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Results of heavy mineral pre-concentration by the Knelson for the geochemical study of soil: a case study in Lithuania

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Heavy minerals (HM) are important bearing phases of many trace elements in soils, glacial and water sediments. They can be used as indicators of different geochemical processes and as a basis to reveal the regional trends of the climate and ecosystem changes during the last glacial–interglacial cycle and the postglacial time. A geochemical study of the Lithuanian soil required representative concentrates of HM with $d > 3$ g/cm^3 in the middle sand fraction (0.25–0.5 mm) and fine sand fraction (0.1–0.25 mm). As voluminous soil samples were needed to be processed, a Knelson Concentrator was used.

The basic idea of the present study was to find out if a Knelson Concentrator could be used to pre-concentrate soil samples and how representative the HM fraction ($d > 3$ cm/g^3) would be in a size range of 0.1–0.5 mm.

The soil samples were taken from horizon A_1. Every initial sample was divided into two subsamples, which were processed differently. The first subsample of 60 kg was wet screened to a < 2 mm fraction and pre-concentrated by the Knelson (KPS – Knelson pre-concentrated sample). The second subsample of 10 kg was directly separated by wet sieving without pre-concentrating (DSS – directly separated sample). Both subsamples were subjected to heavy liquid separation, dry sieved and analyzed for mineral distribution using SEM-EDS. The results from the DSS showed a naturally occurring content and the proportions of HM in the initial sample, except for some trace HM in the middle sand fraction. They also showed that garnet – mostly almandine (d 4.1 g/cm^3) – is originally more abundant than amphibole – predominantly hornblende (d 3.2 g/cm^3) – in the fractions studied. However, a significantly higher garnet content and lower amphibole content were reported in the KPS when compared to the DSS. The heavier garnets were preferentially pre-concentrated over the lighter amphiboles. Not only the density, but also the size of the minerals was an important factor. Because a number of HM (e. g., zircon, xenotime, rutile and monazite) tend to occur as fine-grained crystals, a higher content of these minerals was recovered in the fine sand fraction than in the coarser fraction. As the grain size decreased, recovery shifted more than expected towards denser and more spherical HM types. There was a marked increase in the differences in recoveries between HM of the higher and lower density in the 0.1–0.25 mm fraction. The grain size distribution of the soil and especially the proportion of > 0.25 mm fraction and the HM content of the grain fractions were factors affecting the results of Knelson pre-concentration.

Key words: heavy mineral, Knelson Concentrator, recovery, wet sieving, heavy liquid separation, mineral distribution, soil

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INTRODUCTION

Heavy minerals (HM) are important carriers of many trace elements in soils, glacial and water sediments. They can be used as indicators of different geochemical processes and as a basis to acquire new knowledge on the regional trends of the climate and ecosystem changes during the last glacial–interglacial cycle and the postglacial time. This requires a representative collection of the mineral part of soil.

The mineralogical composition of the Lithuanian soil is characterised by uneven distributions of HM among different grain size fractions, as well as low HM content in fractions of 0.25–0.5 and 0.1–0.25 mm as compared to a fraction of 0.005–0.063 mm (Vareikienė, 2005). Some types of minerals are rare and can only be accounted for in sand fractions after an analysis of more than 1000 grains (Vareikienė and Lehtonen, 2004). It is therefore necessary to pre-concentrate the soil in order to get representative HM fractions in grain size classes of 0.1–0.5 mm. Pre-concentration is also required to produce an adequate volume of HM, in particular grain size classes for further mineralogical and chemical analyses.

When it comes to conventional gravity-based concentrators, the Knelson is considered to be one of the most advanced centrifugal separators available today. The Knelson Concentrator is widely used in the heavy mineral industry for the concentration of desired minerals from mixtures containing other particles. It was originally designed to extract on production level free gold and other precious metals from ores (Knelson Concentrators. Technical specification manuals, 1995; Knelson Concentrators. Technical papers and performance reports, 1995). In the present study, a GTK-modified 4.5" Knelson was used for the pre-concentration. GTK has modified this device so that it could recover from Quaternary samples minerals slightly heavier than the normal rock-forming ones (d 2.6–2.7 g/cm^3) (Chernet et al., 1999). A long-term regular monitoring of the recovery of kimberlite and diamond indicator minerals in the pre-concentration process line has shown an average of slightly less than 90% in the <0.25 mm fraction. For the unsorted sediment, such as till, the recoveries were somewhat higher than those for the sorted sediment (sand).

The main aim of this research was to find out if a Knelson Concentrator could be used to recover a representative heavy mineral fraction ($d > 3.0$ g/cm^3) from soil in the 0.1–0.5 mm grain size classes.

The purpose of the present paper is to evaluate the results of soil pre-concentration using a Knelson Concentrator (KPS) by comparing them with those of directly separated samples (DSS) obtained using wet-sieving fractionation. It will enable the assessment of possible errors and create a basis for the further development of the pre-concentrating procedure for the geochemical study of soils and surficial sediments.

MATERIALS AND METHODS

The studied soils are located in soil regions of Lithuania that differ in respect of the lithology and age of their soil-forming sediments: 1) the soils of the Ašmena Highlands have developed on a marginal formation of the glacial sediments from Saalian glaciation, 2) the soils of the Mūša–Nemunėlis Plain have developed on the basal till from Weichselian glaciation (Fig. 1). The soil of the Mūša–Nemunėlis Plain is underlain by the Upper Devonian dolomite, which lies quite close to the surface in some places. The age difference between the sediments of the last and the preceding glaciations is about 100 thousand years. It is observable in the higher degree of weathering and more homogenous composition of the older sediments.

