

# Experimental research on sorption of petroleum products from water by natural clinoptilolite and vermiculite

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An experimental study of the sorption of petroleum products (PP) from aqueous solutions by natural vermiculite and zeolite (clinoptilolite) was carried out. Two different sizes of zeolite particles were used: 0.63–1.0, 1.0–3.0 mm and 1.0–3.0 mm fractions of vermiculite. Zeolite and vermiculite material was washed and dried at 105 °C in an oven before using it in the filter bed. 40 l of snowmelt water, 1 l of gasoline and 1 l of diesel passed the filter charged with zeolite and vermiculite. Comparison of the results showed that the highest removal of PP from water solution was obtained by using the finest grain-sized zeolite. The removal efficiency was 89.8% and 76.4% respectively for 0.63–1.0 mm and 1.0–3.0 mm particle size zeolite. The highest PP removal efficiency (92.4%) was achieved by using 1.0–3.0 mm particle-sized vermiculite.

**Key words:** zeolite, vermiculite, water treatment, sorption

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## INTRODUCTION

All sorbents used for cleaning soil, water or air have to fit environmental protection requirements, i.e. to be easily collected, harmless to the environment, biodegradable and easily utilized.

Though a high efficiency of water cleaning is reached by using expensive and high sorption capacity material, it is purposeful to focus on searching of new and cheap materials even with a lower sorption capacity. Nowadays in the water treatment area new technologies based on the principle of sorption have been created.

The wide and intensive use of petroleum products (PP) as the main source of energy in Lithuania is a source of soil and water pollution. The problem was observed and began to be solved about 15–20 years ago in Lithuania (Marcionis, Paukštys, 1999). In 2002, the Lithuanian

Republic Environmental Protection Law was passed and provisions of the Environmental Protection action program for Central and East Europe of EU directives in Lithuania were assigned as “Treatment requirements of soil and groundwater polluted with oil products and emission limits” (LAND 9-2002). It is stressed in the requirements that the main problem and task for today is to minimize PP dispersion in the geological environment. Groundwater contamination by PP includes the quantity of light and heavy hydrocarbons in groundwater exceeding the background level. The background level of light hydrocarbons in groundwater is 0.1 mg/l, according to LAND 9-2002 normative requirements of Lithuania. With a view to stop pollution of geological environment by PP and to clean polluted water, a detail study of PP sorption is needed.

In recent years, the investigation of the usage of natural zeolite and vermiculite for removal of heavy

metals and PP from runoff has been started in Lithuania (Anisimova et al., 2004, Brannvall, Kazlauskienė, 2005; Baltrėnas, Brannvall, 2006; Mažeikienė et al., 2005). The capacity of natural powdered zeolite to remove organic matter from drinking water was investigated comparatively recently (Sakalauskas, Valentukevičienė, 2003; Valentukevičienė, Jankauskas, 2004). The Swedish mineralogist A. F. Cronstedt originally coined the term zeolite in the 18th century (Cronstedt, 1756). He observed, upon rapidly heating natural zeolite, that the stones began to dance about as the water evaporated. Using the Greek words which mean “stone that boils” he called this material zeolite. The commonly used description of zeolite is a crystalline aluminosilicate with a cage structure. Technically, zeolite is like a crystalline hydrated aluminosilicate whose framework structure encloses cavities (or pores) occupied by cations and water molecules, both of which have a considerable freedom of movement, permitting ion exchange and reversible dehydration. Zeolite is an ecological, non-toxic material suitable for many applications in industry, agriculture, environmental protection, and for removal of the consequences of ecological catastrophes (Lebedynets, 2004, Sprinsky, 2005).

For the reasons mentioned above, it is purposeful to find out how much PP can be removed from polluted water using natural materials. The purpose of the experiments presented in this paper was to find out the efficiency of natural zeolite (clinoptilolite) and vermiculite sorption of PP from solutions and to compare their sorption capacities. The results of this study will be used for the calculation of sorbent amounts needed to remove certain concentration of PP from runoff.

It is known that sorption capacity increases by crushing the sorbent, because the surface area increases. Therefore two fractions of natural zeolite and one fraction of vermiculite were used in this study.

## MATERIALS AND METHODS

The sorbents investigated in this study were natural zeolite and vermiculite (Figs. 1, 2). Different researchers studied the removal of PP from solutions by vermiculite (Mesyats et al., 1984; Da Silva et al., 2003; Mysore, 2005). Vermiculite can be regarded as a micropore absorbent if we consider the interspaced crystals as pores.

