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Effects of petroleum pollution on clay soil microstructure

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In the paper, microstructural changes of clay soil – glacial till – caused by *in-situ* pollution with diesel oil are presented. STIMAN software was used for a quantitative analysis of scanning electron microscope-based photographs. As a result of pollution, the microstructure underwent substantial qualitative changes: the packing of particles and clayey microaggregates decreased and part of the microaggregates disintegrated, edges and corners of some clay particles were warped the amount of intermicroaggregate pores and edge-to-face (EF) contacts among clay microaggregates increased. Diesel oil pollution entailed significant quantitative changes in the till pore space. The amount of mesopores, the maximum and average pore area, the maximum, minimum and average pore perimeter and the maximum and average pore diameter grew markedly while the value of the total pore perimeter decreased. As indicated by the drop of the microstructural anisotropy index and the amount of fissure pores as well as the growing average form index, the polluted clay became more isotropic. These changes might be due to the reduction of interparticle forces upon pollution with a fluid characterized by a dielectric constant lower than water.

Key words: glacial till, petroleum-derived pollution, microstructure, pore space

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INTRODUCTION

For many years, changes of soil properties resulting from pollution have been a subject of interest of both Polish and foreign scientists. The saturation of soil by fluids characterized by physico-chemical properties different from water has been found to have a deteriorating effect on its mechanical and filtration parameters, plasticity, swelling and others (e. g., Bowders, Daniel, 1987; Stephenson, 1989; Barański, 2000; Herzig, 2001; Korzeniowska-Rejmer, 2001; Garbulewski, Fronczyk, 2004; Izdebska-Mucha, 2005; Korzeniowska-Rejmer, Izdebska-Mucha, 2006; Khamehchiyan et al., 2007).

With respect to changes of engineering-geological parameters, filtration properties in particular, in cohesive soils polluted with organic fluids the microstructural analysis seems to be of particular interest and significance. Results published so far have been limited to data obtained from samples after filtration tests or suspension studies (e. g., Fernandez, Quigley, 1985; Berger et al., 2002; Kaya, Fang, 2005).

Thanks to the use of special software for the scanning electron microscope (SEM)-based microstructural analysis, quantitative characteristics of pore space parameters are given in this paper along with qualitative descriptions. The data reflect microstructural changes noted in cohesive soils contaminated *in situ* with a petroleum-derived substance. These studies are a continuation of quantitative analyses of porosity changes in glacial till artificially saturated with petroleum and diesel oil (Izdebska-Mucha, 2003, 2008) as well as of microstructural studies conducted over many years at the Institute of Hydrogeology and Engineering Geology, Faculty of Geology, University of Warsaw (e. g., Grabowska-Olszewska, 1976, 1983; Grabowska-Olszewska et al., 1984; Kaczyński, Trzciński, 1997; Trzciński, 2003; Izdebska-Mucha, Trzciński, 2007).

METHODOLOGY

Soil samples were collected from the area of a fuel terminal located in north-eastern Poland. Pollution with diesel oil occurred in August 2005 in a field with underground fuel tanks (Fig. 1). In the area where a down-flow and stagnation of diesel oil took place, petroleum-derived substances migrated upwards in the soil ground. Determination of hydrocarbon content in soils, carried out in September and October 2005, revealed the pollution from the ground surface to the groundwater level to exceed 1.5 to 10 times the permissible concentration (Table 1).