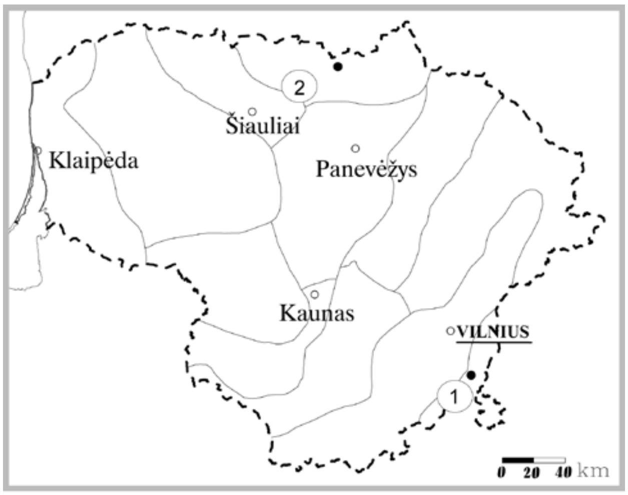


Fig. 1. Location of soil sampling points: *1* – Ašmena Highlands, *2* – Mūša–Nemunėlis Plain. ● – sampling point, / – border of soil region

1 pav. Dirvožemio ėminių paėmimo taškai: *1* – Ašmenos aukštuma, *2* – Mūšos-Nemunėlio lyguma, ● – mėginio paėmimo vieta, / – dirvožemio rajonų ribos

The initial source for heavy minerals in soil-forming sediments is the Fennoscandian Shield, which predominantly consists of Archean–Proterozoic crystalline rocks and younger recycled sedimentary rocks ranging in age from Cambrian to Paleogene (Lietuvos geologija, 1994). The original grain size of the heavies in the provenance area depends on the type of the mineral. For example, common minerals, such as garnet, hornblende, kyanite, epidote, etc., are prevalent in the >0.25 mm fraction, whereas trace minerals, such as chromite, zircon and rutile, which are all very hard and chemically resistant, are concentrated in the finer fractions. The amphiboles, garnets, pyroxenes and epidote were primarily derived from the crystalline rocks of the Fennoscandian Shield. The more resistant zircon, rutile, turmaline, staurolite, monazite, ilmenite and xenotime were secondarily derived from the PreQuaternary sediments (Юозапавичюс, 1974; 1976; 1987). The heavy mineral suite underwent several depositional cycles prior to deposition with other detritus as the Quaternary sediments in Lithuania. The elevated content of the heavies in the finer grain size classes can be logically attributed to a repeated and prolonged recycling resulting in the physical degradation of heavy mineral grains. The mineralogical characteristics of the most abundant heavy minerals in Lithuanian soils are given in Table 1.

SAMPLING AND METHODS

The samples for the present study were collected by a shovel from the upper part of agricultural soil (horizon A_1) on a representative sampling site of every soil region. They were composed of four sub-samples from a 5 m × 5 m square. The uppermost

Table 1. **Characteristic of heavy minerals (d > 3 g/cm^3) found in the soils of Lithuania**
1 lentelė. **Lietuvos dirvožemių sunkiųjų mineralų (d > 3 g/cm^3) apibūdinimas**

Mineral	Hardness	Grain morphology	Habit	Fracture
apatite	5	prismatic crystals	massive–granular	conchoidal
chromite	5.5	equidimentional spherical grains	granular, massive–granular, nuggets	uneven, flat surfaces (not cleavage) fractured in an uneven pattern
diopside	6	short prismatis crystals	blocky, granular, prismatic	brittle, conchoidal
epidote	7	short prismatic crystals shaped like slender prism	crystalline–prismatic, massive uniformly indistinguishable crystals forming large crystals	regular-flat surfaces (not cleavage) fractured in a regular patterns
garnet (mostly almandine)	4.2	equidimentional spherical grains	granular – generally occurs as auhedral to subhedral crystals massive–lamellar – distinctly foliated fine-grained forms	brittle, conchoidal, very brittle fracture producing small, conchoidal fragments
hematite	5	hexagonal and more complexly grained tabular crystals common pseudomorph after magnenite	blocky shape tends to be equant; tabular	conchoidal, fractures developed in brittle materials characterised by smoothly curving surface
hornblende	5	prismatic columnar crystals	columnar; massive–fibrous; massive–granular	sub conchoidal
ilmenite	5	hexagonal tabular and flatly plated crystals equal dimentional spherical grains	massive–lamellar – distinctly foliated fine-grained forms; tabular (form dimensions are thin in one direction)	conchoidal, fractures developed in brittle materials characterised by smoothly curving surface
kyanite	4.5	prolonged tabular, plated	columnar	brittle
monazite	5	thick tabular and isometric crystals	crystalline–fine, occurs as well-formed fine-sized crystals; twinning common	conchoidal
enstatite	5.5	orthorhombic dipyramidal crystals	lamellar, massive, fibrous	brittle
rutile	6	tetragonal dipyramidal and prismatic crystals	massive, granular, accicular	uneven, flat surfaces (not cleavage) fractured in an uneven pattern
staurolite	7	short prismatic crystals	tabular, twinning common	sub conchoidal
titanite	5.5	monoclinic prismatic crystals	crystalline–fine, massive–lamellar	sub conchoidal
tourmaline	7	prismatic crystals shaped like slender prism	acicular, prismatic, striated	brittle, conchoidal, very brittle fracture producing small, conchoidal fragments
xenotime	4.5	tetragonal ditetragonal dipyramidal crystals	massive, granular, prismatic	splintery – elongated fractures produced by intersecting good cleavages or partings
zircon	7.5	short or prolonged prismatic crystals with pyramidal terminations	crystalline–fine, prismatic, tabular	uneven, flat surfaces (not cleavage) fractured in an uneven pattern

organic-rich layer of the soil, roots, other coarse organic particles and boulders were removed before sampling.

Duplicate bulk sampling was conducted in order to ensure adequate material. Based on the experience of several years in the recovery of diamond indicator minerals (Chernet et al., 1999), an initial sample of 80 kg in size was found to be optimal for repeating the results obtained from one or two 0.25–0.50 mm mineral grains in the sample. The soil samples were taken from horizon A_1. Every initial sample was divided in two subsamples. The first subsample of 60 kg was wet screened to a < 2.0 mm fraction and then pre-concentrated using the GTK-modified, 4.5” Knelson Concentrator in order to recover a heavy mineral pre-concentrate (KPS – Knelson pre-concentrated sample). It was processed three times applying the GTK standard parameters for the kimberlite indicator separation. A pre-concentrate of 0.7–1 kg was produced for the further processing. The second subsample of 10 kg was directly separated by wet sieving without pre-concentrating (DSS – directly separated sample). The finer fraction was washed away from the sample and saved in a plastic pail, while the coarser part of the wet sieved sample (> 0.063 mm) was collected. The finer fraction of DSS was separated by sedimentation into fine silt and clay fractions (< 0.005 mm). The Knelson pre-concentrated sample (KPS) and the directly separated sample (DSS) were subjected to heavy liquid media (d > 3 g/cm^3) to liberate the final heavy mineral concentrates. Both heavy mineral concentrates were dry sieved to separate the middle sand (0.25–0.5 mm), fine sand (0.1–0.25 mm), coarse silt (0.063–0.1 mm) and fine silt (0.005–0.063 mm) fractions. Dry sieving was done using a multiscreen shaker with stainless steel sieves. For particle sizes of > 0.1 mm, the classification of detrital sediments in Lithuania (Gaigalas, 1995) was applied. Homogenization and quartering were carried out when splitting the samples for mineralogical analysis in order to minimize any possible errors from grain sorting due to varying density and size of particles.

