PHYSICOCHEMICAL, MINERALOGICAL AND RADIOLOGICAL PROPERTIES OF RED MUD SAMPLES AS SECONDARY RAW MATERIALS

by

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The main goal of the presented research was the preliminary investigation of possibility of red mud – Hungarian dump sites Almasfuzito (sample A) and Ajka (sample B) – application as a pigment or as a raw material for use in the construction materials industry. Also, the aim of this work was the characterization of red mud as industrial waste generated by the Bayer process in the aluminum industry – which may cause environmental problems if appropriate treatment is not carried out. The main mineral phases of both red mud are hematite (Fe_2O_3), calcite ($CaCO_3$), gibbsite ($Al(OH)_3$) and they consists of particles of median particle size 2.1   m (sample A) and 2.5   m (sample B) and have a characteristic red color, which was the reason for its testing for use in the industry of building materials as a pigment for standard concrete mixtures. The radionuclides content in the samples was determined by gamma spectrometry, and the radiological hazards originating from ^{238}U, ^{232}Th, ^{40}K in the samples, were assessed through the radium equivalent activity, and the external radiation hazard index. The absorbed dose rate and the annual effective dose were calculated in accordance with the UNSCEAR 2010 report and the results are presented in this paper.

Key words: gamma-spectrometry, red mud, NORM, mineral composition, physicochemical property

INTRODUCTION

Red mud, or in other words „bauxite residue", is the waste generated during the Bayer's process of the extraction of alumina from bauxite. The Bayer process involves the digestion of crushed and ground bauxite in a concentrated sodium hydroxide solution at temperatures up to 270 °C and at high pressure. Under these conditions, the majority of the aluminum-hydroxide is dissolved, leaving an insoluble residue that is called red mud in the alumina refining industry. This red mud is removed by settling/filtration [1].

It is estimated that 3 billion tons of red mud are accumulated globally, with this amount increasing by approximately 120 million tons per year [2]. Since this material is a hazardous waste, predominantly due to its high alkalinity and sodium-hydroxide content, and because its safe deposition requires high cost investments, the research of environmentally friendly industrial recycling is very important and present worldwide. However, according to the European Waste Catalogue (EWC) red mud is not categorized as hazardous waste. Even if rare and critical elements are aimed for extraction from red muds, a safer and more favored way of re-utilization is their use as additives in construction materials. The most widespread use of red or brown mud processing is in the construction industry (production of building components – bricks, concrete blocks, ceramic materials, *etc*., concrete aggregate, special cement, *etc*.). Brown mud obtained by the calcination method contains suitable reactive components, *e. g.* β-2 $CaO.SiO_2$, so it can be used (unlike red mud) for direct bricks production [3] where it is pressed into blocks and is calcined at a high temperature, while the compression strength of the produced bricks reaches a value of 1.9 MPa [4].

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Mucsi *et al*. [5] investigated the utilization of red mud in synthesis of geopolymers, and the optimal quantity of red mud added to the fly ash geopolymer was found to be 15 %. Due to the geopolymerisation reactions new phases appears in the product, like hydroxy-cancrinite, faujasite and thenardite.

According to an EPA report [6, 7] red mud after neutralization is not classified as hazardous waste [8], because in four tested hazardous properties (corrosivity, reactivity, ignitability and toxicity according to the toxicity characteristic leaching procedure test (TCLP) it does not fulfill criteria for such classification. Performed eco toxicity tests indicated that neutralized red mud could be widely reused not only as a building material, but also as a raw material for metal production in the metallurgic industry or in glass production. Because of its large surface, after activation red mud becomes an excellent adsorbent and coagulant which can be used in remediation of soil in agriculture, the mining industry – neutralization of Acid Mine Drainage [9], in removal of toxic metals in waste treatment plants, in catalysis in the chemical industry, *etc*.

