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Determining the possibility of using the Polish aggregates for recovery of rare earth elements

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Abstract:

The high and constantly growing interest in Rare Earth Elements (REE) results from their desirable properties that are required in the state-of-the-art technologies. These elements are widely distributed in nature but most often found in low concentrations. Poland's resources are limited to a few poor deposits. KOMAG has started research projects aimed at extending the knowledge about the content of REE in Polish raw materials. Five Polish natural aggregates were selected for testing. The method of mass spectrometry with inductively coupled plasma ionization (ICP-MS) were used for analyses. Content of REE in the tested materials was found to be in the range of 190.0-14.8 ppm. Basalt aggregate marked with number 1 and halloysite had the highest content, and the lowest content was in the granite aggregate and sand marked with number 2. Then, the grain class 0.045-0 mm was separated to determine REE content in fine grains of materials. The test results indicate different proportions between light and heavy REE in the samples of the tested materials.

Keywords: rare earth elements (REE), screening, natural aggregates



1. Introduction

Rare Earth Elements (REE) is a group of elements which includes lanthanide and group 3 elements. These elements are of great interest which are constantly growing. This interest is related to a number of desirable properties of these elements. These properties are used in many state-of-the-art technologies. There is a division of these elements into light rare earth elements - LREE (La-Eu) and heavy rare earth elements - HREE (Gd – Lu and Y) [1]. There is also a different, more precise division in the literature, more often used in extraction: light rare earth elements - LREE (La – Pm), medium rare earth elements - MREE (Sm – Gd), heavy rare earth elements - HREE (Tb – Sc and Y) [2].

Rare earth elements are the elements widespread in nature. However, their low concentration cause the problem with their recovery. Deposits of high concentration of these elements are very rare. Currently, Poland does not have its own identified raw minerals resources as an economic source of REE. Their presence in the form of lean deposits is limited to the area of Szklarska Poręba, the Sudetes, the vicinity of Białystok and the sands of the Baltic Sea beaches. Heaps of waste from the phosphogypsum industry and ashes from power plants from coal combustion and co-incineration are considered to be a potential source of rare earths elements. These wastes are an important source of REE recovery. Therefore, the KOMAG Institute of Mining Technology has started searching for the sources of rare earth elements in Poland.

Due to the confirmed occurrence of rare earth elements in natural aggregates [3,4,5], an attempt was made to analyse the Polish aggregates. Earlier studies of the KOMAG Institute of Mining Technology confirmed the presence of rare earth elements in the materials of Polish origin but in small concentrations [6,7,8]. Natural aggregate is a material consisting of grains, derived from crumb deposits - gravel and sand aggregates, or solid rock deposits - crushed aggregate. The remaining aggregates available on the market are artificial and recycled aggregates [9,10]. The main applications of natural aggregates are the production of concrete, asphalts, mortars, adhesives, prefabricated products and others. Refined aggregates are used in the glass industry, foundries, for filtration of water, sewage and others [11]. Currently exploited mineral aggregate deposits may be prospective places for recovery of rare earth elements in Poland. The existing technological lines can be used as a stage of preliminary classification of raw material from the deposit. Even a low content of minerals containing rare earth elements creates the possibility of an economically justified investment in machines and equipment for separation. Currently operating mining plants around the world use deposits for recovery of rare earth elements when their concentration is over 500 ppm [12].

This article is aimed at confirming the presence of rare earth elements in the selected natural aggregates.

In nature, there are no deposits of rare earth elements in their pure form. These elements are most often in the form of minerals or compounds formed after ionization of minerals. Today, science knows more than 250 minerals containing rare earth elements. Due to their small accumulation, few of them are suitable for recovery. Currently, deposits containing mainly the following minerals are exploited for REE recovery: bastnasite, monazite, xenotime and sorption-clay minerals [13,3]. These minerals are characterized later in the publication.

Bastnasite belongs to the group of fluorocarbonate minerals. The chemical formula is - $\text{CeCO}_3(\text{OH}, \text{F})$. Cerium can be replaced by other light rare earth elements (LREE), e.g. lanthanum. This mineral can be found in igneous rocks such as pegmatites and granites. It also occurs in metamorphic rocks, in zones where contact metamorphism plays a dominant role [14]. Other minerals containing rare earth elements such as allanite or fluocerite may coexist with this mineral [15].