Soil samples for laboratory tests were collected from two 2 m deep exploration pits, one being located in an area not

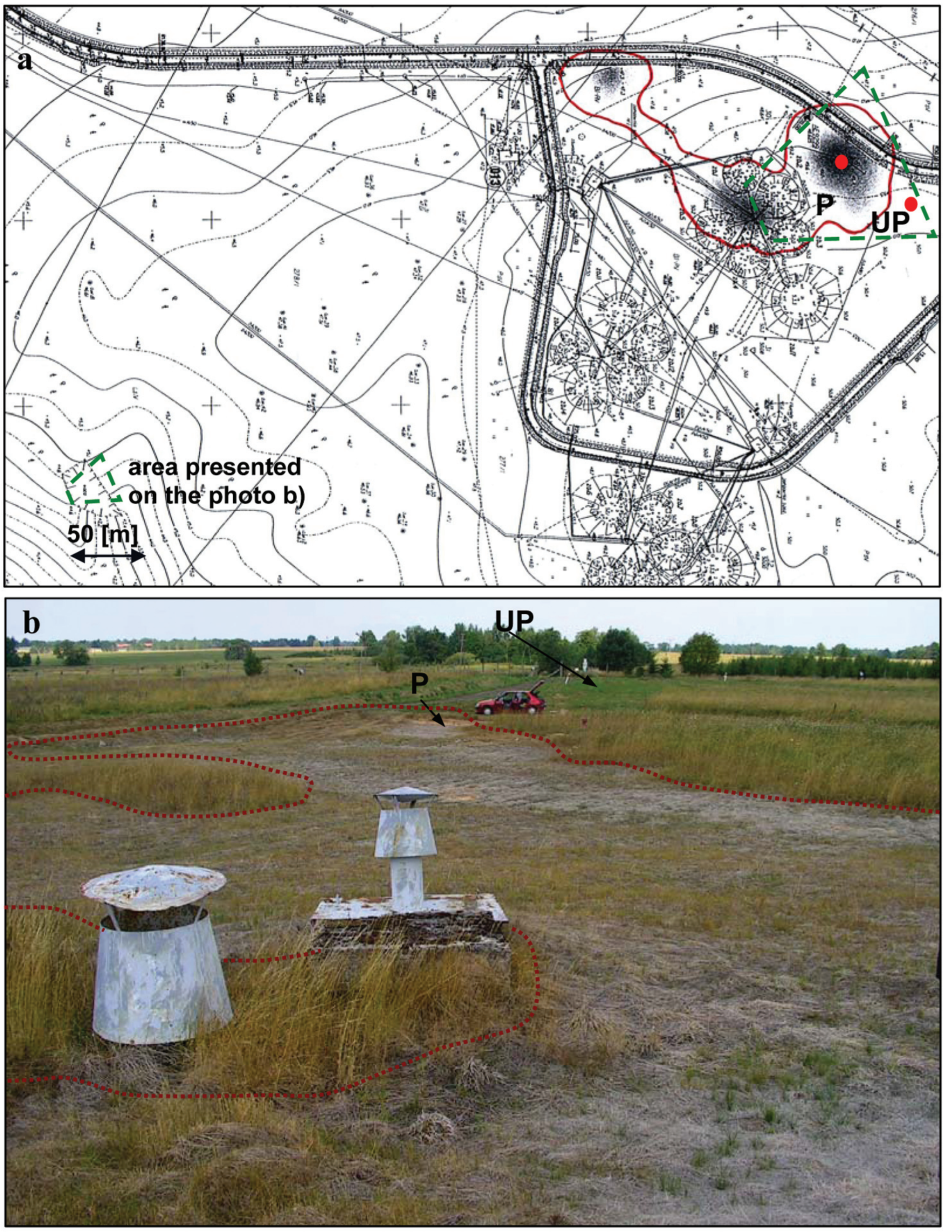

Fig. 1. Location of sampling sites: *a* – map of underground fuel tank field in the fuel terminal area; *b* – photo of a part of the area polluted with diesel oil. UN – non-polluted till, P – till polluted with diesel oil. Red line shows the limits of diesel oil spill

Table 1. **Hydrocarbon content in polluted glacial tills**

Sampling depth, m	Soil type	Hydrocarbon C_6–C_{12} content, mg/kg of dry weight		Hydrocarbon >C_{12} content, mg/kg of dry weight		Total, mg/kg of dry weight
		measured	permissible[1]	measured	permissible[1]	
0.3	Clayey till (horizon A, see Fig. 2)	392.79	500	32147.85	3000	32540.64
2.0	Sandy till (horizon B, see Fig. 2)	1456.6	500	3938.2	3000	5394.8

[1] *Journal of Laws*, No. 165, pos. 1359.

affected by pollution and the other in a polluted area. Two glacial till horizons were found to occur in the vertical section of these artificial exposures (Fig. 2). According to the Geological Map of Poland at a scale 1 : 50 000 (Rabek, Świerszcz, 2003), these tills were formed during the upper stadial of the Vistula Glaciation.