A pilot test bench was constructed for the study. The amount of 40 l of snowmelt water, 1 l of gasoline and 1 l of diesel were poured into a 50 l plastic container. This mixture was stirred mechanically and pumped into the first cylinder ( $\varnothing$  105 mm) in which PP were settled. Samples were taken from the first cylinder through a sample mixer tap to define the PP concentration in the first cylinder. The flexible hose connected the first cylinder to the second one. In the second cylinder ( $\varnothing$  105 mm) water contaminated with PP was filtered through a zeolite and vermiculite layer 20 cm

thick. The filtrate samples were taken from the second cylinder for the determination of PP concentration.

Two different sizes of zeolite particles were used in this study: 0.63–1.0, 1.0–3.0 mm and one vermiculite fraction of 1–3 mm. The test materials were washed and dried at 105 °C in an oven before using it in the filter bed.

The physical properties of snowmelt water used in this study were: suspended particles 700–800 mg/l, pH 7.0–7.5, to 18.5–20.0 °C. Filter charges of the same volume were weighed, and they were as follows: 1756.6 g (0.63–1.0 mm fraction of zeolite), 1814.5 g (1.0–3.0 mm fraction of zeolite) and 191.3 g (1.0–3.0 mm fraction of vermiculite). The concentration of PP sorbed by 1 g of material was calculated. The filtration process was started at a filtration rate of 5 m/h and experiments were stopped when marginal pressure losses

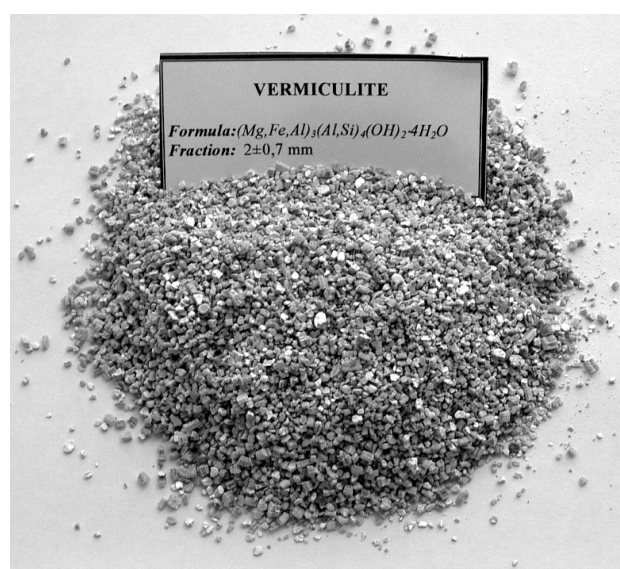


Fig. 1. Expanded vermiculite  
1 pav. Išpūstasis vermikulitas



Fig. 2. Natural zeolite (clinoptilolite)  
2 pav. Gamtinis ceolitas (klinoptilolitas)

were reached and the quality of filtrate was not acceptable. PP concentrations in the experimental water before filter and filtrate samples was measured every hour, five times each. A TOG/TPH analyzer was used for the detection of PP concentrations in water and filtrate samples. The HATR-T Infracal TOG/TPH Analyzer is designed as an alternative for EPA Methods 413.2 and 418.1 that use freon in the extraction procedure as well as for other applications where the concentration of a sample dissolved in an infrared transparent solvent is to be measured. Individual samples were prepared using the Hexane extraction procedure. To determine the con-

tent by using the Hexane extraction procedure and the HATR-T Infracal TOG/TPH Analyzer Model, a sample is placed in the trough of the IR Platform. The Infracal IR Platform can provide quantitative information on oil and grease residue left on the reflecting surface after the hexane extractant has evaporated.

## RESULTS AND DISCUSSION

The results of the experimental research using different particle sizes of zeolite (clinoptilolite) and vermiculite for PP removal from water are shown in Tables 1–3.