Monazite belongs to the group of phosphate minerals. The chemical formula is - $(\text{Ce}, \text{La}, \text{Nd}, \text{Th})(\text{PO}_4)$. Cerium, lanthanum, neodymium and thorium are interchangeable in the mineral structure. This mineral is most often an accessory mineral. It is most often found in igneous or metamorphic rocks, e.g. granite, pegmatite, basalt, slate and gneiss [16,17,18,19]. Monazite can also be found as a component of sedimentary rocks. This mineral is one of the more resistant to the atmosphere impact. As a result of weathering of the parent rocks containing monazite, the mineral may accumulate in the material left over from this process. The crumb deposit created in this way with appropriate amount can be used to obtain rare earth elements and thorium [20].

Sorption-clay minerals are the minerals of a very small size. Due to the small grain size of these minerals and the large specific surface area, they are able to adsorb large amounts of ions of chemical compounds. Sorption-clay minerals containing rare earth elements form during rock weathering. Then the ionization of the compounds containing these elements takes place and then they adsorb on the surfaces of sorption-clay minerals. The main mother rocks of sorption-clay minerals are igneous rocks - granites [4]. These minerals are one of the main sources of heavy rare earth recovery. Due to the weaker (sorption) binding of rare earth ions, obtaining a concentrate of these elements is much easier [21]. Deposits of sorption-clay minerals, mainly located in China, consist of clay (40-70%). The clays in these deposits are halloysite (25-50%), illite (5-20%), kaolinite (5-10%) and montmorillonite (<1%) [22]. Halloysite belongs to the aluminosilicates of the kaolinite subgroup. It occurs very rarely in pure, homogeneous form, most often it is a mineral accompanying kaolinite, but also iron-bearing minerals, feldspars and quartz sands.

Xenotime belongs to the group of phosphate minerals with the formula YPO_4 . One of the main features of this mineral is the content of yttrium above 50% and the low content of light rare earth elements. Compared to monazite, it has a much lower thorium content [23]. This mineral is most often an accessory mineral in igneous rocks. Xenotime may also be found in clastic deposits, the genesis of which is similar to that of monazite-containing clastic deposits [24]. It has been shown that xenotime can also contain tungsten and radioactive elements such as uranium and thorium [25].

Xenotime, monazite and bastnasite are minerals that can be found in rocks such as granite, basalt, pegmatite and others mentioned above. The materials that were used for tests are characterized below.

Granite is a deep igneous rock of an open-crystalline structure. This rock is formed from the slow crystallization of magma beneath the Earth's surface. The main minerals of granite are orthoclase, plagioclase, quartz and biotite. The content of rare earth elements in this rock depends on the content of accessory minerals. Some of the granite accessory minerals contain REE. They are, among others: monazite, xenotime, allanite, titanite and anatase [26,27].

Basalt is an alkaline, pourable (volcanic) solid rock with a very fine-grained (cryptocrystalline) or aphanitic structure, sometimes porphyry. The main minerals of this rock are pyroxene, plagioclase, mica, amphibole. Many studies have demonstrated the presence of rare earth elements in basalt rocks [5,28,29]. The presence of rare earth elements also in these rocks is related to the content of accessory minerals [30].

Conical separators, coiled stream separators, flotation machines, magnetic separators, electrostatic separators or Multi-Gravity separators are used in the mechanical processing of raw materials containing rare earth elements. The mentioned machines and devices can be divided according to their separation efficiency. Devices of relatively low separation efficiency are usually used at the beginning of the technological line for the production of pre-concentrates. Devices of a relatively low separation efficiency of upgrading include conical separators and coiled stream separators. The pre-concentrates are then directed to the devices of higher separation efficiency as a feed. Devices of a relatively high separation efficiency, such as magnetic separators or electrostatic separators, are usually used for the production of high purity final concentrates [31]. Classification of the material according to its grain size could be another way to increase the concentration of rare earth elements. The effectiveness of this method will be greater if the rare earth elements are concentrated in the grains of a certain size. According to the literature information, greater concentration is expected in grains of smaller size [32].

Identification of the material that, when subjected to processing, will allow to obtain a concentrate containing about 60% of rare earth minerals, what, according to the literature, is a condition for the material usefulness in production of high purity rare earth elements is the main objective of tests carried out at the KOMAG Institute of Mining Technology [33]. To achieve this objective, the following testing schedule was drawn up:

- a. Acquisition of raw materials being the natural aggregates. Extracting the representative samples from the acquired materials. Analysis of the samples for the content of rare earth elements. Selection of the three materials with the highest REE content.
- b. Separation of grain classes 0.045-0 mm from previously selected samples. Chemical analysis of the separated fine grain classes of the materials in terms of the content of rare earth elements to check if rare earth elements are more concentrated in the finest grains of the tested

raw material. This will make it possible to determine the variability in rare earth elements content depending on the grain size of the analysed raw materials.