The generation of wastes is daily increasing with the ever faster industrial development and environmental control is becoming stricter, adequate legal restrictions and greater efforts of scientists are expected in the future. Since the recycling of wastes or by-products is gaining more importance nowadays, research related to these materials has been carried out in several countries where waste or by-products are used as additives for building materials [10-13]. A number of researchers have noticed the clay-like structure of the red mud when it is fired, and its possibility of becoming a useful ceramic material. Red mud can be used in the manufacturing of tiles, bricks and insulation materials [14, 15]. But it is known that many ores and raw materials contain relatively high levels of natural radionuclides, and processing such materials can futher increase its concentrations. The radionuclides are present in the bauxites in two major forms: adsorbed ionic species on the hydroxide and clay mineral grains, and as substituting ions or nanoscale oxide inclusions in the monazite and other REE-minerals. According to Bardossy *et al*. [16] 60 % of the Th and 100 % of the U contained in the Hungarian bauxites is retained in the red mud.

A „universal" technique of disposal, management and full utilization of red mud, an alumina production waste, has not yet been developed. Characterization of this waste material can help in determining its usage in different industries, or in revealing the places and methods of management and disposal that would come close to a technology harmless to the environment. The influence of hazardous elements and radioactivity in red mud should be avoided when applying the red mud in the production of building material.

The objective of this paper was to determine the physicochemical, mineralogical and radiological characteristics of two Hungarian red mud samples. Characterization included determination of the radionuclide content in two different samples of red mud, as well as the mineralogical analysis of these samples.

MATERIALS AND METHODS

The red mud (RM) samples were taken from two Hungarian dump sites Almasfuzito (sample A) and Ajka (sample B) tailing. After homogenization, analytical samples for characterization measurements were prepared by Jones type splitter after drying and disaggregation.

Mineralogical composition

The mineralogical composition was determined by a Bruker D8 Advance XRD powder diffractometer (Cu-Kα radiation, 40 kV, 40 mA) in parallel beam geometry (Gobel-mirror). Patterns were recorded in the 2-70° (2θ) range, with 0.007° (2θ) steps in 42 seconds, with a Vantec-1 position sensitive detector (1° window opening). Phase identification was made by Search/Match (multiple iterations) on an ICDD PDF2-Release (2012). Quantitative evaluation was made by Rietveld-refinement in the TOPAS4 software, using FPM based instrumental convolution (using SRM 640d Si), with crystal structure data from the AMCSD database. The broad peak fitting as an amorphous hump was used to test the amorphous content. For phase identification we used the following PDF cards: No: 01-074-7052 for cancrinite, No: 00-0070324 for gibbsite, No: 00-001-1053 for hematite and No: 00-005-0586 for calcite phase identification.

Physical and chemical properties

The particle size distribution (PSD) of the sample materials was measured by a HORIBA LA-950V2 laser diffraction particle size analyzer in wet mode using distilled water as the dispersing media. The geometric (outer) specific surface area (SSA) was calculated using PSD data by the laser sizer software with a Heywood (shape) factor of 1.

Radionuclide content in red mud

The content of radionuclides in the red mud samples was determined by gamma spectrometry. The samples were mechanically prepared (crushed and pulverized) and placed in PVC cylindrical boxes. Masses of measured samples were approximately 100 g. Samples were sealed with bee wax and measured after reaching radioactive equilibrium. Radiological analysis was performed by means of a semiconductor HPGe spectrometer GX5019, Canberra (relative efficiency 55 %; resolution 1.75 keV at 1332 keV (^{60}Co), 0.830 keV at 122 keV (^{57}Co), and 0.791 keV at 5.9 keV (^{55}Fe), associated with standard beam supply electronics units.

The standardized solution of the common mixture of gamma-emitting radionuclides, provided by the Czech Metrological Institute [17], was used to prepare the standards for the energy and efficiency calibration of the spectrometer in accordance with IAEA recommendations [18]. The measurement times were 255 000 s and 181 000 s, for sample A and B, respectively. Spectra were recorded and analyzed using the Canberra Genie 2000 software; net areas of the peaks were corrected for the background, dead time and coincidence summing effects (correction factors were calculated in the GESPECOR software).

Quoted uncertainties (confidence level of 1σ) were calculated by the error propagation calculation. The combined standard uncertainties included the efficiency calibration uncertainty and the statistical uncertainties of the recorded peaks, while uncertainty of nuclear data and measured mass were neglected.

