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Application of the Scanning Electron Microscopy in Soil Fine-Dispersive Fraction Investigations

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This article presents methodological investigations of morphology and particles composition of fine-disperse fraction of the soils developed in moraine and glacial lacustrine deposits. The fine-disperse fraction from bulk soil was separated by a principle of peptization in distilled water according to Gorbunov's method. Following the generally accepted strategy, the XRD method for determination of mineralogical composition of fine-disperse fraction has been used. The scanning electronic microscopes JEOL-35 was used to express mineral formation of separate particles or various their aggregations.

The investigations data obtained have shown substantial differences in morphology and particles composition of fine-disperse fraction of the different soil formation. These results are to be used in further investigations for modelling of heavy metals transport in soil.

Keywords: soil, scanning electron microscopy, morphology.

1. INTRODUCTION

Soils are complex assembles of minerals, organic material, living organisms, water, and gases. The size, shape, and chemical composition of soil minerals may be highly variable. For this reason, observation of soil mineral morphology in the study of soils is very important [1 – 4].

The dispersion of heavy metals as a result of industrial activity of man is a serious ecological problem at present. High toxicity of heavy metals, their interaction in circuits of materials in agrarian activity, the ability of accumulation in the fabrics represents a real ecological threat. The necessity of study heavy metals distribution in a soil cover is caused by nonstationarity of transfer processes which carry heavy metals from the atmosphere into the ground.

The clayey fraction of high dispersivity is the main component, due to which sorption and exchange processes occur in soil (such as nourishing and also toxic materials). So, the clayey fraction of soil is the basic component in which heavy metals are collected.

The clayey fraction represents a wide set of silicate minerals of various dispersivity and sorption and exchange properties, which cause transfer and accumulation of heavy metals in various soil horizons. One of the usual methods of mineralogical investigation of soil structure is X-ray diffraction (XRD) of powder [5]. However, XRD method of the analysis does not allow to obtain any information about dispersity, shape and distribution of the sizes of particles of a clay fraction. On the other hand the electron microscopy analysis of a clayey fraction of colloidal suspension deposits strongly supplements the results of XRD analysis.

The purpose of our work - to study the morphology of fine-dispersive fraction (< 0.005 mm) obtained from the soil samples of various depth horizons.

2. MATERIALS AND METHODS

2.1. Study Area and Soil Samples

The relief in the territory of Lithuania was essentially formed by the glaciers of thick layer several glaciations (3 – 5), which left a thick layer of glacial deposits of 100 – 300 metres in highlands and 20 – 40 metres in lowlands. The surface layer of the deposits where the present cover of the soil was formed and left by Middle Pleistocene (Q_{II}) and Upper Pleistocene (Q_{III}) glaciers.

The study area is located in the Middle Lithuanian Lowland Zone, Region of loamy calcareous *Cambisols*. There are the Central Lithuanian glacier edge formations which are represented by scarce morainic massifs and glacial lacustrine sediments. The land surface absorbs the greatest amounts of radiation in June (490 MJ/m^2), while least amount is absorbed in December – January (20 – 25 MJ/m^2). The annual amount of the absorbed radiation in various landscapes may change approximately 2600 – 3100 MJ/m^2. The mean annual precipitation amount is to 675 mm (the zone of excessive humidity). The absolute age of soil is young – of the Holocene period (<10 000 years). Soil formation proceeds through the *Cambisol* → *Luvisol* → *Albeluvisol* → *Podzol*.

Object of the investigation – fine-dispersive fraction (< 0.005 mm) of the *Calcari Epihypogleyic Luvisol*, ***LVg-p-w-cc*** (FAO-UNESCO, 1998) alkaline reaction soil placed near highway Vilnius – Kaunas – Klaipėda. This road is in function since 1970 and in our days it seems to be among the most important sources of the environment pollution with heavy metals in Lithuania [5, 6]. Selected soils were formed in moraine and glacial lacustrine sediments, a distance from seats of technogenic pollution - 20 and 40 m. Depending on the heterogeneity of the ground, representative soil plots ($10 \cdot 10$ m^2) for sampling have been delineated. The 0 – 10, 10 – 20, 20 – 30, 40 – 50 and 90 – 100 cm layers (reflecting main horizons of the

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soil) have been taken, the description of soil profiles from the soil pit outside (but near to) the selected plot was made. The fine-dispersive fraction of the soil by a principle of peptization in distilled water according to Gorbunov's method [7].