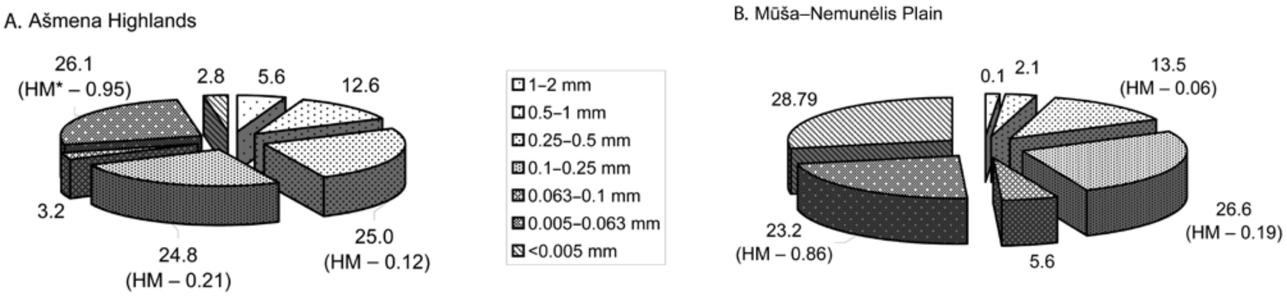


Fig. 2. Grain size analysis (%) of soil samples. HM* – the content of heavy minerals in a particular grain size fraction
2 pav. Dirvožemio mėginių granuliometrinė analizė (%). HM* – sunkiųjų mineralų kiekis granuliometrinėje frakcijoje

Heavy mineral identification and semi-quantitative mineral distribution analysis was carried out using a scanning electron microscope (SEM, JEOL-5600LV) attached to an energy dispersive spectrometer (EDS). Polished thin sections of heavy mineral concentrates – 0.25–0.5 mm, 0.1–0.25 mm and 0.005–0.063 mm fractions – were prepared on glass plates by mounting the mineral grains in Epoxy resin. Backscattered electron images (BSE) provided mineral spectra, and more than 1000 grains were studied from each polished thin section.

The recovery of heavy minerals yielded by the Knelson Concentrator was calculated as follows: recovery % = (mass of HM collected) / (mass of HM introduced) × 100%, where HM equals heavy minerals. Since there is no other practical way to determine the content (or mass) of HM in the soil sample introduced into the Knelson Concentrator, it can be considered to be equal to the HM content in the DSS. During the wet sieving there is no loss of HM and the recovery of HM is normally 100% for the DSS. With a Knelson Concentrator, it can be lower than 100% when loosing HM, or exceeding 100% when pre-concentrating HM.

RESULTS

The particle-size analysis of the Ašmena Highlands soil sample showed that it predominantly consisted of fine silt (26.1%) and fine sand (24.8%). Middle sand accounted for 12.6% and the clay fraction – for only a small percentage of soil (2.8%). The soil of the Mūša–Nemunėlis Plain is characterized by a large clay fraction (about 29%) and a low percentage of middle sand (5.6%), while fine sand accounts for more than 23% (Fig. 2).

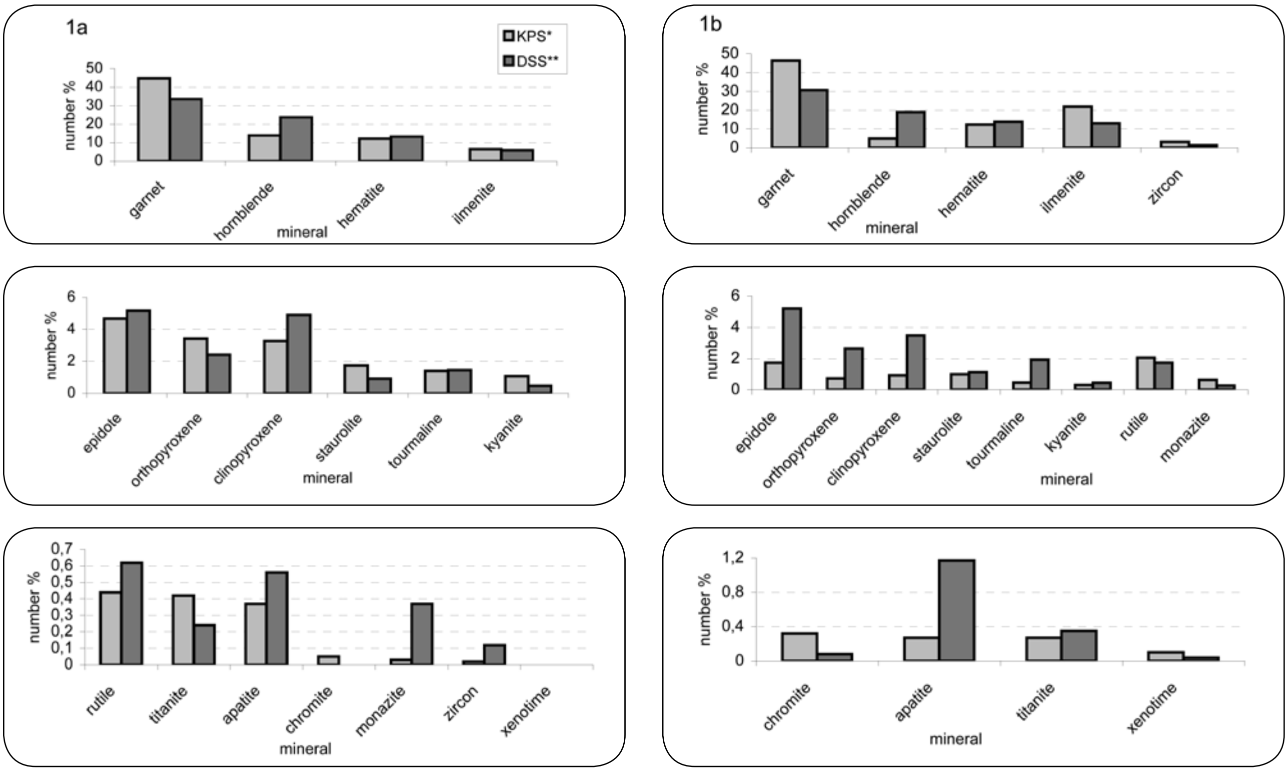
Only a very small percentage of both soils (about 1%) consists of heavy minerals, and their HM content is indirectly related to the size of the fraction. In the soils of the Ašmena Highlands and Mūša–Nemunėlis Plain, the highest content of heavy minerals is found in the fine silt fractions (0.95% and 0.86%, respectively) and the lowest – in the middle sand ones (0.12% and 0.06%, respectively) (Fig. 2).