Dose assessment

The gamma ray radiation hazards due to the specified radionuclides were assessed by the radium equivalent activity (Ra_{eq}) and external radiation hazard index [19]. The radium equivalent activity (Ra_{eq}) and external radiation hazards (H_{ex}) were calculated according to eqs. (1) and (2)

$$(Ra_{eq}) = C_{Ra} + 1.43\, C_{Th} + 0.077\, C_K \qquad (1)$$

$$H_{ex} \quad \frac{C_{Ra}}{370} \quad \frac{C_{Th}}{259} \quad \frac{C_K}{180} \qquad (2)$$

where C_{Ra}, C_{Th}, and C_K denotes specific activities (in Bqkg^{-1}) of ^{226}Ra, ^{232}Th, and ^{40}K, respectively.

Further, the external gamma radiation absorbed dose rate, D, in air at a height of 1 m above ground level was calculated using eq. (3) with conversion factors: 0.462 (nGyh^{-1})/(Bqkg^{-1}) for ^{238}U, 0.604 (nGyh^{-1})/(Bqkg^{-1}) for ^{232}Th and 0.0417 (nGyh^{-1})/(Bqkg^{-1}) for ^{40}K [19].

$$D = 0.462\, C_{Ra} + 0.604\, C_{Th} + 0.0417\, C_K \qquad (3)$$

where D denotes the dose rate in nGyh^{-1} and C_{Ra}, C_{Th}, and C_K denotes the specific activities in Bqkg^{-1}.

The annual outdoors effective dose was calculated utilizing a conversion coefficient of 0.7 Sv/Gy to convert the absorbed dose in air into the effective dose in the human body. This calculation takes into account that people spend about 20 % of their time outdoors (outdoor occupancy factor p is 0,2) and t is the 8,760 h annual exposure time. The annual effective dose, D_E, due to gamma radiation from soil was calculated as [19]

$$D_E = 0.7Dtp \qquad (4)$$

RESULTS AND DISCUSSION

Structural analysis of red mud

The chemical composition, physicochemical properties and mineralogy of red mud vary with the type of the bauxite and the alumina production process, and will change over time. The red mud can pose a serious pollution hazard. Red mud is mainly composed of coarse sand and fine particles. The chemical composition of the two kinds of red mud determined by the XRF analyzer is given in tab. 1.

It can be seen in tab. 1 that the main chemical components of red mud are Fe_2O_3, Al_2O_3, SiO_2, CaO, Na_2O, and TiO. The CaO and SiO_2 contents of sample B (Ajka red mud) are slightly higher than that of sample A red mud, but its Fe_2O_3 content is lower.

The XRD patterns of the red mud sample A and sample B are shown in fig. 1

The mineralogical composition of the investigated red mud samples is given in tab. 2. The mineralogical composition of the red mud depends on the mineral com-

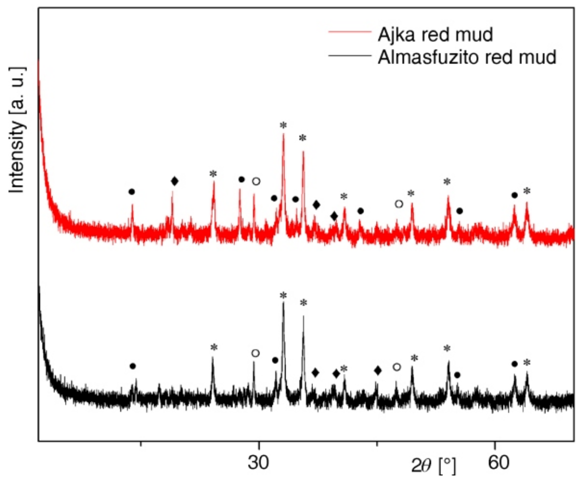

Figure 1. XRD patterns of the red mud samples; *hematite (Fe_2O_3) – marked by a solid star symbol, calcite ($CaCO_3$) – marked by a clear circle symbol, cancrinite ($Na_6Ca_2Al_6Si_6O_{24}(CO_3)_2$ – marked by a solid circle symbol), gibbsite ($Al(OH)_3$ – marked by a solid rhombus symbol)*

Table 1. The main chemical constituents of red mud [%]

Chemical constituent	Fe_2O_3	Al_2O_3	SiO_2	CaO	Na_2O	TiO_2	V_2O_5	Sc_2O_3	Loss on ignition
Almasfuzito red mud	25.13	19.24	7.52	22.84	5.75	8.60 7.98	0.34 0.68	0.76 0.92	9.82 12.54
Ajka red mud	23.11	20.18	8.45	23.79	6.35				

