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Heavy minerals in the study of soil: techniques, their limitations and advantages

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Different methods used in heavy mineral studies of soil samples were evaluated in accordance with mineralogical research of Lithuanian soil.

The main requirement of quantitative mineralogical analysis is precision, which mostly depends on the number of grains counted. Change of the physical properties of minerals during their reworking both in Quaternary and older sediments, the mechanical comminution during glacial transportation and postglacial weathering, soil formation processes and diversity of the chemical composition of some mineral species predetermine that light microscopy alone is insufficient to identify all mineral species, moreover, it is a time-consuming method for quantification of all mineral distribution.

Scanning electron microscopy is the most frequently used auxiliary technique for mineral identification. It is distinguished by several notable advantages such as high resolution, depth-of-field and microanalysis, enabling a quick mineral identification according to their spectra and adequate chemical composition.

Assemblages, distribution and chemical composition of discrete grains can be used for different purposes including determination of mineral provenance, tracing transport paths of soil-forming sediments, mapping soil layers of different origin, outlining areas of distribution of various soil types and explaining the origin of clastic geochemical anomalies in soil.

Key words: heavy mineral fraction, precision of mineral identification, binocular microscope, scanning electron microscope

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INTRODUCTION

Heavy mineral fractions separated from soil samples are often represented by various mineral species. Their assemblages, distribution and the chemical composition of discrete grains can be used for different purposes such as determination of mineral provenance, tracing transport paths of soil-forming sediments, mapping soil layers of different origin, outlining areas of distribution of various soil types and explaining the origin of clastic geochemical anomalies in soil.

Mineralogical studies of heavy minerals in soil began in the middle of the 20th century. Since then many publications have considered the role of soil heavy minerals in different aspects. The mineralogy of bulk samples from soil (horizons E and B) and till (horizon C) has been used for studying the podzolization effect expressed with respect to changes in hornblende, biotite, K-feldspar, quartz, muscovite_and plagioclase contents (Melkerud et al., 2000). Mineralogical studies of soil developed on a Pb-mineralized sandstone in France revealed precipitation of Pb as plumbogummite, which was considered as the main speciation form of Pb in this area (Morin et al., 2001). As soil minerals are one of the sources for nutrients, their weathering processes in soils have been intensively studied. Some

minerals distinguished by low stability in conditions of acidic weathering have been used for the diagnostics of acidic leaching resulting in the depletion of these minerals (Morton, 1986). The order of heavy mineral stability in till was set by Lång (2000) who studied mineral weathering under acidic soil conditions. Accumulation of certain elements in <0.1 mm Lithuanian soil (horizon A_1) analyzed by chemical methods was resolved by studying primary heavy minerals in the corresponding soil fraction (Vareikienė et al., 2003). The host minerals for the selected elements were determined and quantified, and the accumulation of these elements was explained by their high contents in carrier phases.

The aim of this the current investigation was to evaluate the methods used in the study of heavy minerals as part of research related to explaining the origin of geochemical anomalies in Lithuanian soil (horizon A_1). Therefore, it is important to discuss the mineralogical methods applied to the study of heavy minerals from soil samples, evaluating their advantages and limitations. Such analysis could be a valuable practice for applying the discussed methods for a mineralogical analysis of differently originated samples (glacial sediments, marine bottom sediments).

MATERIAL

The soil samples studied were taken from the A_1 horizon (5–15 cm depth). The upper A_0 horizon of soil enriched in organic matter was mechanically removed before sampling; all coarse organic particles were removed by hand during sampling.

The samples were dried and dry-sieved to 2 mm fraction. During pre-treatment, hydrogen peroxide at concentrations gradually increasing from 3 to 30% were used to destroy the rest part of organic matter in the sieved soil samples. After pre-treatment, inorganic matter was dry-sieved and fractions 0.5–0.25, 0.25–0.1 and <0.1 mm were chosen for further mineralogical investigation. For grain size fractions >0.1 mm, heavy liquid with a density (*d*) 3 g/cm³ was used, and slightly diluted bromoform (*d* 2.78 g/cm³) was chosen to separate the fine fractions. Polished thin sections of grain size fractions 0.5–0.25, 0.25–0.1. <0.1 mm were prepared on glass plates by mounting the mineral grains in Epofix resin.