Table 1. Sorption of petroleum products by natural zeolite (clinoptilolite), fraction 0.63–1.0 mm  
1 lentelė. Naftos produktų sorbcija 0,63–1,0 mm frakcijos gamtiniu ceolitu (klinoptilolitu)

Start / Stand by time	Measurement time, h	Mixing period, h	$C_{su}$ , mg/l	$C_p$ , mg/l	Concentration reduction, mg/l	Removal effectiveness, %
30 03 2005						
Start 9.00	14	5	4.20	0.43	3.77	89.8
	16	8	6.31	0.72	5.59	88.6
Stand by 18.00						
31 03 2005						
Start 9.00	11	11	6.43	0.74	5.69	88.5
	13	12	6.47	0.82	5.65	87.4
	15	14	6.48	0.95	5.53	85.3
	16	15	6.50	1.10	5.40	83.0
All day working	17	16	6.52	1.15	5.37	82.4
01 04 2005						
	11	34	7.01	1.80	5.21	74.3
	13	36	6.90	1.82	5.08	73.6
	14	37	7.00	1.86	5.14	73.4
	15	38	7.03	1.90	5.13	73.0
Stand by 17.00	16	39	6.73	1.83	4.90	72.8
04 04 2005						
Start 10.00	12	42	7.33	2.02	5.31	72.5
	14	44	7.41	2.07	5.34	72.0
	16	46	7.70	2.17	5.53	71.8
Stand by 17.00						
05 04 2005						
Start 10.00	13	50	8.43	2.40	6.03	71.5
	14	51	9.14	2.57	6.57	71.9
	15	52	9.94	2.86	7.08	71.2
	16	53	10.29	2.98	7.31	71.0
Stand by 17.00						
06 04 2005						
Start 10.00	12	56	11.68	3.4	8.28	70.9
	13	57	12.71	3.71	9.00	70.8
	15	59	13.68	4.04	9.64	70.5
Stand by 17.00						

Initial set concentration in mixing unit was  $C_1 = 15.0$  mg/l;  $C_{su}$  – solution concentration in sedimentation unit, mg/l;  $C_f$  – solution concentration in filtered water, mg/l.

Pastaba: pirminė koncentracija ( $C_1$ ) maišymo talpykloje buvo 15,0 mg/l;  $C_{su}$  – tirpalo koncentracija nusodintuve mg/l;  $C_f$  – koncentracija filtrate mg/l.

Table 2. Sorption of petroleum products by natural zeolite (clinoptilolite), fraction 1.0–3.0 mm  
2 lentelė. Naftos produktų sorbcija 1,0–3,0 mm frakcijos gamtiniu ceolitu (klinoptilolitu)

Start/Stand by time	Measurement time, h	Mixing period, h	$C_{su}$ , mg/l	$C_f$ , mg/l	Concentration reduction, mg/l	Removal effectiveness, %
09 05 2005 Start 11.30 Stand by 17.00	15	3.30	4.00	0.94	3.06	76.4
10 05 2005 Start 11.30 Stand by 17.00	13	7	4.23	1.06	3.17	75.0
12 05 2005 Start 10.30 Stand by 17.00	14	14.30	5.00	1.27	3.73	74.6
16 05 2005 Start 10.30 Stand by 17.00	13 14	20 21	6.33 7.00	1.69 1.89	4.64 5.11	73.3 73.0
17 05 2005 Start 8.30 Stand by 17.00	10 11 12 13 15	25.30 26.30 27.30 28.30 30.30	12.90 12.80 13.70 13.90 14.00	3.55 3.55 9.48 4.29 5.07	9.35 9.25 9.48 9.61 8.93	72.5 72.3 69.2 69.2 63.8

Initial set concentration in mixing unit was  $C_1 = 15.0$  mg/l;  $C_{su}$  – solution concentration in sedimentation unit, mg/l;  $C_f$  – solution concentration in filtered water, mg/l.

Pastaba: pirminė koncentracija ( $C_1$ ) maišymo talpykloje buvo – 15,0 mg/l;  $C_{su}$  – tirpalo koncentracija nusodintuve mg/l;  $C_f$  – koncentracija filtrate mg/l.