Microstructural studies presented in this paper were completed on sandy till samples from the lower horizon B (Fig. 2). The hydrometer sedimentation test and sieving analysis revealed the till to contain 1% of gravel, 53% of sand, 25% of silt and 21% of clay fraction on the average. The mineral composition resulting from the XRD diffraction analysis is as follows: illite, kaolinite, smectite, quartz and accessory minerals (Fig. 3).

Till samples of undisturbed structure in the form of monoliths were taken, of which approximately 1 cm^3 cubes were cut. These were subsequently freeze-dried and subjected to SEM-based quantitative microstructural analysis (Trzciński, 2004). The microstructure of non-polluted (NP) till and that polluted with diesel oil (P) was analysed on images with magnifications ranging from 100 to 6 500 times. The SEM used in these studies was the Jeol JSM 6380LA model, and the quantitative procedures were conducted in accordance with the instruction for the respective STIMAN software (Sokolov et al., 2002). The studies were accomplished at the Laboratory of Scanning Electron Microscopy and Microanalysis, Institute of Hydrogeology and Engineering Geology, Faculty of Geology, University of Warsaw.

Fig. 2. Exploration pit where polluted soil was sampled. *A* – upper till horizon, thickness about 1.3 m, brown-red clayey till with few pebbles, *B* – lower till horizon, analysed brown sandy till

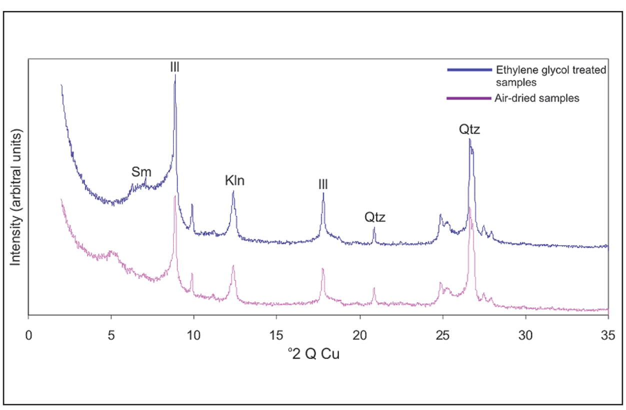

Fig. 3. XRD analysis of glacial till from horizon B. Samples sedimentated on glass slides. Sm – smectite, Ill – illite, Kln – kaolinite, Qtz – quartz

MICROSTRUCTURAL CHARACTERISTICS OF NON-POLLUTED TILL AND TILL POLLUTED WITH DIESEL OIL

Qualitative description of the microstructure. The glacial till examined has a matrix microstructure (according to classification by Sergeyev et al., 1980; Grabowska-Olszewska et al., 1984). Taking into account the relative packing of structural elements, the non-polluted till was classified into subtype B – medium packed (according to classification by Trzciński, 2003). The matrix microstructure consists of a clayey mass, the so-called matrix (thin arrow in Fig. 4a) with individual sandy (lower arrow in Fig. 4b) and silty (upper arrow in Fig. 4b) grains embedded in it. The clayey matrix is aggregated and composed of microaggregates (bold right arrow in Fig. 4c) which together with grains form clayey-silty and clayey-sandy aggregates. The grain surface is covered with clay film. The contacts among the microaggregates are of the face-to-face FF (thin upper arrow in Fig. 4c), edge-to-face EF (thin lower arrow in Fig. 4c) or edge-to-edge EE (lower thin arrow in Fig. 4d) type. There is a lack of visible orientation of structural elements. The pore space is composed mainly of intermicroaggregate pores (left bold arrow in Fig. 4c), interparticle pores (bold arrow in Fig. 4d) and subordinate interaggregate pores (bold arrows in Fig. 4a) according to porosity classification by Grabowska-Olszewska et al. (1984).