MINERALOGICAL TECHNIQUES

The precision of heavy mineral analysis greatly depends on the number of grains counted on a microscope slide or a thin section. As far back as 1931, Dryden illustrated graphically the reliability of heavy mineral data in relation to the number of counted grains and observed a rapid increase in the accuracy when the counts exceeded 300 (Dryden, 1931).

When counting not only main but also rare mineral species, it is usually necessary to identify a bigger amount of minerals. The study of heavy mineral separates from the anomalies in Lithuanian soil showed that some rare mineral species (xenotime, chromite, pyrite, monazite) were encountered only when more than 1500 grains were identified (Table 1).

Table 1. **Results of grain counting of heavy minerals (density (*d*) >3 g/cm³) from Lithuanian soil samples using scanning electron microscope (SEM) equipped with energy dispersive spectrometer (EDS)**
1 lentelė. **Lietuvos dirvožemio mėginių sunkiųjų mineralų (*d* >3 g/cm³) paplitimo analizės rezultatai panaudojus elektroninį skenuojantį mikroskopą (SEM), sujungtą su rentgenospektrometru (EDS)**

Mineral	Number of grains	Total number*	Fraction	Location of soil sample
Chromite	1	1707	0.25–0.1	Middle Lithuanian plain (near Kėdainiai town)
Kyanite	4	1707	0.25–0.1	Middle Lithuanian plain (near Kėdainiai town)
Pyrite	1	1534	0.25–0.1	Mūša-Nemunėlis glaciolacustrine basin
Xenotime	1	1534	0.25–0.1	Mūša-Nemunėlis glaciolacustrine basin
Tourmaline	2	1828	0.25–0.1	Ašmena highland (preconcentrated heavy mineral separate)
Xenotime	2	1828	0.25–0.1	Ašmena highland (preconcentrated heavy mineral separate)
Chromite	3	1828	0.25–0.1	Ašmena highland (preconcentrated heavy mineral separate)
Kyanite	3	1828	0.25–0.1	Ašmena highland (preconcentrated heavy mineral separate)
Chromite	4	2208	<0.1 mm	Mūša-Nemunėlis glaciolacustrine basin
Staurolite	3	2208	<0.1 mm	Mūša-Nemunėlis glaciolacustrine basin
Pyrite	1	2208	<0.1 mm	Mūša-Nemunėlis glaciolacustrine basin
Xenotime	1	2208	<0.1 mm	Mūša-Nemunėlis glaciolacustrine basin

*Total number of mineral grains indentified in thin sections of heavy mineral separates.

Therefore, the requirement of precision has to be considered carefully when choosing an appropriate mineralogical method to quantify heavy mineral distribution.

Light microscopy. Widely used for mineralogical investigations, the binocular stereomicroscope can be applied to mineral identification in soil samples only cautiously to avoid misinterpretation of further results. Using this technique, the main difficulty lies in a reliable identification of minerals because of their physical properties changed due to many factors including the reworking effect in Quaternary and older sediments, mechanical comminution during glacial transport, postglacial weathering and soil formation processes. When working with the stereomicroscope, soil minerals usually need an additional treatment to liberate the grains from adhering clays or iron oxide coatings. The removal of the latter involves chemical reagents affecting some minerals groups.

Polished thin sections of heavy minerals mounted in Epofix resin can be studied more precisely under a polarizing microscope. However, in spite of the improved capacity of identification of soil minerals, problems with their reliable identification cannot be avoided. The changed physical properties and the chemical complexity of some minerals imply that optical observations alone are inadequate to recognize particular species, and for better identification other mineralogical analyses are needed. The above-mentioned optical methods are widely used for mineral identification, but they are too time-consuming for the quantification of mineral distribution, especially when not only major but also rare heavy accessories should be identified and counted.

Over the recent years, additional advanced techniques have been employed in heavy mineral research. These new techniques provide information both on the chemical composition and structural-textural characteristics and thus considerably increase the accuracy of heavy mineral analyses, permitting more confident interpretations.