- c. Analysis of the results obtained in items a and b. Determining the suitability of new materials. Summary of the work carried out.

2. Materials and Methods

2.1. Materials

Aggregates from igneous deposits: granite, basalt and aggregates from sedimentary deposits: sand and halloysite were selected for testing. Each of the sample weighed 20 kg. The characteristics of the materials used in the tests are presented below.

- Granite aggregate.

This material was taken from a granite mine conventionally marked by the symbol "A". This mine produces basalt aggregates in a wide range of grain sizes. The mine deposit is mined by drilling and blasting. Technology includes multi-stage classification and crushing. Material used in the tests is one of the mine's commercial products: 2-0 mm granite aggregate. The granite was collected by miners.

- Basalt aggregate 1, basalt aggregate 2.

These materials was taken from the "B" and "C" granite mines. The mines produce basalt aggregates in a wide range of grain sizes. Deposits of these mines are mined by drilling and blasting. Technology includes multi-stage classification and crushing. One of the mine's commercial products: 2-0 mm basalt aggregates was the material used in tests. The aggregates were collected by miners.

- Halloysite.

This material was taken from an open pit halloysite mine marked by the symbol "D". Halloysite from the mine deposit is a product of basalt rock weathering. The raw material has a homogeneous composition throughout the cross-section, there are no interlayers and vein-type inclusions.

Composition of the mineral is as follows:

- halloysite: 75-80%,
- iron oxides (mainly hematite and magnetite): 18-22%,
- iron and titanium oxides (mainly ilmenite): 2-4%.

Raw material taken from the deposit by miners was the material used in testing.

- Sand 1.

This material taken from the sand mine marked by the symbol "E". The mine extracts a sand and gravel from the deposit. Technology involves extraction of raw material from the deposit using a suction dredger. The material undergoes several stages of washing, classification and dewatering. Vibrating screens, circular dehydrator or pulsating classifier were the devices used for this purpose. Bottom product from the pulsating classifier sieves with a grain size <2 mm was the material used in the tests. The sand was collected by the personnel of gravel plant.

- Sand 2.

This material taken from the sand mine marked by the symbol "F". The mine extracts backfilling sand from the deposit as the main mineral and a sand and gravel mix as an accompanying minerals. The deposit is excavated using a bucket-wheel excavator. Then the material is transported to the processing plant, where it is washed, classified and dried. The commercial products are the following: construction sands, washed and dried sands and gravel. The material was collected from the deposit by employees of the KOMAG Institute of Mining Technology.

2.2. Methods

Reducing the samples to 0.5 kg by the quartering method was the first stage of testing. Reduced samples were sent to the Laboratory of Materials Engineering and Environment at KOMAG for determination of rare earth elements content. In the laboratory, the material samples were mineralized in a wet closed system. Microwave mineralizer was used for this purpose. Dissolution of the test samples was visually inspected. As a result of the mineralization process, a colourless and clear solution was obtained for most of the samples. The solutions were analysed for the of rare earth elements content using the mass spectrometry method with inductively coupled plasma ionization

(ICP-MS). On the basis of the test results, 3 materials of the highest rare earth elements content were selected, and were used in the further part of testing. Laboratory vibrating screen was used to obtain grain classes 0.045-0 mm. Samples of the selected materials weighing 1 kg were separated using the quartering method. The screening process was wet, using a sieve with a square mesh of size 0.045 mm. After the process completion, the products were dried and their mass output was determined. Then, the dried samples were sent to the laboratory for the determination of their content of rare earth elements using the ICP-MS method.

3. Results

Content of rare earth elements in the raw samples are presented in Table 1 and in Fig. 1. HREE is marked in red, MREE in orange and LREE in blue.