Significant microstructural changes were noted between till P and till NP, a comparison of their features being presented in Table 2.

Quantitative description of the pore space. Substantial differences were observed in the morphometric and geometric parameters between NP and P tills (Table 3). The porosity value remains unchanged, but the coefficient of variation is higher for NP till and the minimum value is higher for P till. The number of pores is smaller in P till and the maximum value is much higher in NP till.

An analysis of the morphometric parameters (area, perimeter, diameter) provided the following information. There was no significant change of the total pore area in P till, whereas the maximum and average pore area parameters clearly shifted towards the higher values. The minimum pore area value remained unchanged. The total pore perimeter was lower, but the maximum, minimum and average values of this parameter were higher for P till. Elevated maximum and average pore diameter parameters were found for P till, but the minimum pore diameter value was unchanged. The distribution of pore size substantially changed. The amount of micropores decreased, while mesopores proved more abundant in P till.

In addition, the geometric (shape, anisotropy, degree of orientation) parameters also changed. The average form index value grew in P till. Observations of the pore shape revealed a smaller amount of fissure pores and an elevated share of anisometric and isometric pores. The microstructural anisotropy coefficient-like value decreased markedly for P till.

INTERPRETATION AND DISCUSSION OF THE RESULTS

The results (Table 3) revealed that, compared to NP till, the average porosity value for P till remained unchanged. The low value