Scanning electron microscope (SEM) is probably the most frequently used auxiliary instrument in heavy mineral studies. The SEM has several notable advantages such as high resolution (1 nm at magnifications of up to 1 million times), depth-of-field (characterizing the extent to which the image resolution degrades with distance above or below the best focus plane), and microanalysis (the ability to analyze sample composition).

SEM has been used, for example, to identify minerals, to map element distributions in specimens, to determine mineral phases causing anomalies in heavy-mineral samples, as well as to examine their surface textures and to distinguish their shape (To-

vey and Krinsley, 1991; Protz et al., 1992; Krinsley et al., 1998).

A modern SEM is usually equipped with an energy-dispersive X-ray spectrometer (EDS), which enables an elemental analysis and thus allows a quick mineral identification. Robson (1982) described a computerized SEM-EDS technique identifying and counting the constituents including heavy minerals of unconsolidated marine sediments. Knight and others (2002) characterized grain mineralogy and quantified mineral abundances using computer-based image analysis protocols and combinations of EDS element images.

With EDS, the emitted X-ray radiations are measured on the basis of their energies. Characteristic X-rays collected by the X-ray spectrometer are counted, sorted and plotted as a graphical spectrum in which peaks correspond to elements present in the mineral. The abundance of elements and the intensity of their peaks are used as the main source for mineral identification (Fig. 1). For instance, a high peak of Ti to-
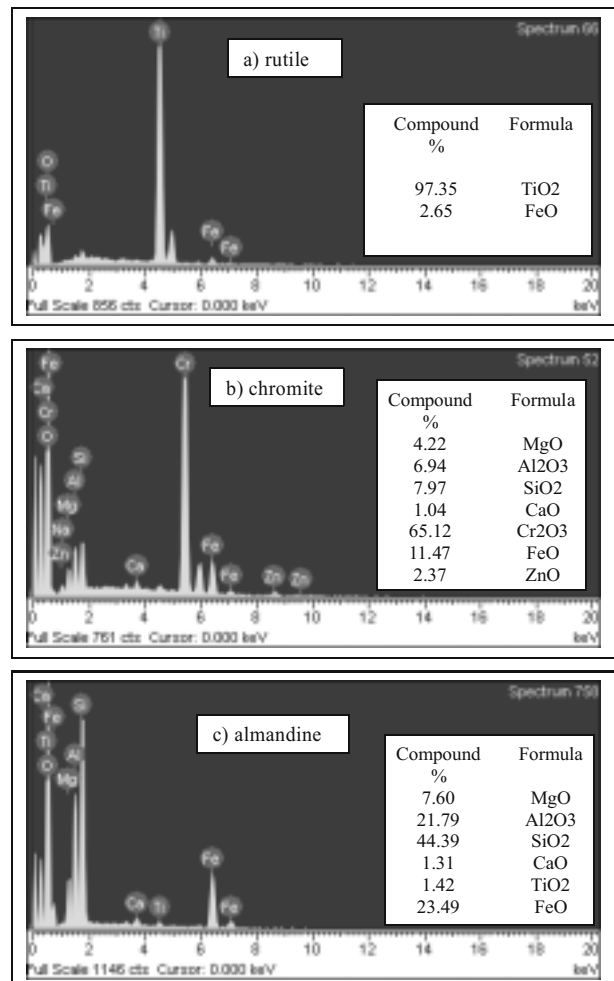

Fig. 1. SEM-EDS spectra of some heavy minerals with corresponding chemical compositions
1 pav. Atskirų sunkiųjų mineralų spektrai su atitinkama chemine sudėtimi panaudojus elektroninį skenuojantį mikroskopą, sujungtą su rentgenospektrometru

gether with a very low content (or complete absence) of Fe is characteristic of rutile (Fig. 1a). A high peak of Cr and considerably lower peaks of Fe, Mg and Al are diagnostic for chromite (Fig. 1b), while identifying garnets not only the intensity of element peaks but also combinations of their contents are essential for mineral identification (Fig. 1c).