Table 3. Sorption of petroleum products by vermiculite, fraction 1.0–3.0 mm  
3 lentelė. Naftos produktų sorbcija of 1,0–3,0 mm frakcijos vermikulitu

Start/Stand by time	Measurement time, h	Mixing period, h	$C_{su}$ , mg/l	$C_f$ , mg/l	Concentration reduction, mg/l	Removal effectiveness, %
13 04 2005 Start 11.00 Stand by 15.30	15	4 4.30	5.03	0.38	4.65	92.4
14 04 2005 Start 8.30 Stand by 13.30	11 13	4.30 7 9 9.30	5.42 5.60	0.42 0.53	5.00 5.07	92.3 90.5
18 04 2005 Start 10.00 Stand by 14.30	11 12 14	9.30 10.30 11.30 13.30 14	6.25 6.60 7.17	1.00 1.07 1.33	5.25 5.53 5.84	84.0 83.8 81.5
19 04 2005 Start 13.00 Stand by 16.00	15 16	14 16 17	7.98 8.73	1.78 2.03	6.20 6.70	77.7 76.7

Initial set concentration in mixing unit was  $C_1 = 15.0$  mg/l;  $C_{su}$  – solution concentration in sedimentation unit, mg/l;  $C_f$  – solution concentration in filtered water, mg/l.

Pastaba: pirminė koncentracija ( $C_1$ ) maišymo talpykloje buvo – 15,0 mg/l;  $C_{su}$  – tirpalo koncentracija nusodintuve mg/l;  $C_f$  – koncentracija filtrate mg/l.

The different periods of mixing and filtration were varied by using different filter media. The water filtration cycle using vermiculite was the shortest. Because of the high vermiculite porosity, the filter medium was obstructed and the filtration rate became too low; after that the filtration was stopped. The longest (59 h) filtration time was used for 0.63–1.0 mm particle size of the zeolite filter medium. The filter was obstructed more slowly compared with the vermiculite filter media, but the effectiveness of PP removal from the water during the last filtration stage was only 76.9%. Using the 1.0–3.0 mm particle size of zeolite, filtration lasted 30.5 h. Even the filtration rate was constant till the end of the cycle when the water treatment effectiveness was up to 63.8%.

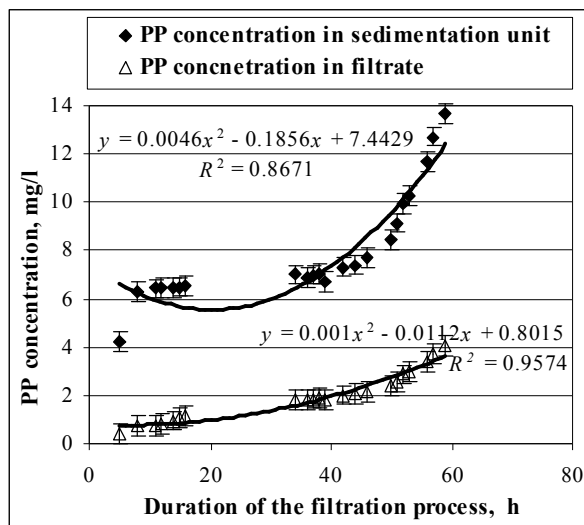
During the experiment, the constant initial concentration of solution was established by using a mixture of melted snow with PP from gasoline and diesel. PP concentration in the sedimentation unit (first reservoir) was lower than in the primary reservoir, but it became higher during the mixing and filtration processes because of different water solubility of hydrocarbon ingredients of gasoline and diesel. A floating layer of PP appeared on the surface of water in the sedimentation unit, and the layer of PP was not removed during filtration. After a long period, part of hydrocarbons from petroleum products passed into the water mixture and PP concentration increased. Conventional units of PP removal are operated with the floating PP layer on the top of the sedimentation unit, and when the accumulated quantity of PP exceeded the allowable level it had to be removed because of the possibilities for increasing PP concentration before the filter unit. The experimental conditions were adjusted to real conditions. PP concentrations following the initial concentration in the water and the sedimentation unit are presented in Figs. 3–5.

The result was evaluated using a polynomial model when the statistical determination coefficient was  $R^2 = 0.87$  of results from the sedimentation unit and  $R^2 = 0.96$  for the filter. PP concentration increased during the longer filtration period. According to the results presented in Fig. 1, the filtration with 0.63–1.0 mm particle size of zeolite lasted less than 14–15 h when the PP concentration in treated water did not exceed the levels permitted by LAND 9-2002. When the initial PP concentration was 15 mg/l, filtered water could contain >1 mg/l of petroleum products.