2.2. Analyses of Fine-Dispersive Fraction Mineralogy and Morphology

To estimate the content of crystalline minerals in the fine-dispersive fraction [3] there has been used a semiquantative XRD method. Diffractograms for the treatment were performed using CuK_α radiation, produced by DRON-3 instrument (Burevestnik, Sankt-Peterburg, Russia) operating at 30 kV voltage and 30 mA current; the scanning rate was 2 grad / min. X-ray diffraction was performed on oriented samples for the clay fractions.

Scanning electron microscopy (JEOL-35) was performed on fine-dispersive fraction specimens spotted onto Si plates with very little roughness from sediments by colloid clay fraction solution.

For quantitative electronic microscopic investigation preparations of samples becomes very important and, besides, such a procedure combines two particular actions:

- selection of very low concentration fine- dispersive fraction, attempting if possible, after drying suspension, to maximum separate particles into monomineral aggregates on the surface,
- coating of conductive layer in order to reduce charge effect.

Prior to the preparation of the samples for SEM, the fine-dispersive fraction was treated by ultrasonic vibration of 20 kHz for 5 minutes.

3. RESULTS

The dispersity of soil fine-dispersive fraction is particularly important in two physical and chemical processes that occur dusting heavy metals absorption into particles surface and their exchange with internal volumetric particles.

The X-ray phase analysis of fine-dispersive fraction is given in Tables 1 and 2. In essence it is a mixture of illite, kaolinite, smectite and quartz, but in some samples there is vermiculite and chlorite. The last one was found only in moraine soil formation material.

Table 1. Mineral composition of fine-dispersive fraction of the glacial lacustrine soil-forming material

	Sample taking depth, cm				
	0 – 10	10 – 20	20 – 30	40 – 50	90 – 100
Chlorite	6	7	6	13	15
Illite	53	55	62	66	59
Smectite	15	–	–	–	–
Vermiculite	–	3	5	–	5
Caolinite	25	34	26	20	18
Quartz	1	1	1	1	3

Table 2. Mineral composition of fine-dispersive fraction of the moraine soil-forming material

	Sample taking depth, cm				
	0 – 10	10 – 20	20 – 30	40 – 50	90 – 100
Chlorite	8	7	20	18	10
Illite	68	60	46	51	60
Smectite	–	6	9	8	6
Vermiculite	–	–	–	–	4
Caolinite	23	26	22	21	18
Quartz	1	1	3	2	2

Scanning electron microscopy (SEM) of clay fractional dispersive fraction allows to investigate separate components of the minerals, their aggregates, to define more exactly series of mineral fractions (when there is good pictures coincidence with the standard pictures of the identified minerals).

SEM allows to determine the morphologic as properties of minerals (plane, scaly, tube-like, etc. structure), the extent of their crystallization and disintegration, presence of insertions. When there is a certain experience, it is possible to determine approximately also mineral kind of clay. Clay fraction SEM photographs are presented in Figures 1 – 10. It may be seen that there was not possible to avoid absolutely aggregate products. However, parts of the whole area are illustrated in separate parts, the size of which is smaller than 1 μm. All clayey minerals are classified into two groups: I - isometric and II - lengthened particles shape. As may be seen from the literature sources, in SEM photographs of muscovite particles - they are particles of irregular angular shape, plane and most often isomeric [3]. Illite, smectite group very little differs from that of muscovite one because there is a transitional form in a clay illitization stage. Particles shapes most often are scaly and plane and irregular. It is possible to define more clearly quartz grains – there clearly are exposed crumbly particles, which are opaque and with well exposed edges.

Fig. 1 and Fig. 2 illustrate SEM photographs of suspension of fine-dispersive fraction from the depth of 0 – 10 cm for glacial lacustrine and moraine soils.