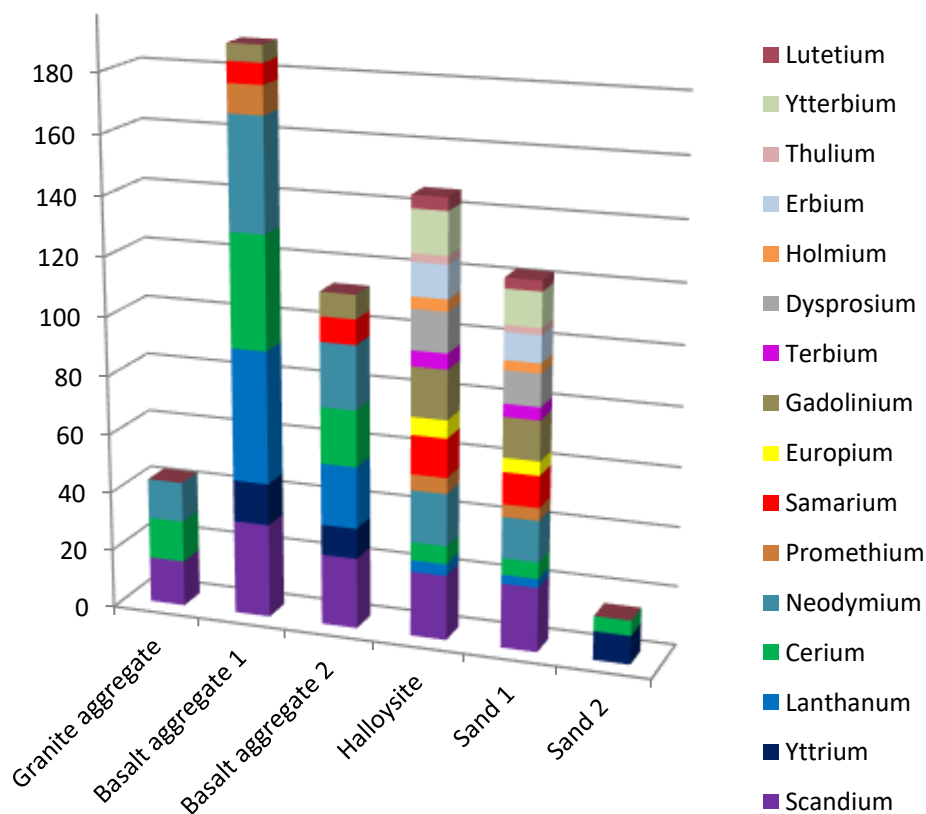


Fig. 1. Diagram of REE content in the raw materials [own source]

Table 1. Results of REE content [own source]

Material	Content of rare earth elements [ppm]									
	La	Ce	Nd	Pr	Y	Er	Tm	Yb	Lu	
Granite aggregate	-	14.0	13.5	-						
	Tb	Dy	Ho	Sc	Y	Er	Tm	Yb	Lu	
	-	-	-	15.5	-	-	-	-	-	-
	Sm	Eu	Gd	Σ REE=43.0						
	-	-	-							
Basalt aggregate 1	La	Ce	Nd	Pr						
	45.0	38.4	38.2	9.7						
	Tb	Dy	Ho	Sc	Y	Er	Tm	Yb	Lu	
	-	-	-	31.9	14.1	-	-	-	-	-
	Sm	Eu	Gd	Σ REE=190.0						
	7.0	-	5.7							
Basalt aggregate 2	La	Ce	Nd	Pr						
	20.9	19.1	21.7	-						
	Tb	Dy	Ho	Sc	Y	Er	Tm	Yb	Lu	
	-	-	-	24.0	10.3	-	-	-	-	-
	Sm	Eu	Gd	Σ REE=112.5						
	8.4	-	8.1							
Halloysite	La	Ce	Nd	Pr						
	4.0	6.1	17.6	5.4						
	Tb	Dy	Ho	Sc	Y	Er	Tm	Yb	Lu	
	5.3	13.5	4.0	22.0	-	11,1	2,8	14,2	4,4	
	Sm	Eu	Gd	Σ REE=146.2						
	13.1	6.1	16.6							
Sand 1	La	Ce	Nd	Pr						
	3.0	5.6	13.9	4.3						
	Tb	Dy	Ho	Sc	Y	Er	Tm	Yb	Lu	
	4.3	11.0	3.3	21.8	-	9,1	2,3	11,5	3,5	
	Sm	Eu	Gd	Σ REE=122.5						
	10.7	4.8	13.4							
Sand 2	La	Ce	Nd	Pr						
	-	5.1	-	-						
	Tb	Dy	Ho	Sc	Y	Er	Tm	Yb	Lu	
	-	-	-	-	9.7	-	-	-	-	-
	Sm	Eu	Gd	Σ REE=14.8						
	-	-	-							

From the tested raw material samples, basalt aggregate has the highest total content of rare earths, i.e. 190.0 ppm. The next material, in terms of the total content of the analysed elements, is halloysite - 146.03 ppm and sand 1 - 122.22 ppm. These materials were used in the further part of the testing aimed at determining the content of rare earth elements in fine grain classes. Table 2 presents the contents of HREE, MREE and LREE in the raw material samples.