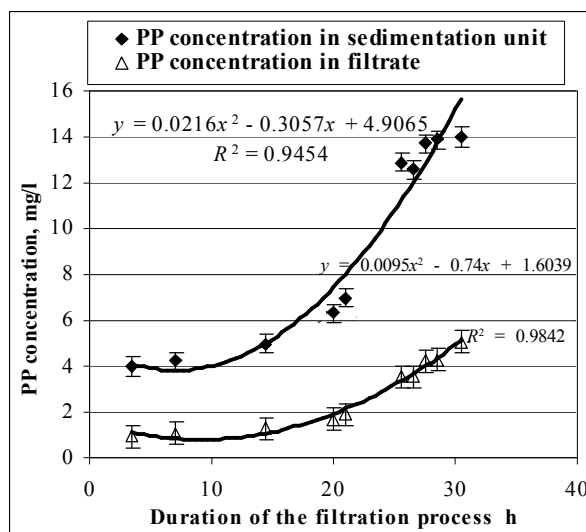
The particle size 1.0–3.0 mm of zeolite and vermiculite filter medias was tested for comparison and results are presented in Figs. 4 and 5.

The following results are presented in Figs. 2 and 3: PP concentration increased during a longer filtration period in sedimentation unit and in filtered water. The results were statistically evaluated with  $R^2 = 0.94–0.99$ .

The best results of PP removal from water were obtained when vermiculite and zeolite with particle size 1.0–3.0 mm were used (Fig. 6).



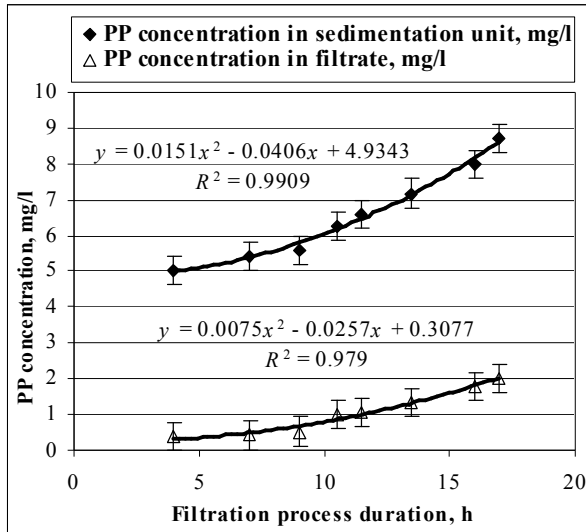
**Fig. 3.** Sorption of petroleum products by natural zeolite (clinoptilolite), fraction 0.63–1.0 mm, at the initial concentration of PP 15 mg/l and the initial filtration rate  $V_0 = 5$  m/h  
**3 pav.** Naftos produktų sorbcija 0,63–1,0 mm frakcijos gamtiniu ceolitu (klinoptilolitu), kai pirminė naftos produktų koncentracija – 15 mg/l, pirminis filtravimo greitis  $V_0 = 5$  m/h



**Fig. 4.** Sorption of petroleum products by natural zeolite (clinoptilolite), fraction 1.0–3.0 mm, at the initial concentration of PP 15 mg/l and the initial filtration rate  $V_0 = 5$  m/h  
**4 pav.** Naftos produktų sorbcija 1,0–3,0 mm frakcijos gamtiniu ceolitu (klinoptilolitu), kai pirminė naftos produktų koncentracija – 15 mg/l, pirminis filtravimo greitis  $V_0 = 5$  m/h

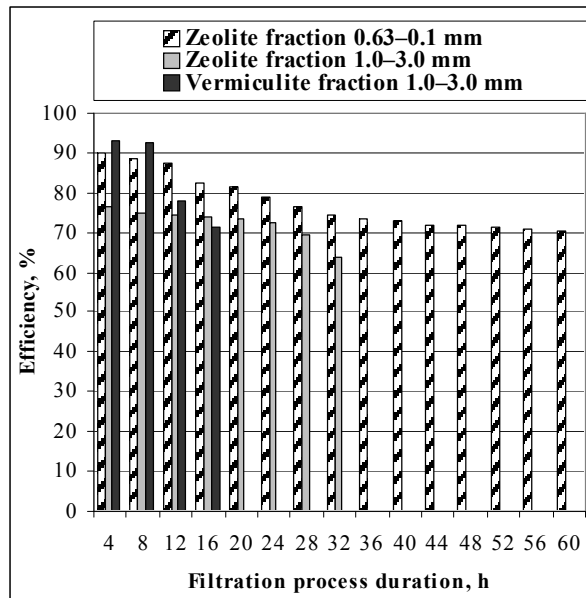
The results presented in Fig. 4 show that during a long-term treatment the efficiency decreased to 63.8% using 1.0–3.0 mm particle-sized zeolite. The best sorption efficiency was obtained during filtration for no longer than 14–15 h, when the efficiency decreased to 74.6%. When zeolite filter medium with a particle size of 1.0–3.0 mm was used, the highest PP removal efficiency was 76.4%.