The data from the semi-quantitative mineralogical analysis of the DSS from the Ašmena Highlands soil shows that garnet is slightly more abundant than amphibole in the middle and fine sand fractions. Ilmenite and zircon content is respectively two and ten times higher in the fine than in the middle sand. No notable variations in the distribution of hematite, epidote and orthopyroxene was evident between the fractions studied. Rutile, chromite, apatite, titanite, xenotime, staurolite and turmaline are more characteristic of fine sand. In the Mūša–Nemunėlis Plain, amphiboles and garnets are the predominant minerals in the middle sand fraction and respectively account for 32% and 30% of all the grains analysed. Clinopyroxenes, epidote and orthopyroxenes are less abundant (12%, 6% and 6%, respectively). Hematite and ilmenite are encountered in smaller amounts (4.9% and 3%, respectively). The rare minerals were represented by apatite and staurolite (2.6% and 1.3%, respectively), as well as trace amounts of titanite, tourmaline and monazite (> 0.1%) (Fig. 3).

Table 2. **Recovery* (%) of heavy minerals in particular grain size fractions of the soil pre-concentrated by the Knelson Concentrator**
2 lentelė. **Sunkiųjų mineralų atgavimas* (%) atskirose granuliometrinėse frakcijose, išskirtose iš Knelsono aparatu sodrinto dirvožemio**

Density (g/cm³)	**Mineral**	**Mūša–Nemunėlis Plain**	**Ašmena Highlands**	
		grain size fraction, mm		
		0.25–0.5	0.25–0.5	0.1–0.25
3.1	tourmaline	82.71	96.57	24.22
3.2	apatite	120.31	66.07	23.07
3.2	hornblende	54.33	58.36	25.91
3.3	clinopyroxene	48.65	66.93	26.64
3.4	epidote	92.14	90.52	33.39
3.5	titanite	82.71	175	77.14
3.6	kyanite	0	227.65	69.56
3.6	orthopyroxene	24.59	141.90	27.92
3.8	staurolite	134.41	192.30	88.59
4.1	garnet	182.68	133.78	151.43
4.25	rutile	51020.4 (NE*)	70.96	118.39
4.5	chromite	0	50	400
4.6	zircon	25510.2 (NE)	16.66	228.98
4.66	xenotime	0	0	250
4.8	ilmenite	161.19	109.76	168.61
5	monazite	82.71	8.10	232.14
5.3	hematite	40.68	90.88	88.80

* Recovery % = (content of the HM in the KPS) / (content of the HM in the DSS) × 100, where KPS – Knelson pre-concentrated sample, DSS – directly separated sample by wet sieving, HM – heavy minerals.
NE* – nugget effect in the DSS affecting a number of recovery of the HM in the KPS.
* Atgavimas % = (HM kiekis KPS)/(HM kiekis DSS) × 100; čia KPS – dirvožemio mėginys, sodrintas Knelsono aparatu, DSS – dirvožemio mėginys, suskirstytas šlapio sijojimo metodu, HM – sunkieji mineralai.
NE* – DSS nukrypimo paklaida, pasireiškusi vertinant HM sodrinimą KPS



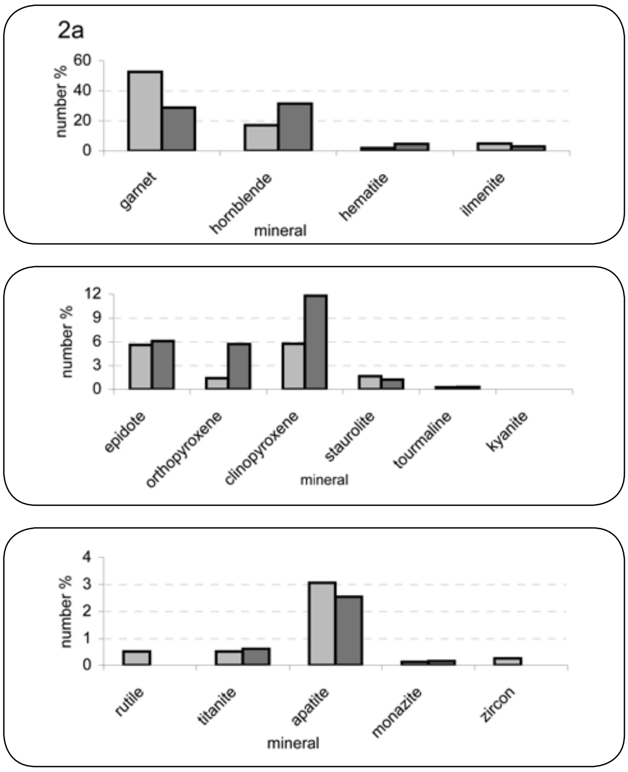


Fig. 3. Heavy mineral (d > 3 cm/g^3) distribution in the grain size fractions 0.5–0.25 mm (*a*), 0.25–0.1 mm (*b*) of the soil samples (horizon A_1) pre-concentrated by the Knelson Concentrator and directly separated by wet sieving (*1* – Ašmena Highlands, *2* – Mūša–Nemunėlis Plain)

3 pav. Sunkiųjų mineralų (d > 3 cm/g^3) pasiskirstymas dirvožemio (A_1 horizontas) granuliometrinėse frakcijose 0,5–0,25 mm (a), 0,25–0,1 mm (b), išskirtose iš Knelsono aparatu sodrintų ir tiesiogiai šlapio sijojimo metodu suskirstytų ėminių (*1* – Ašmenos aukštuma, *2* – Mūšos-Nemunėlio Lyguma)

* Content (%) of heavy minerals (d >3 g/cm^3) in the grain size fractions of the soil pre-concentrated by the Knelson Concentrator.