Table 2. Total content of HREE, MREE and LREE in raw materials [own source]

Material	Content of rare earth elements [ppm]		
	LREE	MREE	HREE
Granite aggregate	27.5	0	15.5
Basalt aggregate 1	131.3	12.7	46.0
Basalt aggregate 2	61.7	16.5	34.3
Halloysite	33.1	35.8	77.3
Sand 1	26.8	28.9	66.8
Sand 2	5.1	0	9.7

The previously mentioned material samples, which showed the highest content of rare earth elements, were used to determine concentration of these elements in a fine grain class of 0.045-0 mm. Then, the percentage yields of the obtained grain classes were calculated and presented in Table 3.

Table 3. Output of 0.045-0 mm grain class in the samples of halloysite, sand and basalt [own source]

Material	Output of class 0.045-0 mm [%]
Basalt aggregate 1	11.5
Halloysite	38.7
Sand 1	0.65

After determining the outputs, the samples were analysed for the content of rare earth elements, using the ICP-MS method specified at the beginning of this section. Results of the analysis are given in Tables 4, 5 and in Fig. 2.

Table 4. Results of REE content in 0.045-0 mm grain class [own source]

Material	Content of rare earth elements [ppm]									
	La	Ce	Nd	Pr	Sc	Y	Er	Tm	Yb	Lu
Basalt aggregate 1	73.6	151.4	61.3	15.4						
	1.7	8.4	1.5	17.2	4.6	3.8	0.5	2.9	0.3	
	11.6	4.3	15.8	ΣREE=374.3						
Halloysite	2.0	8.4	2.3	1.0						
	0.1	0.9	0.1	16.9	-	0.5	-	0.5	-	
	0.7	0.3	1.0	ΣREE=34.7						
Sand 1	3.9	8.3	5.6	1.2						
	0.3	1.6	0.3	12.3	-	0.6	-	0.5	-	
	1.6	1.0	2.0	ΣREE=39.2						

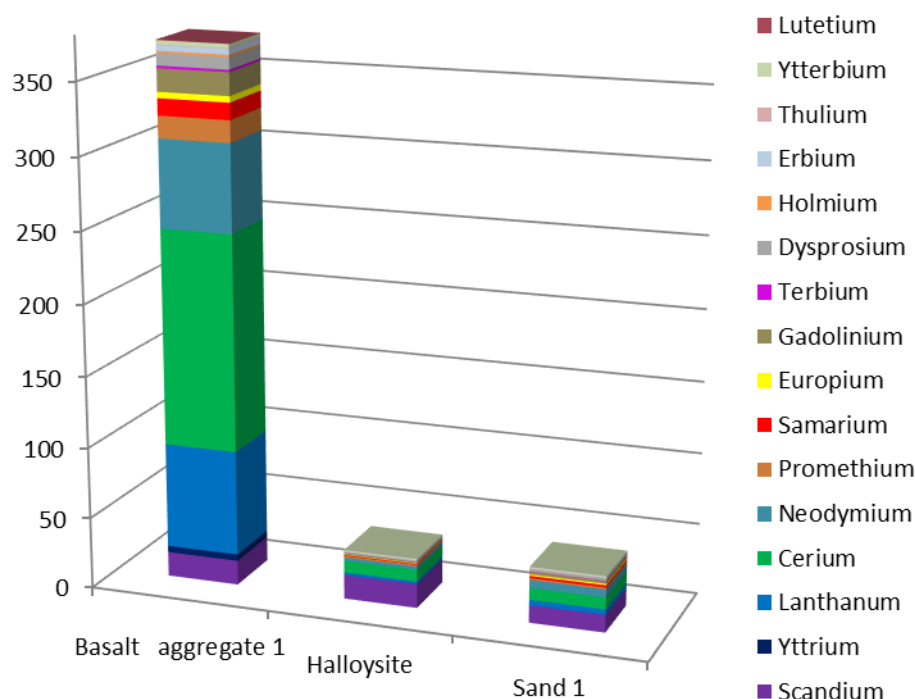


Fig. 2. Content of REE in analysed minerals [own source]