Using a smaller zeolite particle size, the treatment efficiency after 14 h was 85.3% and after 59 h de-



**Fig. 5.** Sorption of petroleum products by vermiculite, fraction 1.0–3.0 mm, at the initial concentration of PP 15 mg/l and the initial filtration rate  $V_0 = 5$  m/h

**5 pav.** Naftos produktų sorbcija 1,0–3,0 mm frakcijos vermikulitu, kai pirminė naftos produktų koncentracija – 15 mg/l, pirminis filtravimo greitis  $V_0 = 5$  m/h

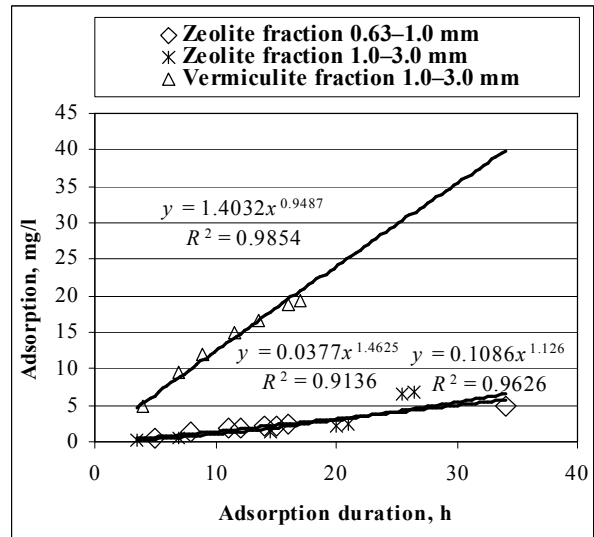


**Fig. 6.** Comparison of the efficiency of different filter media  
**6 pav.** Sorbentų efektyvumo palyginimas

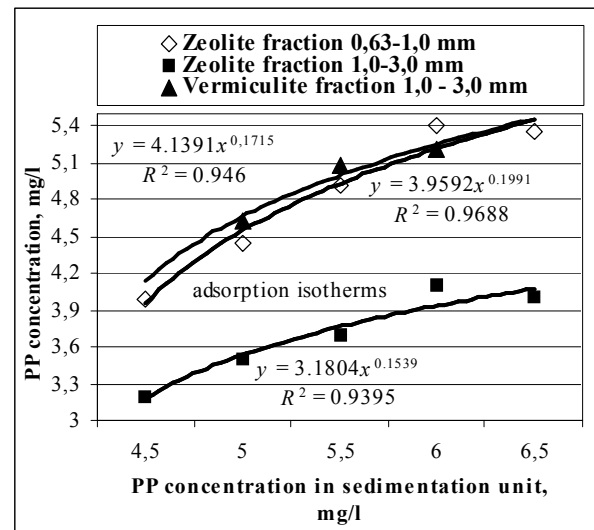
creased to 70.5%. The highest treatment efficiency was equal to 89.8%. Using vermiculite, PP removal after 14 h was 81.5%, and the highest treatment efficiency was 92.4%.

A comparison of results for different sorbents used for PP removal is presented in Figs. 7 and 8.

The biggest adsorbed quantity of PP was obtained by using vermiculite as a filter medium, and the filtration cycle was the shortest compared with both particle sizes of the zeolite filter medium. A comparison of adsorbed PP quantities shows that the biggest amount of adsorbed PP using vermiculite was 13 times bigger



**Fig. 7.** Dependence of PP adsorption on process duration  
**7 pav.** Naftos produktų sorbcijos priklausomybė nuo proceso trukmės



**Fig. 8.** Dependency of PP concentration decrease on initial concentration in sedimentation unit

**8 pav.** Naftos produktų koncentracijos sumažėjimo filtrate priklausomybė nuo pirminės koncentracijos nusodintuve

than with the 0.63–1.0 mm particle-sized zeolite and 37 times bigger than with the 1.0–3.0 mm particle-sized zeolite filter medium. The same comparison of the decreased concentrations of PP is provided for different kinds of filter media with the primary PP concentration after the sedimentation unit.