SEM-EDS analysis can be applied to discrete mineral grains or to polished thin sections. The analysis is reliable only for major elements in a mineral, *i.e.* with concentrations higher than 1%, but usually that is enough for mineral identification. All major components in a mineral are measured simultaneously. SEM-EDS can be used also in compiling distribution maps of selected elements in a specimen by analyzing the whole field of view instead of making single point analyses.

Polished thin sections are best suited for quantification of mineral distribution, because they can be studied repeatedly. Specific areas or mineral grains in a sample can be re-imaged and re-analyzed if needed, and the same preparations are suitable for other instruments such as optical microscope and microanalyzer.

SEM-EDS ANALYSIS OF MINERALS FROM LITHUANIAN SOIL SAMPLES

Samples from Lithuanian geochemical soil anomalies were studied using a back-scattered electron image (BEI) analysis by SEM JEOL-5900LV with LINK EDS Oxford Instruments. A semiquantative mineralogical analysis was performed on polished thin sections prepared from a heavy mineral ($d > 3$ g/cm^3) of the soil samples. More than 1500 mineral grains were identified and counted from each thin section to get reliable results. Back-scattered images were taken systematically in order to cover equally all parts of a thin section and to ensure representativeness of the results. According to grain size and technical parameters required for high quality images, 10–15 to 80–100 heavy mineral grains were identified and counted in every image taken from a thin section (Fig. 2).

A contrast in back-scattered images (BEI) is derived primarily from point-to-point differences in the average atomic number of elements in the mineral. Although BE images are not always easy to inter-
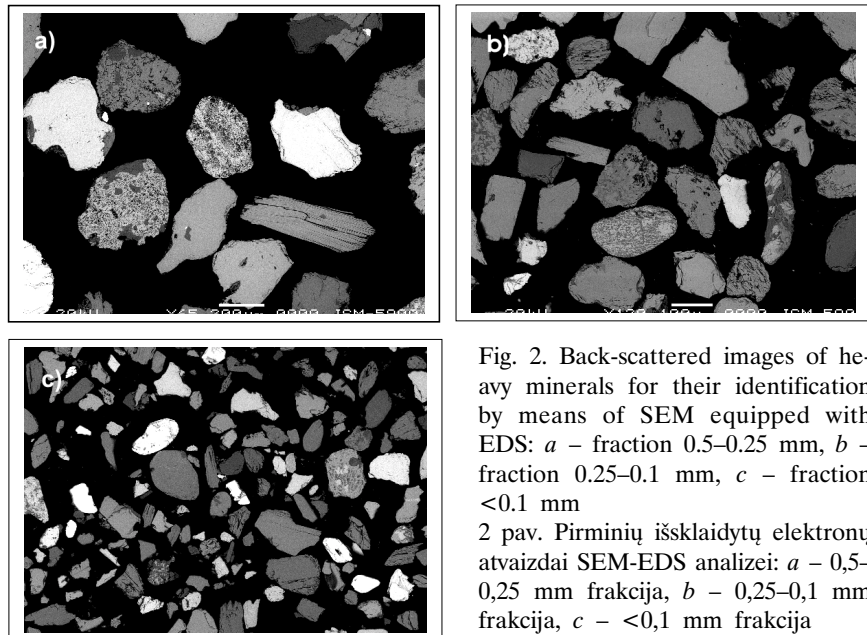

Fig. 2. Back-scattered images of heavy minerals for their identification by means of SEM equipped with EDS: *a* – fraction 0.5–0.25 mm, *b* – fraction 0.25–0.1 mm, *c* – fraction <0.1 mm