* Sunkiųjų mineralų (d > 3g/cm^3) kiekiai (%) granuliometrinėse frakcijose, išskirtose iš Knelsono aparatu sodrinto dirvožemio.

** Content (%) of heavy minerals (d > 3 g/cm^3) in the grain size fractions of the soil directly separated by wet sieving.

** Sunkiųjų mineralų (d > 3 g/cm^3) kiekiai (%) granuliometrinėse frakcijose, išskirtose iš šlapio sijojimo metodu suskirstyto dirvožemio.

A comparison of the heavy mineral distribution in the KPS and DSS from the Ašmena Highlands showed that in the 0.25–0.5 mm fraction, the Knelson-processed subsample of 60 kg yielded a garnet content (mostly almandine, d 4.1 g/cm^3), some 10% higher than in the DSS, and correspondingly about 10% lower amphibole content (mostly hornblende, d 3.2 g/cm^3) (Fig. 3). Lower contents of clinopyroxene, epidote, tourmaline, hematite and apatite as well as higher contents of staurolite, kyanite, titanite and chromite were encountered in the KPS. The content of some heavy minerals with a density of > 4 g/cm^3, e. g., rutile, monazite, zircon, was found to be higher in the DSS (Fig. 3). In the KPS, the recoveries of medium dense minerals (d > 4.2 g/cm^3), such as rutile, chromite, zircon, monazite, of 0.25–0.5 mm in size are considerably lower than the recoveries of less dense minerals (d 3–3.4 g/cm^3). The Knelson pre-concentration of heavy minerals with a density of 3.5–4.1 g/cm^3 resulted in the recovery exceeding 100% within the 0.25–0.5 mm fraction (Table 2).

A comparison of the HM distribution in the KPS and the DSS in fine sand showed that the pre-concentration of soil using the Knelson Concentrator resulted in the same proportion of the main heavy minerals as in the middle sand: a higher garnet content and a lower hornblende content in the KPS (Fig. 2). Only the loss of amphibole was much higher than in the middle sand. In fact, the recovery of hornblende in the KPS was only 25% of that recovered from the 0.1–0.25 mm fraction of the DSS, while in the middle sand it was about 60% (Table 2). In the 0.1–0.25 mm fraction, the differences in heavy mineral distribution grew, especially for heavy minerals with a density of > 4.0 g/cm^3 (Fig. 2). The recoveries of these minerals (rutile, chromite, zircon, xenotime, monazite) in KPS exceeded 100% in the range from 120 to 400%.

The recovery of minerals with densities closer to 3 g/cm^3 (in the range of 3–3.4) was less than 40% (Table 2). Therefore, the fine sand fraction of the KPS is characterised by the highest recoveries of heavy minerals with densities above 3.8 g/cm^3 and the lowest recoveries for the group of minerals within density ranges from 3 to 3.8 g/cm^3 (Table 2).

In the soil of the Mūša–Nemunėlis Plain, a higher garnet content and lower hornblende content were found in the 0.25–0.5 mm fraction of the KPS as compared to the DSS. Smaller differences in mineral recovery between the KPS and DSS can be observed in hematite, epidote, titanite, ortho- and clinopyroxenes content. These minerals have been washed away during the Knelson processing, while medium dense minerals, such as ilmenite, rutile, monazite and zircon, were effectively pre-concentrated. As compared to the Ašmena Highlands, the Mūša–Nemunėlis Plain's KPS had a higher garnet content and lower hornblende content. A higher content of medium dense minerals (ilmenite, rutile, monazite and zircon) was also found in the middle sand fraction (Fig. 3). In this fraction, the recovery of the main heavy minerals followed almost the same trend as in the Ašmena Highlands. For minor and trace minerals, the recovery shifted to denser minerals.

DISCUSSION

In the present study, a GTK-modified 4.5" Knelson Concentrator was used to recover quantitative pre-concentrates of heavy minerals (d > 3.0 g/cm^3) from soil samples within grain sizes of 0.25–0.5 and 0.1–0.25 mm. In principle, the Knelson Concentrator, including the GTK-modified one, best recovers coarser heavy mineral particles. Another feature of the device is that it tends to reject heavy minerals of lower density at the expense of minerals of higher density, which are recovered first.

The results of the test performed on 10 kg of the original sample (< 2 mm) applying a direct separation by wet sieving, heavy media and analysis using SEM-EDS can be considered a basis for the evaluation of the efficiency of heavy mineral processing using the Knelson Concentrator. Although these results are not considered to be completely representative – due to the small size of the sample tested and the rarity of some heavy minerals in the Lithuanian soils – they do show the contents and proportions of heavy minerals as they naturally occur in the soil. The significance of the skewed proportions of heavy mineral types arising from the Knelson pre-concentration was assessed by comparing the recoveries of these mineral types to those obtained from direct separation of the soil sample by wet sieving.

The first comparison revealed that the recoveries of some light heavy minerals were not even close to being complete. After the analysis of heavy mineral distribution in the 0.25–0.5 mm and 0.1–0.25 mm fractions of the KPS, significantly higher garnet content and lower amphibole content were reported as compared to the heavy mineral fractions of the DSS.

The mineral distribution results from the DSS showed that garnets, mostly represented by almandine (d 4.1 g/cm^3), are originally more abundant in the soil than amphiboles, mostly represented by hornblende (d 3.2 g/cm^3) (Fig. 3). A comparison of heavy mineral distribution in the KPS and DSS from the Ašmena Highlands soil showed that in the 0.25–0.5 mm and 0.1–0.25 mm fractions of the KPS, heavier garnets were preferentially pre-concentrated over amphiboles. Density differences are therefore the main factor controlling the recovery of these minerals. However, not only the density, but also the size, and to a lesser extent, the shape also influence the recovery of heavy minerals. Even if it is not possible to evaluate the effect of the shape directly, it is noticeable in the higher content of equidimensional minerals (garnet and epidote) and the lower content of elongated minerals (hornblende and tourmaline) in the KPS. The size factor is important when it comes to the recovery of some heavy minerals, e. g., zircon, xenotime, rutile, monazite. Since these minerals tend to occur as fine-grained crystals, their content in the 0.1–0.25 mm fraction was higher than in the coarser fraction (Table 1, Fig. 3). These minerals tend to be sized closer to the lower limit of the coarser sand. In spite of their high density, they can be pushed out of the centrifugal bowl by less dense but bigger grains during the pre-concentration process. This resulted in a lower recovery of these minerals in the coarser fractions of the Ašmena Highlands soil (Table 2). The wide range of grain size classes present in the sample, therefore, leads to a preferential concentration of bigger and denser minerals, and finer-grained heavy minerals can be lost during the processing.