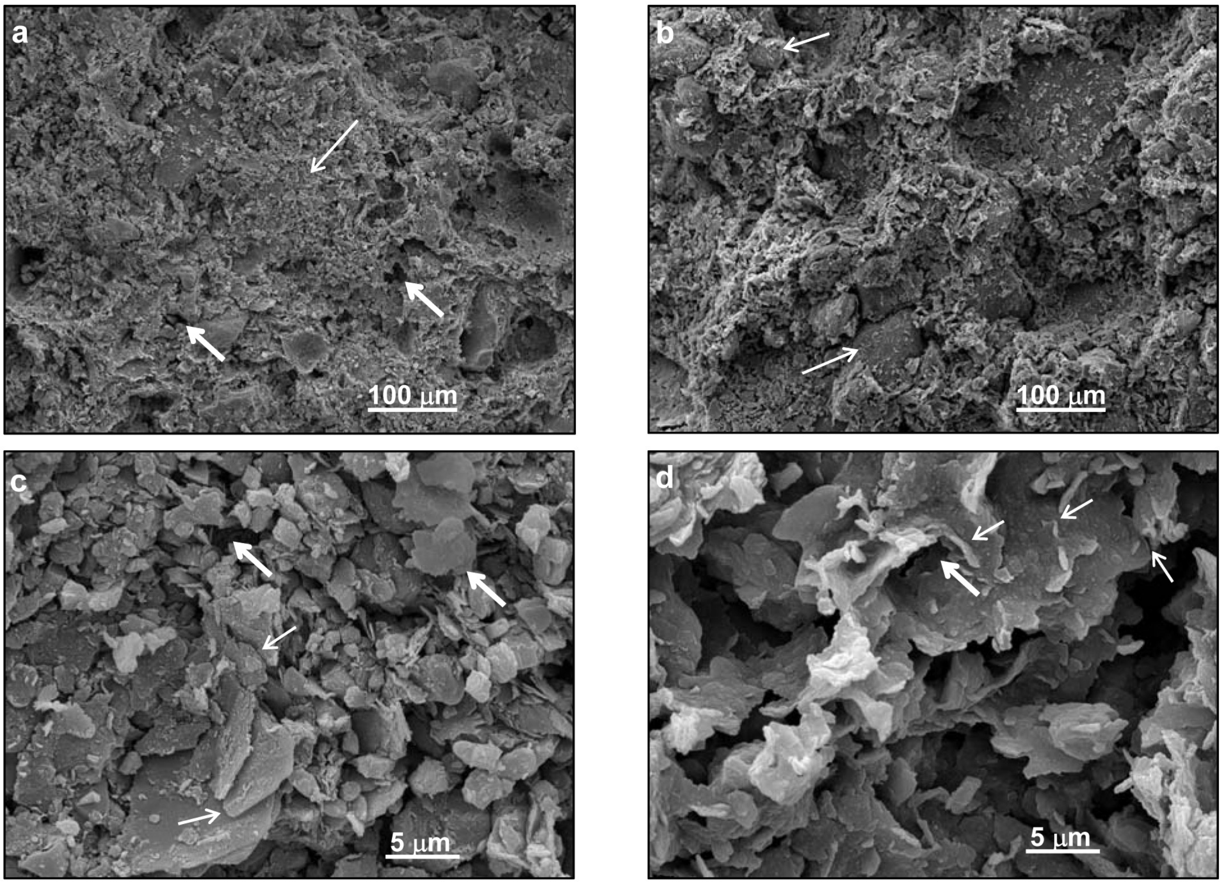

Fig. 4. Matrix microstructure of glacial till: *a* and *c* – NP – non-polluted till; magnification 200×, *b* and *d* – P – till polluted with diesel oil; magnification 3300×. Detailed description in the text

Table 2. **Comparison of microstructural features of non-polluted till (NP) and till polluted with diesel oil (P). Types of contacts: FF – face-to-face, EE – edge-to-edge, EF – edge-to-face**

NP till	P till
The clayey matrix is relatively strongly aggregated	The clayey matrix is significantly less aggregated
Clay particles forming aggregates and microaggregates are densely packed	Clay particles forming aggregates and microaggregates are more loosely packed, and some microaggregates are disintegrated
Clay particles and microaggregates tightly adhere to each other along their surfaces forming a smoother surface	Some clay particles on the microaggregate surface have distinctly warped edges and corners (Fig. 3d – thin upper arrows) forming a rougher surface (compare Figs. 3a and 3b)
Clayey film tightly adheres to the surface of grains, particularly to the silty ones	Clayey envelopes do not adhere tightly to the grain surface
Among clayey microaggregates predominant are contacts of the FF and EE types	Among clayey microaggregates predominant are contacts of the EF type
The amount of intermicroaggregates equals that of interparticle pores	Intermicroaggregate pores are predominant (compare Figs. 3c and 3d)

of the coefficient of variability for P till and its high value for NP till points to a more uniform distribution of porosity in the polluted soil. There was a noticeable drop of the pore amount in P till, coupled with a much lower maximum value of this parameter which, in the absence of changes of the porosity, could suggest significant qualitative and quantitative changes in the pore space of the polluted soil. These changes found confirmation in the distribution of morphometric parameters. The amount of micropores decreased with the growth of the number of mesopores in P till. The redistribution of pore space in favour of larger pores resulted in an increase of the maximum and average pore area, the maximum, minimum and average pore perimeter, the maximum and average pore diameter and in a considerable drop of the total pore perimeter value in P till. The lack of changes in the minimum pore area and pore diameter indicates that pores formed in P till were not smaller than those in NP till.