Experimental data provided in Fig. 6 were analyzed independently of different weights of the filter media (the same volume of filter media was used). The results show that vermiculite has the biggest adsorption capacity. The 0.63–1.0 mm particle size of zeolite has a higher adsorption capacity than the particle size 1.0–3.0 mm of zeolite filter media.

The experimental results have shown that natural zeolite (clinoptilolite) and vermiculite can be used for PP adsorption from natural runoff water. The preliminary

experimental results of this research should be investigated more extensively in the future, with evaluation of suspended matter, different chemical ingredients, pH and other factors that possibly influence the adsorption process.

## CONCLUSIONS

1. During the laboratory experiment with a pilot test bench, the possibility to use two different particle sizes (0.63–1.0 mm and 1.0–3.0 mm) of natural zeolite (clinoptilolite) and vermiculite with a particle size of 1.0–3.0 mm for the PP removal from runoff water was shown.

2. The highest PP removal efficiency (92.4%) was achieved using 1.0–3.0 mm particle-sized vermiculite. When the initial PP concentration in water solution was 15 mg/l, the PP residual in filtered water was 0.38 mg/l.

3. Of the two vermiculate fractions, the highest removal of PP from water solution was shown by the finest zeolite fraction. The removal efficiency was 89.8% and 76.4% respectively for 0.63–1.0 mm and 1.0–3.0 mm size fractions of zeolite. When the initial PP concentration was 15 mg/l, the residual PP was 0.43 mg/l and 0.94 mg/l in filtered water depending on zeolite particle size.

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## GAMTINIŲ KLINOPTILOLITŲ IR VERMIKULITŲ GALIMYBIŲ SORBUOTI NAFTOS PRODUKTUS IŠ VANDENS EKSPERIMENTINIAI TYRIMAI

### Santrauka

Eksperimentiniai naftos produktų sorbcijos iš vandeninių tirpalų tyrimai buvo atlikti naudojant gamtinio ceolito (klinoptilolito) (0,63–1,0, ir 1,0–3,0 mm) bei vermikulito (1,0–3,0) mm frakcijas. Prieš naudojimą gamtinis ceolitas buvo išplautas ir išdžiovintas krosnyje apie 105°C temperatūroje. Pro filtro įkrovą (vienu atveju – smulčiausia gamtinio ceolito, kitu – stambesnė, o trečiu – vermikulito frakcija) praleistas eksperimentinis tirpalas iš 40 l sniego tirpimo vandens su 1 l dyzelino ir 1 l benzino. Lyginant dviejų gamtinio ceolito frakcijų eksperimento rezultatus nustatyta, kad smulkesnė gamtinio ceolito frakcija efektyviau sorbuoja naftos produktus iš vandeninių tirpalų: naftos produktų šalinimo efektyvumas 0,63–1,0 mm ir 1,0–3,0 mm gamtinio ceolito frakcijose siekė atitinkamai 89,8% ir 76,4%. Geriausiai naftos produktus sorbavo vermikulitas, kurio naftos produktų šalinimo iš vandeninių tirpalų efektyvumas siekė 92,4%.

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**ЭКСПЕРИМЕНТАЛЬНОЕ ИССЛЕДОВАНИЕ  
СОРБЦИИ НЕФТЕПРОДУКТОВ ИЗ ВОДЫ  
НАТУРАЛЬНЫМИ КЛИНОПТИЛОЛИТОМ И  
ВЕРМИКУЛИТОМ**

**Резюме**

Экспериментально исследованы возможности использовать цеолит (клиноптилолит) и натуральный вермикулит для сорбции нефтепродуктов из водяных растворов.

Использовались: цеолит двух различных фракций (0,63–1,0 мм и 1,0–3,0 мм) и вермикулит (1,0–3,0 мм). Природный цеолит был промыт и высушен при температуре 105 °С. Талая вода (40 дм<sup>3</sup>), бензин (1 дм<sup>3</sup>), дизельное топливо (1 дм<sup>3</sup>) пропускались через фильтрующий слой из цеолита различных фракций и вермикулита. Экспериментальные данные показали, что очистка воды с использованием мельчайшей фракции цеолита (клиноптилолита) (0,63–1,0 мм) более эффективна: соответственно 89,8 и 76,4%. Наиболее эффективная очистка воды от нефтепродуктов (92,4%) была достигнута при использовании фракции вермикулита 1,0–3,0 мм.