The recovery of dense heavy minerals in the middle sand of the Mūša–Nemunėlis Plain was more effective than in the same fraction of the soil from the Ašmena Highlands (Fig. 2, Table 2). The results of the recent studies, especially those investigating the mechanisms of heavy mineral pre-concentration by the Knelson Concentrator, showed that the recovery of heavy minerals is affected by the grain size distribution of the original sample. One of the factors is the proportion of the fines (< 0.063 mm). The presence of an adequate proportion of the fines promotes the formation of a suspension, which is the feed for the pre-concentrator. A comparison test performed with till and esker sand samples from Finland showed that the recoveries of diamond indicator minerals from the till samples were close to 100%, but lower when processing the esker sample (Chernet et al., 1999). In the present study, the proportion of the fines in both original soil samples was about the same as in the till samples, and hence they were optimal for processing and keeping heavy minerals in slurry (Fig. 2). The recovery of heavy minerals was therefore more dependent on the content level of the coarsest fractions (0.25–2 mm). The recovery of fine and denser heavy minerals from the Mūša–Nemunėlis Plain soil was higher than that from the soil of the Ašmena Highlands, even in spite of the lower total heavy mineral content in all the samples and the middle sand (Table 2). The differences can be attributed to the fact that the Ašmena

Highlands soil had more than a double amount (35%) of the > 0.25 mm fraction as compared to the Mūša–Nemunėlis Plain soil (about 15%). The GTK standard parameters used in the work have been set to maximize a concentrate of > 0.25 mm grains. Due to the small proportion of that grain size range in the clay-rich soil of the Mūša–Nemunėlis Plain, the recovery rates of fine and denser heavy minerals are high and also extend better towards the < 0.25 mm fraction.

The results of the mineralogical study conducted on the DSS from the Mūša–Nemunėlis Plain showed a naturally occurring content and proportions of heavy minerals in the sample, excluding the heavy minerals present in trace quantities, e. g., zircon, rutile in coarser fractions. The nugget effect in the DSS caused exceptionally high recoveries of these minerals in the KPS (Table 2).

The tests revealed that in the KPS from the Ašmena Highlands soil, slightly heavy minerals (d 3–3.4 g/cm^3) coarser than 0.25 mm were recovered more quantitatively than the same minerals in the fine sand fraction (0.1–0.25 mm). In the fine sand fraction, the recovery of denser heavy minerals was more efficient. In the 0.1–0.25 mm fraction, the differences in recoveries between the heavy minerals of the higher and the lower density grew markedly (Table 2). Therefore, as the grain size decreased, the recoveries in the soil samples pre-concentrated by the Knelson Concentrator shifted towards denser and more spherical types of heavy minerals.

CONCLUSIONS

A geochemical study of the Lithuanian soil and glacial sediments requires a representative sample of heavy minerals with d > 3 g/cm^3 in sand fractions (0.25–0.5 and 0.1–0.25 mm), where the content of heavy mineral fraction is low and some trace heavy minerals are rare. As voluminous soil samples were needed to process, a Knelson Concentrator was used.

The evaluation of mineral recovery in the Knelson pre-concentrated sample showed that heavier garnets were preferentially pre-concentrated over lighter amphiboles. Not only the density but also the size of grains is an important factor. The tendency for minerals to occur in particular grain classes that usually reflect their initial size in parent rocks has an effect on the results of the pre-concentration. Because a number of heavy minerals (e. g., zircon, xenotime, rutile and monazite) tend to occur as fine-grained crystals, a higher content of these minerals (d > 4 g/cm^3) was recovered in the fine sand fraction than in the middle sand. Therefore, the use of a wide grain size range leads to the preferential concentration of bigger and heavier minerals, and fine-grained heavy minerals may be lost during the processing.

As the grain size decreased, the recoveries in soils shifted towards denser and more spherical types of heavy minerals. In the 0.1–0.25 mm soil fraction, the differences in the recoveries between heavy minerals of higher density and heavy minerals of lower density grew markedly. The grain size distribution in the original soil sample, especially the content of grains coarser than 0.5 mm and the content of heavy minerals in the grain fractions also affected the efficiency of the Knelson pre-concentration.

The main conclusions from this study can be listed as follows:

1. The processing of a small sample (10 kg) by a direct separation using wet sieving followed by a heavy media separation seems to give good yields for major and minor minerals in all the grain size classes studied and for trace minerals in the fine sand fraction. The exception is the result of some trace minerals, e. g., zircon and rutile, in the middle sand fraction.
2. By processing a bigger sample by wet sieving, the nugget effect associated with a small sample size and the rarity of some heavy (d > 4 cm/g^3) minerals in the middle sand fraction can be minimized.
3. Using a bigger sample for wet sieving (> 10 kg) could give a higher accuracy for determining the actual content of trace HM in the sand fractions of the soil.
4. A direct separation of a small sample using wet sieving would normally liberate representative heavy mineral concentrates below the 0.1 mm grain size range.
5. For both grain sizes, 0.25–0.5 and 0.1–0.25, heavy minerals in the Knelson pre-concentrated sample were not recovered quantitatively. Therefore, semi-quantitative recalculations linking the chemical parameters and mineralogy of the soil with the direct identification and quantifying of element-bearing phases turned out to be unreliable.
6. When using the Knelson Concentrator, trace heavy minerals should also be studied from the coarser fractions, even if they naturally occur in finer grain size classes.
7. For grain sizes between 0.1 and 0.25 mm, repeating the pre-concentration of the rejected fraction would probably result in a higher recovery. However, the expected improvement in the recoveries would again shift towards higher density and coarser grains.
8. The pre-concentration process requires further research and testing in order to adapt the Knelson Concentrator more quantitatively for the geochemical study of soil and glacial sediments.