The variability of geometric pore parameters is reflected in changes of their shape and orientation. The growth of the form index in P till results from the growing amount of pores more isometric in shape, which is confirmed by a decreasing amount of

Table 3. **Quantitative microstructural parameters of non-polluted till and till polluted with diesel oil**

	Non-polluted till[1]					Polluted till[2]				
	Min.	Max.	Average value	Standard deviation	Coefficient of variation, %	Min.	Max.	Average value	Standard deviation	Coefficient of variation, %
Porosity n (%)	19.2	30.8	**24.9**	4.64	19	21.5	26.8	**24.4**	2.12	9
Number of pores $N \times 10^3$	178	1503	**730**	456	62	265	752	**420**	195	46
Total pore area $S_t \times 10^3$ (μm²)	446	2866	**2102**	882	42	1979	2530	**2276**	210	9
Maximum pore area S_{max} (μm²)	9356	130565	**92301**	48697	53	43782	375622	**140862**	135013	96
Minimum pore area S_{min} (μm²)	0.07	0.07	**0.07**	0.00	0	0.07	0.07	**0.07**	0	0
Average pore area S_{av} (μm²)	1.91	4.65	**3.15**	0.96	30	3.36	8.99	**6.09**	2.04	34
Total pore perimeter $P_t \times 10^3$ (μm)	1105	7932	**4027**	2298	57	2240	4267	**2689**	884	33
Maximum pore perimeter P_{max} (μm)	2320	15131	**9900**	4554	46	8089	27827	**15032**	7920	53
Minimum pore perimeter P_{min} (μm)	1.23	1.41	**1.37**	0.08	5.52	1.32	1.58	**1.41**	0.12	8.68
Average pore perimeter P_{av} (μm)	5.28	6.53	**5.73**	0.53	9	5.68	8.52	**6.74**	1.28	19
Maximum pore diameter D_{max} (μm)	109	408	**326**	118	36	236	692	**392**	179	46
Minimum pore diameter D_{min} (μm)	0.30	0.30	**0.30**	0.0	0	0.30	0.30	**0.30**	0.00	0
Average pore diameter D_{av} (μm)	0.70	0.92	**0.80**	0.08	10	0.8	1.13	**0.93**	0.15	15.7
Micropores 0.1 < ∅ < 10 μm (%)	24.6	46.9	**31.6**	9.2	29	17.3	30.8	**21.4**	5.5	26
Mesopores 10 < ∅ < 1000 μm (%)	53.1	75.4	**68.5**	9.2	13	69.2	82.7	**78.6**	5.5	7.04
Maximum form index K_{fmax} (–)	0.922	0.981	**0.950**	0.022	2.36	0.908	0.982	**0.948**	0.032	3.39
Minimum form index K_{fmin} (–)	0	0.046	**0.008**	0.019	245	0	0.023	**0.005**	0.010	224
Average form index K_{fav} (–)	0.34	0.429	**0.398**	0.032	8.12	0.399	0.458	**0.421**	0.028	6.53
Isometric pores a/b < 1.5 (%)	10.8	13.2	**12.2**	1.0	8.4	9.5	17.4	**12.4**	3.1	25
Anisometric pores 1.5 < a/b < 10 (%)	84.8	88.3	**85.9**	1.3	1.48	80.8	88.9	**86.4**	3.2	3.73
Fissure-like pores a/b > 10 (%)	0.6	3.0	**2.0**	1.0	51	0.2	2.7	**1.3**	1.0	77
Anisotropy coefficient K_a (%)	0.78	20.8	**11.2**	7.6	68	2.2	11.7	**6.0**	3.7	63

[1] Number of tests 6.
[2] Number of tests 5.
∅ The equivalent diameter of pore.
a/b The ratio of the two most different dimensions of pore.

fissure-like pores and more abundant isometric and anisometric pores. The change of shape into more isometric is responsible for a significant drop of the microstructural anisotropy coefficient in P till. Because of the subsequent reorientation of particles and microaggregates the contaminated soil becomes more isotropic.