2 pav. Pirminių išsklaidytų elektronų atvaizdai SEM-EDS analizei: *a* – 0,5–0,25 mm frakcija, *b* – 0,25–0,1 mm frakcija, *c* – <0,1 mm frakcija

pret, they can be useful evaluating volume proportions of separate minerals phases in a mixed grain, based on the different brightness of the minerals. The visually distinguished mineral phases can be then confirmed by EDS analysis (Fig. 3).
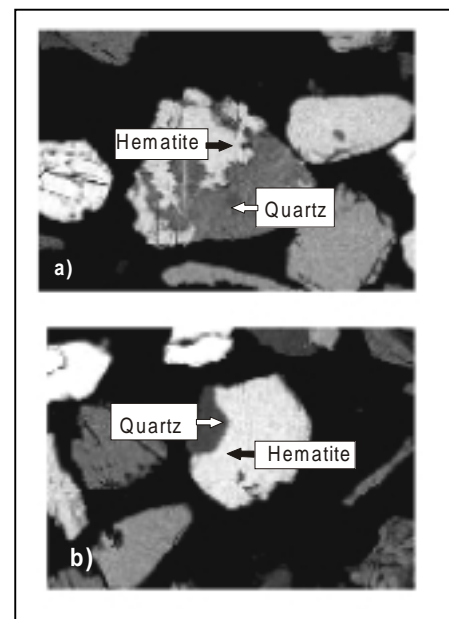

Fig. 3. Back-scattered images for counting minerals according to their volumes in mixed grains

3 pav. Pirminių išsklaidytų elektronų atvaizdų panaudojimas kiekybinei mineralų paplitimo analizei jų sąaugose

In Lithuanian soil samples, the phase comprising more than 50 vol. % of mixed grain was chosen as the main mineral phase (Fig. 3b). In case of almost equal areal distribution of different minerals in a

bigger aggregate, both phases were counted as separate minerals. Usually aggregates consisting of more than two mineral phases were not frequent. Using this approach for determining the relative abundance of different heavy minerals in a sample expressed as volume per cent, the precision of the results was increased.

When identifying minerals by SEM-EDS, it must be borne in mind that the "sinking effect" of heavy minerals occurring in thin sections can be an abstacle in a reliable identification of flaky and strongly weathered mineral grains. Also, distortion of results because of a wide grain size variation cannot be avoided when studying the distribution of heavy minerals by SEM-EDS. This negative effect can be minimized by limiting grain size.

CONCLUSIONS

The precision of the quantitative mineralogical analysis mostly depends on the number of identified grains, because the study of heavy mineral separates from anomalies in Lithuanian soil showed that some rare species (xenotime, chromite, pyrite, monazite) were encountered only when more than 1500 mineral grains were identified.

Change of the physical properties of minerals during their reworking both in the Quaternary and older sediments, mechanical comminution during glacial transportation and postglacial weathering, soil formation processes and the diversity of the chemical composition of some mineral species predetermine that light microscopy alone is insufficient to identify all mineral species, moreover, it is a time-consuming method for quantification of mineral distribution. Scanning electron microscope is the most frequently used auxiliary technique for mineral identification, distinguished by several notable advantages such as high resolution, depth-of-field and microanalysis. Modern SEMs are usually equipped with an energy-dispersive X-ray spectrometer (EDS) for elemental analysis, enabling quick mineral identification according to their spectra and adequate chemical composition.

Applying SEM-EDS for mineralogical investigation, the proportions of different minerals in a mixed grain can be evaluated, based on the contrast in backscattered images and the EDS data. Using such an approach in assessing the relative abundance of different heavy minerals expressed as volume percentage, the precision of the results can be increased.

The "sinking effect" of heavy minerals on thin sections usually causes some difficulties in reliable identification of flaky and strongly weathered mineral grains. The distortion caused by variability of grain size could not be avoided when studying the

mineral distribution by SEM-EDS. Such an effect can be minimized by limiting the grain size of heavy minerals.

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SUNKIEJI MINERALAI DIRVOŽEMIO TYRIMUOSE: MINERALOGINIAI METODAI, JŲ PRIVALUMAI BEI TRŪKUMAI

S a n t r a u k a

Dirvožemio sunkiųjų mineralų grūdelių kiekybinio pasiskirstymo analizės rezultatai plačiai naudojami dirvožemio tyrimuose: nustatant dirvodarinių nuogulų kilmę, kartografuojant skirtingų dirvožemio tipų paplitimą plote, identifikuojant atskirus dirvožemio sluoksnius, aiškinant geocheminių anomalijų dirvožemiuose kilmę. Pasiskirstymo analizės patikimumas priklauso nuo identifikuotų mineralų grūdelių skaičiaus. Sunkiųjų mineralų, išskirtų iš gamtinių geocheminių anomalijų Lietuvos dirvožemiuose, kiekybinė mineraloginė analizė rodo, kad kai kurie mineralai (ksenotimas, chromitas, piritas, monacitas) buvo identifikuoti tik suskaičiavus daugiau nei 1500 mineralų grūdelių (1 lentelė). Taigi analizės patikimumas yra vienas iš kriterijų pasirenkant mineraloginį metodą.