Given that the density differences of the mineral suite under separation are small, the grain size variation in the feed should therefore be minimized in order to obtain better recovery. To improve recovery, a narrower grain size fraction (< 1 mm) should be used for the Knelson pre-concentration instead of the < 2 mm size. Focusing on a certain grain size range, e. g., 0.1–0.25 mm, would be beneficial before subjecting the sample to the Knelson processing to wash it to the upper limit of the fraction. This would, perhaps, result in an improved recovery of heavy minerals.

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SUNKIŲJŲ MINERALŲ SODRINIMAS KNELSONO APARATU ATLIEKANT GEOCHEMINIUS DIRVOŽEMIO TYRIMUS (LIETUVA)

Santrauka

Dirvožemiuose, ledyninėse nuogulose, ežerų ir upių nuosėdose akcesoriniai sunkieji mineralai (HM) yra svarbūs daugelio cheminių elementų nešėjai. HM bei su jais susijusių mikroelementų kiekiai gali būti panaudoti geocheminės aplinkos tyrimams siekiant įvertinti paskutiniojo tarpledynmečio–ledynmečio ciklo ir poledynmečio klimato bei ekosistemų regioninės kaitos dėsningumus.

Ledyninėse nuogulose ir ant jų susiformavusiuose dirvožemiuose (< 2 mm) dažniausiai randami maži HM kiekiai (< 1%). Mineralinė Lietuvos dirvožemių sudėtis charakterizuojama nevienodu HM pasiskirstymu tarp atskirų granuliometrinių frakcijų, taip pat mažais jų kiekiais 0,25–0,5 ir 0,1–0,25 mm frakcijose, lyginant su 0,005–0,063 mm frakcija. Atskiri mineralai (pvz., ksenotimas, chromitas, monacitas) identifikuojami tik suskaičiavus daugiau nei 1000 mineralų grūdelių, todėl, vykdant geocheminius dirvožemio bei kitų nuogulų tyrimus, o ypač siekiant susieti jų cheminę ir mineralinę sudėtis, juos reikia sodrinti. Knelsono aparato taikymą Lietuvos dirvožemių sodrinimui geocheminiuose tyrimuose sąlygojo būtinumas išgauti, naudojant didelio tūrio pirminius dirvožemio ėminius, reprezentatyvų HM (d > 3 g/cm^3) mėginį vidutinio (0,25–0,5 mm) ir smulkaus (0,1–0,25 mm) smėlio frakcijose bei pakankamą jų kiekį cheminei ir mineraloginei analizėms.

Knelsono aparato efektyvumui įvertinti dirvožemio ėminiai paimti iš A_1 horizonto. Kiekvienas pradinis dirvožemio ėminys suskirstytas į du atskirus ėminius, kurie buvo paruošiami skirtingais būdais. Pirmas ėminys (apie 10 kg) tiesiogiai suskirstytas šlapio sijojimo metodu (DSS), išplaunant < 0,063 mm frakciją, antras ėminys (apie 60 kg) išplautas iš < 2 mm frakcijos ir toliau sodrintas Knelsono aparatu (KPS). Naudojant sunkiuosius skysčius šlapiai sijotame, taip pat Knelsono aparatu sodrintame ėminyje, išskirta HM frakcija, kuri sauso sijojimo metodu padalyta į atskiras granuliometrines frakcijas. Smėlio frakcijose (0,25–0,5 mm ir 0,1–0,25 mm) nuskaitančiu mikroskopu ištirtas HM procentinis pasiskirstymas.

Šlapio sijojimo metodu suskirstytų ėminių mineralogine analize nustatyti gamtiniai HM kiekiai ir jų santykiai, išskyrus tų HM, kurių kiekiai vidutinio smėlio frakcijoje labai maži, pvz., cirkono, rutilo. Su minėtų mineralų mažu kiekiu susijęs „nukrypimo efektas“ lėmė ypač aukštą šių HM išgavimą Mūšos-Nemunėlio lygumos KPS ėminyje (>100%). Mineraloginė analizė atskleidė, kad DSS ėminyje granatai (daugiausia almandinas, d–4,1 g/cm^3) plačiau paplitę nei amfibolai (daugiausia raginukė, d–3,2 g/cm^3), bet išanalizavus KPS, pastebėtas kur kas didesnis granatų ir mažesnis amfibolų kiekis nei DSS. Sunkesnių granatų sodrinimo efektyvumas pastebimai didesnis nei lengvesnių amfibolų. Palyginus mineralų paplitimą vidutinio ir smulkaus smėlio frakcijose, išskirtose iš KPS ir DSS, matyti, kad:(1) sodrinto dirvožemio HM 0,25–0,50 mm frakcijoje nustatytas 10% didesnis granatų ir 10% mažesnis amfibolų išgavimas nei iš šlapio sijojimo metodu paruošto dirvožemio; (2) dar didesnis skirtumas tarp sunkesnių ir lengvesnių HM išgavimo pastebėtas smulkaus smėlio 0,25–0,1 mm HM frakcijoje. Amfibolų išgavimas KPS sudarė tik apie 25% mineralo kiekio DSS.

Ne tik tankis, bet ir mineralų dydis yra svarbus sodrinimo veiksnys. Mineralų tendencija kauptis tam tikroje granuliometrinėje frakcijoje, atspindinti jų pirminį dydį uolienose, turi įtakos sodrinimo rezultatams. Kadangi tam tikri mineralai (pvz., cirkonas, ksenotimas, rutilas, monocitas) dažniausiai yra smulkiagrūdžiai, didesnis jų kiekis išgautas smulkaus smėlio frakcijoje, lyginant su stambesniąja. Taigi plačių ribų granuliometrinių frakcijų naudojimas lemia išskirtinę didesnių ir sunkesnių mineralų koncentraciją, o smulkiagrūdžiai mineralai gali būti prarasti sodrinimo metu.