The pollution of the soil ground with diesel oil caused a substantial microstructural transformation of the glacial till in question, which should be related to changes in the chemistry of the pore fluid. Diesel oil is a mixture of hydrocarbons, a non-polar fluid characterized by a low dielectric constant value ε = 2.1. According to double layer theory, a decrease of the ε value in the pore fluid entails a reduction in the thickness of this layer (Verwey, Overbeek, 1948; Mitchell, 1993). Studies by Kaya, Fang (2000, 2005) have confirmed that the lower the ε value, the bigger the drop of the electric potential on the clay particle surface and of interpaticle forces, repulsion forces in particular (Fig. 5). The data presented imply that, as a consequence of partial exchange of water to diesel oil coupled with the reduction of repulsion forces among the clay particles, bonds between the structural elements of the soil were weakened, part of the microaggregates disintegrated, part of the clayey films were detached from the grain surface, edges of some of the clay particles were warped, and the size of pores was redistributed in favour of mesopores.

Changes in the pore space of cohesive soils polluted with organic fluids have been also recognized by, e. g., Fernandez, Quigley (1985), Anandarajah (2003) in samples previously subjected to filtration studies. They have reported the formation of macropores and fissures responsible for the increased permeability of the soils examined.

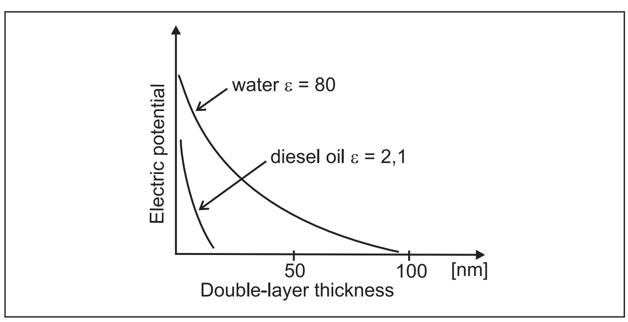

Fig. 5. Schematic diagram of electric potential vs. double-layer thickness of clay particle in water and diesel oil (according to Fernandez, Quigley, 1985 – partly changed), ε – dielectric constant

CONCLUSIONS

In this paper, presented are microstructural changes in glacial till after *in-situ* pollution with diesel oil. Studies were conducted using SEM and STIMAN software for the quantitative microstructure analysis. Statements based on the results obtained are as follows:

1. Glacial till is characterized by a matrix microstructure and a medium packing of microstructural elements.

2. Pollution of the clay soil with diesel oil entailed substantial microstructural changes: relatively loose packing of clay particles and their detachment from grain surface, disintegration of a certain amount of microaggregates, growth of the amount of edge-to-face (EF) contacts among the clayey microaggregates and of intermicroaggregate pores, as well as the warping of corners and edges of clay particles.

3. Polluted till shows a more even porosity distribution and a lower variability of morphometric pore space parameters.

4. In polluted till, due to a redistribution of pore size, the amount of mesopores grew with a drop of the micropore amount.

5. As a result of changes of porosity distribution in polluted till in favour of larger pores, the values of such parameters as the maximum and average pore area, the maximum, minimum and average pore perimeter and the maximum and average pore diameter increased along with a considerable drop of the total pore perimeter.

6. In polluted till, because of the reorientation of structural elements, the microstructure became more isotropic. The amount of fissure-like pores and the microstructural anisotropy coefficient value were found to decrease along with an increase of the average form index.

7. The weakening of structural bonds at the contacts of clay particles and microstructural remodeling in tills polluted with diesel oil can be accounted for by changes of surface properties of clay particles in a non-polar fluid environment with a dielectric constant lower than that of water.