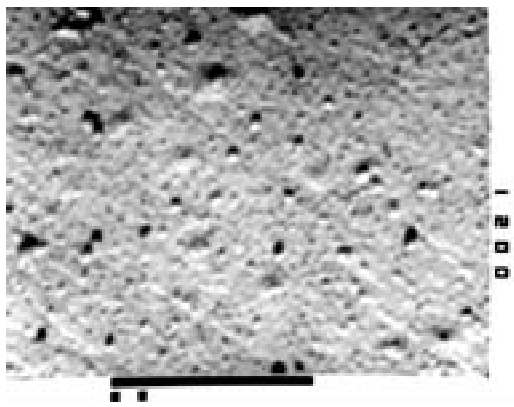

Fig. 1. SEM photograph of fine-dispersive fraction of the glacial lacustrine material in depth 0 – 10 cm (marker value 10 μm)

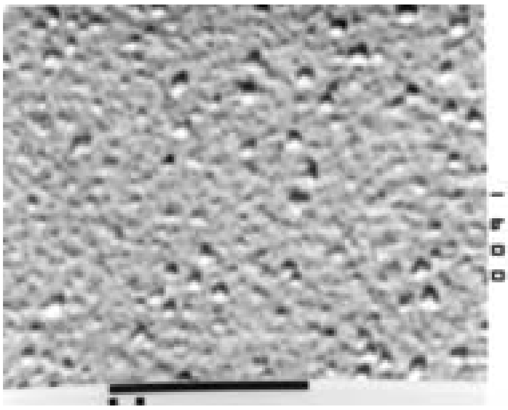

Fig. 2. SEM photograph of fine-dispersive fraction of the moraine material in depth 0 – 10 cm (marker value 10 μm)

A quartz mottling of 0.1 – 0.3 μm size particle of isometric shape in glacial lacustrine material may be seen in Fig. 1. Absence of smectite scales in the photograph may be explained by masking effect of fine-dispersive illitic phase.

On the illustrated moraine fraction (Fig. 2) one may see practically monodispersive globules of minerals. Better uniformity of fine-dispersive fraction of moraine origin is defined by the nature of the soil surface layer formation. Kaolinite concentration in both samples is apparently not sufficient, that its particles might be identified in photographs.

Fig. 3 and Fig. 4 show morphology of fine-dispersive fraction of soils from samples selected in depth of 10 – 20 cm.

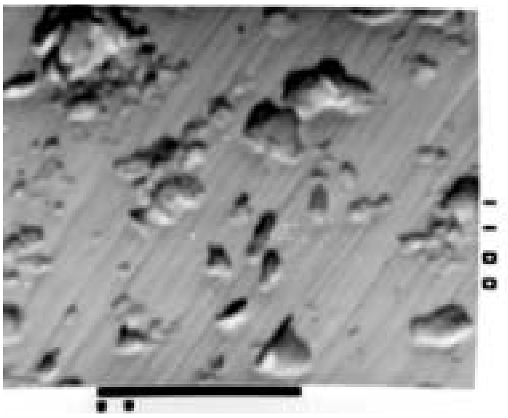

Fig. 3. SEM photograph of fine-dispersive fraction of the glacial lacustrine material in depth 10 – 20 cm (marker value 10 μm)

The morphology of fine-dispersive fraction of glacial lacustrine soil appears as a selection of scaly aggregates with wide dispersion of dimensions from 0.5 to 5 – 7 μm. Apparently, electrolytic properties, when the sample was being formed, caused aggregation of illite and vermiculite in flakes of asymmetric shape. The fact that particles are composed of plane aggregates (scales), may be explained by nonuniformity of optical plane of particles - there are darker and lighter portions.

In Fig. 4 (moraine material) one may see a great number of isometric dark particles on the background of still finer gray portions, i. e. mainly particles of 0.1 – 0.5 μm of irregular shape. In some places of the photograph there may be seen aggregated accumulations.

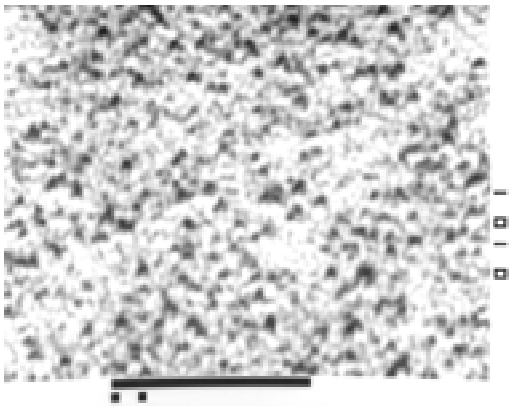

Fig. 4. SEM photograph of fine-dispersive fraction of the moraine material in depth 10 – 20 cm (marker value 10 μm)

In Fig. 5 and Fig. 6 morphology of fine-dispersive fraction of soils from depth of 20 – 30 cm is shown.