Tyrimas atskleidė, kad:

- Šlapio sijojimo metodu paruoštoje sunkiųjų mineralų frakcijoje aptinkami reprezentatyvūs pagrindinių ir mažųjų HM kiekiai visose smėlio granuliometrinėse frakcijose ir akcesorinių HM kiekiai smulkaus smėlio frakcijoje. Išimtis – atskiri HM, kurių akcesoriniai kiekiai randami vidutinio smėlio frakcijoje.
- Šlapio sijojimo metodu paruošiant didesnį dirvožemio ėminį, „paklaidos efektas“, susijęs su mažu pradinio dirvožemio ėminio dydžiu ir atskirų HM labai mažais kiekiais vidutinio smėlio frakcijoje, gali būti sumažintas.
- Iš mažo dirvožemio ėminio (10 kg) šlapio sijojimo metodu paruošta HM frakcija reprezentatyviai atstovauja HM < 0,1 mm frakcijose.
- Naudojant Knelsono aparatą dirvožemio sodrinimui, sunkiųjų mineralų išgavimo procentas paslenkamas sunkesnių ir apvalesnę formą turinčių sunkiųjų mineralų naudai. Tai ypač aiškiai pasireiškia mažėjant frakcijos dydžiui.
- Vidutinio ir smulkaus smėlio frakcijose HM išgavimas KPS nebuvo kiekybinis, todėl jų procentiniai kiekiai nėra tinkami skaičiavimams, susiejantiems dirvožemio cheminę ir mineralinę sudėtį.

– Siekiant pagerinti dirvožemio HM išgavimo rezultatus, reikalingi papildomi Knelsono aparato pritaikymo sodrinimo procesui tyrimai.

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ОБОГАЩЕНИЕ МИНЕРАЛЬНОЙ ЧАСТИ ПОЧВ В ЛИТВЕ ПОСРЕДСТВОМ КНЕЛЬСОН-ОБОГАТИТЕЛЯ ДЛЯ ГЕОХИМИЧЕСКИХ ИССЛЕДОВАНИЙ

Резюме

В ледниковых отложениях и в сформировавшихся на них почвах (<2 мм), в озерных и речных донных отложениях акцессорные тяжёлые минералы (НМ) являются основными носителями многих микроэлементов. Поэтому распределение НМ и с ними связанных микроэлементов может быть использовано при геохимических исследованиях окружающей среды, а также для оценки и выявления закономерностей регионального изменения экосистемы и климата в цикле последнего межледникового-ледникового и послеледникового периодов.

В почвах (< 2 мм) акцессорные тяжёлые минералы присутствуют в малых количествах (< 1%). Минеральный состав почв Литвы также характеризуется неодинаковым распределением НМ между гранулометрическими фракциями и небольшим количеством НМ в средне- и мелкопесчаной фракциях (0,5–0,25 и 0,25–0,1 мм) по сравнению с мелкоалевритовой фракцией (0,063–0,005 мм). Отдельные минералы (ксенотим, хромит, монацит) могут быть определены только в результате исследования больше 1000 зёрен минералов. Поэтому при геохимическом исследовании почв и в особенности при оценке химического и минерального состава почвы и выявлении закономерностей их изменения необходимо провести обогащение минеральной части почвы.

Применение Кнельсон-обогатителя при геохимическом исследовании почв Литвы предопределено необходимостью получить используя для процесса обогащения образцы почв большого объёма, репрезентативный образец НМ (d > 3 г/см^3) в средне- и мелкопесчаной фракциях и достаточное количество НМ для химического и минералогического анализов. Для оценки эффективности обогащения минеральной части почв с помощью Кнельсон-обогатителя образцы почв были приготовлены двумя различными способами. Первый образец (DSS) (около 10 кг) был разделён на фракции посредством мокрого просеивания, вымыванием фракции < 0,063 мм, другой (KPS) (около 60 кг) – был вымыт до фракции < 2 мм и далее использован для обогащения посредством Кнельсон-обогатителя. Используя тяжёлую жидкость, из обоих образцов выделены фракции НМ, которые методом сухого просеивания подразделены на отдельные гранулометрические фракции. В средне- и мелкопесчаной фракциях сканирующим микроскопом определено процентное содержание НМ.

Процентное соотношение НМ в DSS соответствует их природному количеству и соотношению, за исключением тех НМ, которые содержатся в среднепесчаной фракции в следовых количествах. Вследствие „эффекта отклонения“, обусловленного следовыми количествами некоторых НМ, процент их обогащения в KPS из почв равнины Муша–Нямунелис намного превысил 100%. Минералогический анализ показал, что в DSS гранаты (в основном альмандин d-4,1 г/см^3) более распространены, чем амфиболы (более всего роговая обманка d-3,2 г/см^3). Сравнительный анализ распределения минералов в KPS и DSS показал, что: 1) во фракции почвы 0,5–0,25 мм, обогащенной посредством Кнельсона, обогащение гранатов на 10% выше и амфиболов на 10% ниже, чем в DSS; 2) более высокая разница в обогащении более тяжелых и более лёгких НМ обнаружена в мелкопесчаной фракции. Обогащение амфиболов в KPS составило только 25% количества минерала в DSS. Не только плотность, но и размер минералов является важным фактором в процессе обогащения. Тенденция минералов концентрироваться в определенной гранулометрической фракции в зависимости от их первичного размера в коренных породах оказывает влияние на результаты обогащения.

Исследование показало, что:

– фракция НМ, приготовленная методом влажного просеивания, хорошо представляет главные и малые НМ во всех песчаных гранулометрических фракциях и следовые НМ – в мелкопесчаной фракции. Исключение составляют некоторые НМ, присутствующие в среднепесчаной фракции в следовых количествах;

– „эффект отклонения“ в DSS, обусловленный малым объёмом первичного образца почвы и следовыми количествами некоторых НМ в среднепесчаной фракции, может быть ниже при использовании для влажного просеивания больших объёмов почвы (>10 кг);

– при использовании для просеивания 10 кг образца почвы НМ фракции <0,1 мм представлены в репрезентативном количестве;

– при использовании Кнельсон-обогатителя для обогащения почвы процент обогащения НМ сдвинут в пользу более тяжелых и имеющих округлую форму НМ. Эта тенденция особенно очевидна при уменьшении размера фракции;

– обогащение НМ в средне- и мелкопесчаной фракциях KPS почв не было полностью качественным и поэтому процентные количества НМ не могут быть использованы для расчётов, связывающих химический и минеральный составы почвы;

– необходимы дополнительные исследования для установки параметров Кнельсона в целях более эффективного обогащения минеральной части почвы.