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NAFTOS PRODUKTŲ TERŠALŲ POVEIKIS RIŠLIŲ GRUNTŲ MIKROSTRUKTŪRAI

Santrauka

Straipsnyje pateikiami mikrostruktūriniai moreninio priemolio pokyčiai, atsirandantys dėl naftos produktų teršalų poveikio *in situ* sąlygomis. Tyrimai atlikti nuskaitančiu elektroniniu mikroskopu. Kiekybinei mikroskopinių nuotraukų analizei panaudota STIMAN kompiuterinė programa. Veikiant teršalams moreninio priemolio mikrostruktūra pakito: sumažėjo dalelių ir molio mikroagregatų glaustumas, dalis mikroagregatų suiro, kampai ir briaunos užlinko, padidėjo porų tarp mikroagregatų dydis bei kontaktų briauna – plokštuma (EF) tarp molio mikroagregatų. Dizelinio kuro teršalai sukėlė nemažus kiekybinius priemolio porų pokyčius: padidėjo mezoporų kiekis, maksimalus ir vidurkinis porų skersmuo, porų maksimalus, minimalus ir vidurkinis perimetras. Sumažėjo bendro porų perimetro vertė. Užterštas priemolis tapo labiau izotropinis – tai rodo sumažėjęs mikrostruktūros anizotropijos koeficientas bei didesnė vidurkinė porų formos koeficiento vertė. Nustatyti pokyčiai galėjo atsirasti dėl molio dalelės redukcijos veikiant mažesnės dialektrinės konstantos vertės skysčiui.

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WPŁYW ZANIECZYSZCZEŃ ROPOPOCHODNYCH NA MIKROSTRUKTURĘ GRUNTÓW SPOISTYCH

Streszczenie

W artykule przedstawiono zmiany mikrostrukturalne gruntu spoistego – gliny lodowcowej – spowodowane zanieczyszczeniem olejem napędowym w warunkach *in situ*. Badania zostały wykonane z zastosowaniem skaningowego mikroskopu elektronowego (SEM) oraz programu komputerowego STIMAN do ilościowej analizy obrazu na podstawie zdjęć mikroskopowych. Mikrostruktura gliny uległa wyraźnym zmianom jakościowym na skutek zanieczyszczenia: upakowanie cząstek i mikroagregatów ilastych zmniejszyło się, część mikroagregatów rozpadła się, uległy podgięciu krawędzie i naroża pojedynczych cząstek ilastych, zwiększyła się ilość porów międzymikroagregatowych oraz kontaktów typu krawędź–płaszczyzna (EF) pomiędzy mikroagregatami ilastymi. Zanieczyszczenie olejem napędowym wywołało znaczne zmiany ilościowe w przestrzeni porowej gliny. Wzrosła liczba mezoporów, maksymalna i średnia powierzchnia porów, maksymalny, minimalny i średni obwód porów, maksymalna i średnia średnica porów, a spadła wartość całkowitego obwodu porów. Zanieczyszczona glina stała się bardziej izotropowa, na co wskazuje spadek wartości wskaźnika anizotropii mikrostruktury i liczby porów szczelinowatych, oraz wzrost średniej wartości współczynnika formy porów. Zaobserwowane zmiany mogły powstać w wyniku redukcji sił wzajemnego oddziaływania pomiędzy cząstkami ilastymi po zanieczyszczeniu gruntu cieczą o niższej niż woda wartości stałej dielektrycznej.

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ВЛИЯНИЕ ЗАГРЯЗНЕНИЯ НЕФТЕПРОДУКТАМИ НА МИКРОСТРУКТУРЫ СВЯЗНЫХ ГРУНТОВ

Резюме

Показаны изменения микроструктуры моренных суглинков при загрязнении нефтепродуктами в условиях *in situ*. Исследования проводились сканирующим электронным микроскопом. Для количественного анализа микроскопических снимков использовалась компьютерная программа STIMAN. Под воздействием загрязнения микроструктура моренных суглинков изменилась: уменьшилась плотность упаковки частиц и микроагрегатов глины, часть микроагрегатов разрушилась, углы и ребра загнулись, увеличивались поры и количество контактов ребро–плоскость (EF) между агрегатами глин. Соответственно увеличились число мезопор, максимальный и средний диаметр, максимальный, минимальный и средний периметр пор. Уменьшилось значение общего периметра пор. Загрязненный суглинок стал более изотропным. На это указывают уменьшение коэффициента анизотропии микроструктуры и увеличение среднего значения коэффициента формы пор. Указанные изменения могли произойти из-за редукции глинистых частиц под воздействием жидкости с меньшим значением диэлектрической константы.