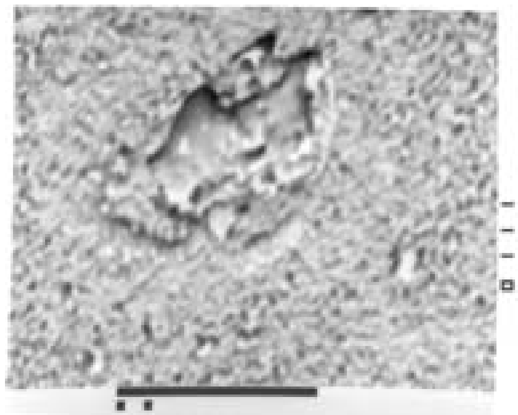

Fig. 5. SEM photograph of fine-dispersive fraction of the glacial lacustrine material in depth 20 – 30 cm (marker value 10 μm)

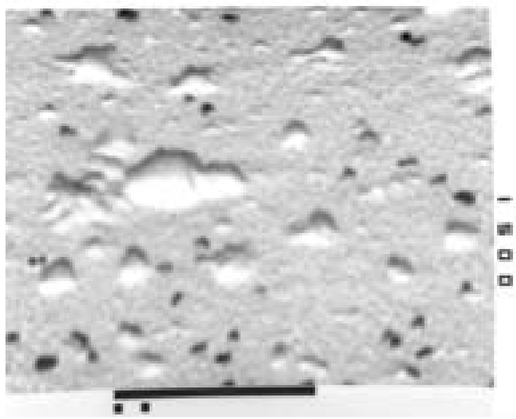

Fig. 6. SEM photograph of fine-dispersive fraction of the moraine material in depth 20 – 30 cm (marker value 10 μm)

The morphology of fraction from glacial lacustrine soil is represented by plane aggregate of asymmetric shape with sizes of about 10 μm in longer axis with growth in perimeter also plane particles of fine size. The remaining plane of illustration - fine particles of irregular and isometric shape of 0.1 – 0.5 μm size. Most likely, the centre aggregate in photograph - a joint of illite and vermiculite, fine aggregate - particles of caolinite of various orientation.

In Fig. 6 (moraine material) are most successfully shown two types of mineral particles - illite and smectite aggregates composed of from scales and dark fine quartz mottling of 0.1 – 0.5 μm size, the concentration of which is in this fraction the greatest.

Fig. 7 and Fig. 8 illustrate morphology of fine-dispersive fraction from soils in the depth of 40 – 50 cm.

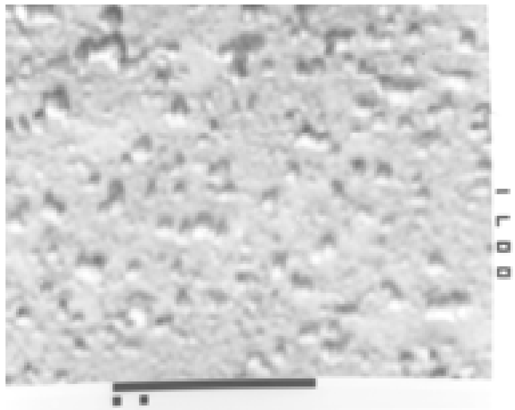

Fig. 7. SEM photograph of fine-dispersive fraction of the glacial lacustrine material in depth 40 – 50 cm (marker value 10 μm)

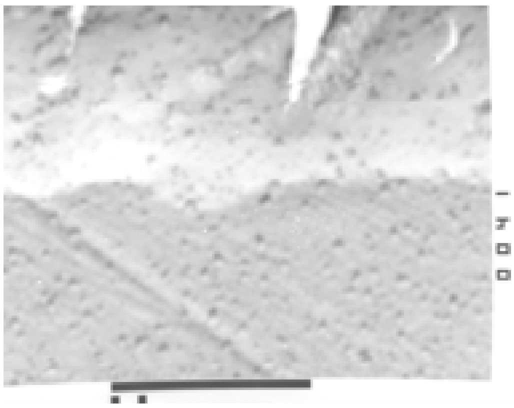

Fig. 8. SEM photograph of fine-dispersive fraction of the moraine material in depth 40 – 50 cm (marker value 10 μm)

In glacial lacustrine material (Fig. 7) – a great number of asymmetric particles of various optic density – formation and accumulation of illite and caolinite of particles size from 0.1 to 2 μm.

