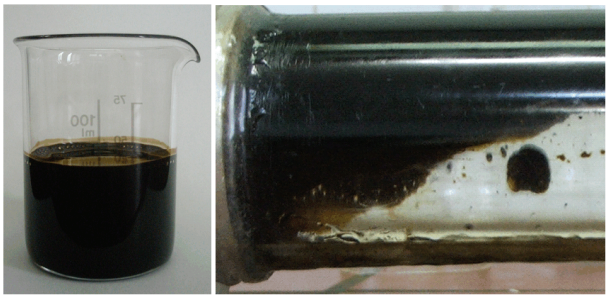

Figure 6. Mineral oil A before experiment and after separation of emulsion using polypropylene fibre bed.

In the case of oil A4 (Figure 7) the change in colour of oil phase, as well as changes in the oil structure leaving the bed were observed. The observations suggest that oil A4 emulsifies when passing through the bed of polypropylene fibres and that a small amount of water is retained in this oil.

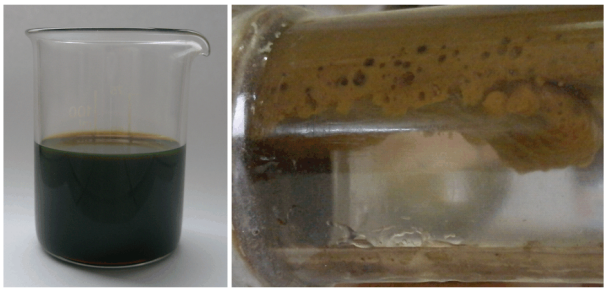

Figure 7. Mineral oil A4 before experiment and after separation of emulsion using polypropylene fibre bed.

Analysing the properties of the investigated oils the following can be pointed out: oil A has an average value of the density, viscosity and relative molecular mass. But oil A has the highest value of emulsivity and neutralization number. According to the mentioned properties oil A has the highest polarity suggesting that such a feature of oil is favoured when working with polypropylene fibres. Since the mineral oils are a complex mixture of many compounds, droplets are chemically heterogeneous in nature. Furthermore, in the composition of the mineral oils, there are also natural surfactants that are measurable through the emulsivity and neutralization number.

CONCLUSION

During the separation of oil A using the polypropylene fibre bed, low values of the effluent concentration were observed at fluid velocities below 50 m/h and at the whole range of studied bed permeabilities. For oils A4 and P1 a region in which the effluent concentration rises steeply at lower values of bed permeability was detected. This region is defined as the critical bed permeability and is the region in which the coalescer is incapable to operate. It was determined that the polypropylene fibres have the lowest separation efficiency for oil P1, since the operating range is extremely narrow. It can be concluded that polypropylene fibres show significant sensitivity to changes in the nature of the dispersed oil phase. This phenolmenon is a disadvantageous for the material used for coalescence.

Acknowledgement

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IZVOD

SEPARACIJA KAPI ULJA RAZLIČITE PRIRODE PRIMENOM SLOJA VLAKANA POLIPROPILENA

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(Naučni rad)

U ovom radu su ispitane mogućnosti separacije model emulzije ulja u vodi primenom koalescencije u sloju vlakana polipropilena. Eksperimenti su realizovani u širokom opsegu fizičko-hemijskih karakteristika mineralnog ulja, permeabilnosti sloja i radne brzine. Cilj rada bio je da se analizira uticaj promene prirode uljne faze i geometrije sloja na efikasnost separacije kroz sloj vlakana polipropilena. Promena geometrije sloja vlakana polipropilena postignuta je povećanjem njegove kompresije tj. variranjem permeabilnosti sloja. Na taj način ispitivana je fleksibilnost primene vlakana polipropilena u koalesceru. Analiza fenomena koalescencije izvršena je na osnovu 3D dijagrama zavisnosti izlazne koncentracije ulja od permeabilnosti sloja i radne brzine. Na osnovu toga su ispitani i definisani radni parametri koalescera sa horizontalnom orijentacijom proticanja koji omogućavaju efikasno izdvajanje dispergovanog ulja i postizanje izlazne koncentracije niže od 15 mg/l. Može se konstatovati da polipropilenska vlakna u najširem opsegu permabilnosti i radne brzine efikasno separišu ulje koje je izrazito polarno, ulje A. Prilikom separacija druga dva ulja, ulje A4 i P1, pojavljuje se pri niskim vrednostima permeabilnosti nepovoljna oblast za rad, to je oblast kritične permeabilnosti. U oblasti kritične permeabilnosti izlazna koncentracija naglo raste. Takođe, oblast kritične permeabilnosti je nepovoljna za rad koalescera. Fenomen kritične permeabilnosti su autori već razmatrali u svojim ranijim radovima. Polipropilenska vlakna efikasno separišu sva tri ispitivana ulja pri najvišoj permeabilnosti sloja. Kako su svi ogledi fotografisani, analiza fotografija omogućila je da se dođe nekih objašnjenja fenomena koji su uočeni. Izgled, oblik, količina i boja ulja formiranog posle sloja različita je pri radu sa ova tri ulja. Zaključeno je da mehanizmi koalescencije značajno zavise od osobina uljne faze, permeabilnosti sloja, prirode vlakana, kao i od njihovog međusobnog uticaja.

Ključne reči: Separacija zauljenih voda • Koalescencija u poroznom sloju • Vlaknasti materijal • Polipropilen