Table 5. Content of HREE, MREE and LREE in raw materials [own source]

Material	Content of rare earth elements [ppm]		
	LREE	MREE	HREE
Basalt aggregate 1	301.7	31.7	40.9
Halloysite	13.7	2.0	19.0
Sand 1	19.0	4.6	15.6

4. Discussion

Tests of granite aggregate showed the total content 43.0 ppm of 3 rare earth elements i.e. scandium 15.5 ppm, cerium 14.0 ppm, neodymium 13.5 ppm. The total 8 REE content in the basalt aggregate 1 was 190.0 ppm, of which lanthanum was of the highest content (45.0 ppm). Basalt aggregate 2 had 7 REE of total content 112.5 ppm. In the basalt aggregate 2, scandium has the highest content (22.0 ppm). Halloysite had 15 REE of total content 146.03 ppm, of which scandium has the highest content equal to 24.0 ppm. Sand 1 had an REE content of 122.5 ppm, comprising 15 elements, of which scandium had the highest content - 21.8 ppm. Sand 2 had REE concentration of 14.8 ppm. Yttrium - 9.7 ppm and cerium - 5.1 ppm were found only in this material.

Table 2 shows the share of heavy, medium and light rare earth elements in the tested raw materials. Crushed igneous aggregates: granite aggregate and basalt aggregate have a greater affinity for light rare earth elements (LREE). Other materials such as halloysite, sand 1 and sand 2 have a higher affinity for heavy rare earth elements (HREE).

From among the raw material samples tested, basalt aggregate has the highest total content of rare earth elements, i.e. 190.0 ppm. Halloysite - 146.2 ppm and sand 1 - 122.5 ppm were the next materials, in terms of total content of the tested elements. The lowest total content of rare earth elements was found in granite aggregate - 43.0 ppm and in sand 2 - 14.8 ppm.

The following materials: basalt aggregate 1, halloysite and sand 1 were selected for testing the content of rare earth elements in the grain class <0.045 mm. Halloysite had the highest content of grains <0.045 mm - 38.7%, and sand 1 the lowest - 0.65%. The expected increase in the content of rare earth elements after fine material separation was found only in the case of the basalt 1 aggregate. Total content of REE in this material increased from 190.0 ppm to 374.3 ppm, what means an increase of about 97%. Based on the results from Table 5, it can be concluded that the content of light rare earths increased the most. Before separation of a grain class 0.045-0 mm from the raw material, light rare earths accounted for 68% of the total REE content. After screening, total content of light rare earth elements in the grain class <0.045 mm was 81% of the total REE content. However, the increase in REE content was still insufficient for using the basalt aggregate for beneficial recovery of rare earth elements.

5. Conclusions

In 2009, the international Organization for Economic Cooperation and Development included rare earth elements on the list of the so-called strategic resources. In 2011, the European Union published a list of critical raw materials for the Union, raw materials of strategic importance for the development of state-of-the-art technologies. This list also includes rare earth elements. Due to strategic importance of these elements, it is necessary to look for their alternative sources. For this reason, tests were carried out on the content of rare earth elements in natural aggregates.

The aim of the project was to determine if the analysed natural aggregates contain rare earth elements at concentration enabling their economic recovery. The results of the tests indicate the presence of rare earth elements in the tested material. The results indicate a predominance of light rare earth elements (LREE) in basalt aggregate 1 and 2 and granite aggregate, while heavy rare earth elements (HREE) are predominant in halloysite, sand 1 and sand 2. Moreover, it was found that the cerium is the most common element, as it was determined in all samples of the tested materials.

In the further part of the tests, three materials of the highest content of the tested elements were selected, from which grain classes of 0.045-0 mm were separated. A decrease in the content of rare earth elements in halloysite 1 and sand 1 was observed. The only increase in the content of REE elements was found for the basalt aggregate 1. The content of REE in this material increased from 190.0 to 374.3 ppm, what gives an increase of about 97%.

The content of rare earth elements in the collected materials was in the range of 190.0-14.8 ppm. Even in the case of material containing the REE highest content, i.e. basalt aggregate, it is too low for beneficial recovery of these elements. The expected increase in the content of tested elements in the finest grains of materials was achieved only in the case of basalt aggregate - 374.3 ppm. The obtained, higher concentration of the REE is also insufficient for an economically justified recovery of rare earth elements.

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