In moraine material (Fig. 8) may be seen only fine particles of 0.1 – 0.3 μm. size. Separate particles are of isometric shape. To identify a definite mineral fraction is not possible.

Fig. 9 and Fig. 10 represent photographs of fine-dispersive particles from the soils in depth 90 – 100 cm.

In glacial lacustrine material (Fig. 9) may be seen a great number of asymmetric flakes of various sizes - from 0.1 to 10 μm - most likely aggregates of plane scales of illite and kaolinite.

In moraine material (Fig. 10) may be seen uniformly dispersed particles of rounded shape 0.1 – 0.5 μm; practically they are not accumulated into aggregate complexes.

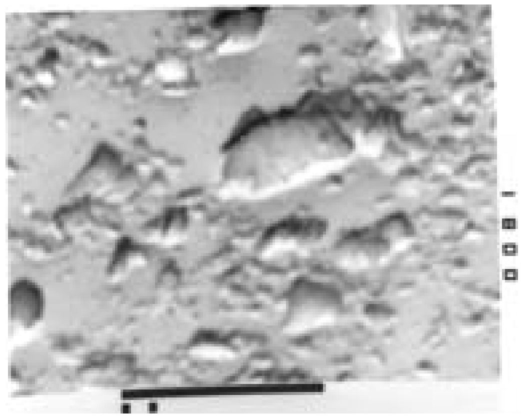

Fig. 9. SEM photograph of fine-dispersive fraction of the glacial lacustrine material in depth 90 – 100 cm (marker value 10 μm)

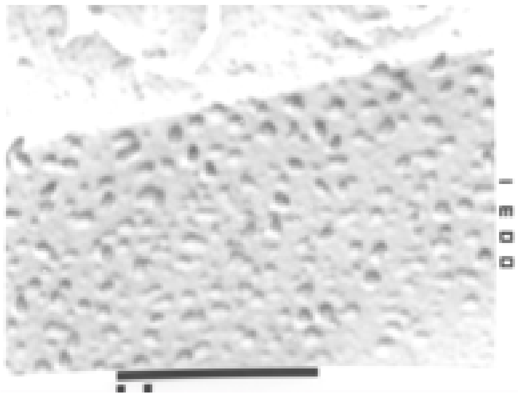

Fig. 10. SEM photograph of fine-dispersive fraction of the moraine material in depth 90 – 100 cm (marker value 10 μm)

The results of X-ray phase analysis and study of soils fine-dispersive fraction by SEM makes possible to identify separate minerals and to assess their sizes and shapes. Nevertheless, unambiguous visual identification and confirmation of the presence of all minerals in a sample was quite probabilistic. Most likely, without separation of fine-dispersive fraction for sufficiently narrow in sizes fractions, cover of larger particles upon the fine ones and total adherence into aggregates will shade the identification of morphologic characters of mineral particles.

4. CONCLUSIONS

In all horizons of taken samples of glacial lacustrine and moraine soils mineralogical composition is defined basically by the presence of illite and kaolinite. In the fraction of moraine origin smectite is found, while in glacial lacustrine one – some vermiculite. A small concentration of quartz may also be found in all tested samples.

Morphological analysis by SEM photographs of fine-dispersive fraction has shown that there does not occur any evident division of particles into separate identifiable minerals. Only quartz particles may be identified unambiguously. Illite, smectite and vermiculite form asymmetric flakelike aggregates of various sizes.

The separation of fine-dispersive fraction into smaller in size fractions by sedimentation technique may enrich the composition of these fractions with one or another material, while a complex morphological analysis of electronic-microscopic photographs of all these fractions will allow to identify all minerals in a sample.

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