Ikikvaterinių bei kvaterinių nuosėdų perklostymas, uolienų mechaninis smulkinimas ledynams keliaujant, poledyninis dūlėjimas, dirvodaros procesai nulėmė atskirų mineralų fizines savybes. Dėl jų pokyčių, taip pat mineralų cheminės sudėties įvairovės nepakanka plačiai taikomų mineraloginiuose tyrimuose šviesos mikroskopinių metodų (binokuliarinis ir poliarizacinis mikroskopai), siekiant tikslios mineralų identifikacijos kiekybiniam mineralų pasiskirstymui įvertinti. Be to, minėti metodai reikalauja ilgesnės analizės.

Tikslesniam mineralų nustatymui gali būti taikomi kiti mineraloginės analizės metodai, teikiantys informaciją tiek apie mineralų cheminę sudėtį, tiek ir apie jų struktūrinius-tekstūrinius ypatumus. Skenuojantis elektroninis mikroskopas (SEM) – vienas dažniausiai naudojamų pagalbinių instrumentų tiriant sunkiuosius mineralus, nes jis išsiskiria aukšta skiriamąja geba bei dideliu regėjimo lauko gyliu. Šiuolaikiniame SEM dažniausiai įrengtas dispersinis rentgeno spindulių spektrometras (EDS), palengvinantis elementinę analizę ir taip pagreitinantis mineralų identifikavimą pagal jų spektrus. Pastaruosiuose pikai atitinka mineralą sudarančius elementus, kurių koncentracija viršija 1%. Elementų pikai bei jų intensyvumas yra pagrindiniai mineralų atpažinimo rodikliai (1 pav. a, b, c). Kiekybiniam mineralų pasiskirstymui įvertinti su SEM ypač tinka poliruoti šlifai, nes juose galima pakartotinai analizuoti mineralus bei panaudoti šlifą analizei su kitais mikroskopais.

Tiriant geocheminių anomalijų mėginius iš Lietuvos dirvožemių, sunkiųjų mineralų (tankis > 3 g/cm³) mineraloginė analizė poliruotuose šlifuose buvo atliekama su SEM-EDS, analizuojant pirminių išsklaidytų elektronų (BE) atvaizdus. Kiekviename šlife buvo identifikuota bei suskaičiuota daugiau nei 1500 mineralų grūdelių. Vertinant mineralų pasiskirstymą šlife kaip homogenišką, BE atvaizdai buvo imami tolygiai paskirsčius juos per visą šlifą. Priklausomai nuo analizuojamos sunkiųjų mineralų frakcijos dydžio ir techninių parametrų, reikalingų aukštos kokybės atvaizdams, kiekviename atvaizde buvo identifikuota bei suskaičiuota nuo 10–15 iki 80–100 mineralų grūdelių (2 pav.).

Spalviniai BE atvaizdų kontrastai bei EDS analizės duomenys leidžia parinkti iš mineralų sąaugos labiau išplitusį mineralą arba skaičiuoti sąaugą kaip du atskirus mineralus (3 pav., a, b). Tokiu būdu buvo padidintas sunkiųjų mineralų grūdelių procentinio pasiskirstymo įvertinimo tikslumas. Analizuojant mineralus su SEM-EDS, sunkiųjų mineralų „skendimo efektas" šlifuose gali apsunkinti kai kurių sluoksniuotų bei stipriai išdūlėjusių mineralų identifikavimą. Taip pat neišvengiama mineralų grūdelių procentinio kiekio paklaida, susijusi su plačiomis granuliometrinių frakcijų ribomis. Tokį neigiamą efektą galima sumažinti susiaurinus granuliometrinių frakcijų ribas.

Ольга Варейкене, Марья Лехтонен

ТЯЖЕЛЫЕ МИНЕРАЛЫ В ИССЛЕДОВАНИЯХ ПОЧВ: МИНЕРАЛОГИЧЕСКИЕ МЕТОДЫ, ИХ ПРЕИМУЩЕСТВА И НЕДОСТАТКИ

Р е з ю м е

Химический анализ минеральных групп и отдельных минералов, выделенных из образцов почв, а также результаты их процентного распределения могут быть использованы для выяснения генезиса минералов, определения направлений переноса почвообразующих ледниковых отложений, площадного картографирования различных типов почв, идентификации отдельных слоев почвенного профиля, выяснения генезиса минералогических геохимических аномалий. Достоверность анализа распределения минералов зависит от числа определенных и сосчитанных минеральных зерен. Количественный минералогический анализ тяжелых минералов, выделенных из образцов, отобранных из геохимических аномалий в почвах Литвы, показал, что некоторые минералы (ксенотим, хромит, пирит, монацит) были определены только сосчитав больше чем 1500 минеральных зерен (табл. 1). Таким образом, требование достоверности количественного анализа является одним из критериев при выборе минералогического метода.

Переотложение дочетвертичных и четвертичных отложений, механическая дезинтеграция пород в процессе переноса ледником, постледниковое выветривание, почвообразующие процессы и разнообразие химического состава отдельных минералов предопределили, что применение широко используемых в минералогических исследованиях световых микроскопов для определения всех минералов при проведении количественного анализа их распределения является недостаточным и трудоемким.

Для более точного определения минералов могут быть применены другие минералогические методы, позволяющие получить информацию как о химическом составе минералов, так и о их структурно-текстурных особенностях.

Сканирующий электронный микроскоп (SEM) – наиболее часто применяемая техника в исследованиях тяжелых минералов, так как он характеризуется высокой разрешающей способностью и большой глубиной поля зрения. В современных моделях

к SEM обычно подсоединен рентгеноспектрометр (EDS), облегчающий элементный анализ и, таким образом, способствующий более быстрой идентификации минералов по их спектрам, в которых пики соответствуют составляющим минерал элементам с концентрацией выше 1%. Пики элементов и их интенсивность являются основными характеристиками, используемыми при определении минералов (рис. 1a, b, c).

Работая с SEM, полированные шлифы наиболее пригодны для количественной оценки распределения минералов, потому что возможно повторно анализировать не только тот же шлиф, образ, но и отдельные минералы, а также использовать шлиф для дополнительного минералогического анализа с другими микроскопами.

Для SEM–EDS минералогического анализа шлифов тяжелых минералов (плотность > 3 г/см³), выделенных из образцов, отобранных из геохимических аномалий в почвах Литвы, использовали образы минералов, созданные при регистрации первичных „отскачивших" электронов (BE). Оценивая распределение минералов как гомогенное, BE образы были отобраны по всему шлифу, следуя принципу равномерного их распределения по площади. В зависимости от размерности анализируемой гранулометрической фракции и от технических параметров, устанавливаемых для создания высококачественных образов, в каждом отдельном образе было проанализировано от 10–15 до 80–100 (рис. 2), а в каждом отдельном шлифе – >1500 зерен минералов.

Цветовые контрасты минералов в их образах и соответствующие данные EDS анализа позволяют из минерального сростка выбрать наиболее распространенный по площади минерал или считать сросток как 2 отдельных минерала (рис. 3a, b). Таким образом, точность анализа процентного распределения тяжелых минералов была повышена.

При минералогическом анализе с SEM–EDS „эффект погружения" тяжелых минералов в шлифе может вызвать трудности при определении слоистых и сильно выветрелых минералов. Кроме того, искажение результатов количественного распределения минералов, связанное с границами гранулометрических фракций, не может быть предотвращено при работе с SEM–EDS. Снижение такого отрицательного эффекта возможно при сужении размеров гранулометрических фракций.