Table 2. Mineralogical composition of red mud samples

Mineral	Sample A (Almasfuzito red mud) [%]	Sample B (Ajka red mud) [%]
Hematite	45.47	49.95
Calcite	7.93	8.64
Cancrinite	35.48	12.38
Gibbsite	9.32	2.89
Siderite	–	2.15
Bohmite	–	6.34
Hydrogranate	–	17.65
Lime	1.8	–

position of the source material – bauxite. Bauxite is a multiphase ore that may contain, according to some references, more than a hundred minerals. Its essential constituents, however, are the minerals of aluminum, iron, silicon, titanium, calcium, magnesium, *etc*. Depending on the type of mineral deposits, the amounts of the essential and accessory minerals may vary within wide ranges. Aluminum is contained in bauxite in the form of hydrous oxides: hydrargillite, boehmite and diaspore, and at lower rates as corundum (Al_2O_3) or various aluminosilicates. Aluminum minerals are naturally concentrated and mixed in ores with many metals and petrogenic minerals. The most abundant gangue mineral in bauxites is free silica (various forms of crystalline SiO_2) or bound silicon oxide (in the form of aluminosilicates, commonly kaolinite). Iron in bauxite occurs in various minerals forming the principal waste (red mud) component. Iron minerals are hematite, magnetite, hydrohematite, goethite, and limonite. Its principal carbonates are siderite and ankerite. The commonest in bauxite are hematite and goethite, and less common are magnetite and limonite. Titanium is almost always found in bauxites, in the form of rutile, anatase or brookite. Bound titanium dioxide may be contained in bauxites in the form of sphene, perovskite or ilmenite. Carbonate constituents are calcite, magnesite, dolomite, hydromagnesite, and ankerite [20].

The red mud considered in this work largely depends on chemical and mineral compositions of the mineral ore, grinding fineness and effective leaching (decomposition). From the XRD patterns (fig. 1) it can be seen that the main mineral phases of red mud are hematite (Fe_2O_3), calcite ($CaCO_3$), gibbsite ($Al(OH)_3$), *etc*. It is indicated that the examined samples have obviously different mineral compositions. An amorphous content was not detected.

The particles size distribution of the two kinds of red mud is shown in fig. 2. It can be seen that the sample B (Ajka red mud) particle size distribution is slightly finer, in the size range below 3 μm with a median particle size value of 2.1 m. Compared with sample B (Ajka red mud), the sample A (Almasfuzito red mud) has a relatively coarser particle size distribution. The median particle size of sample A (Almasfuzito red mud) is 2.5 m.

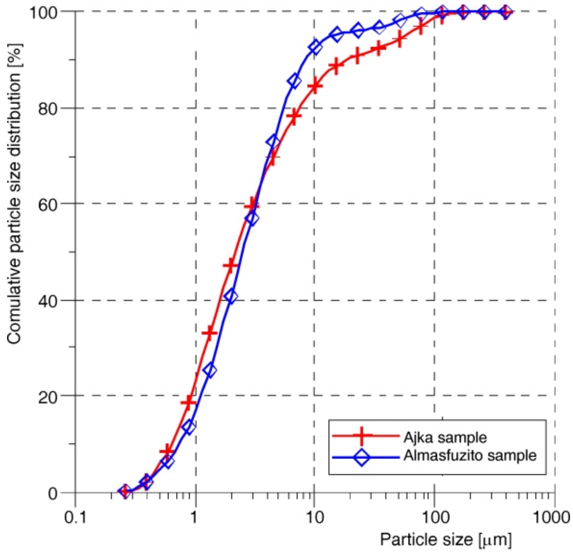

Figure 2. Particle size distribution of red mud samples

The measured values of the density of red mud are 3.19 gcm^{-3} and 3.03 gcm^{-3} for sample B (Ajka red mud) and sample A (Almasfuzito red mud), respectively. The differences of particle size and density values have a significant influences on the performances of red mud as the raw materials for cement and concrete. The geometric specific surface area of the Ajka and Almasfuzito red mud samples was 12450 cm^2g^{-1} and 11970 cm^2g^{-1}, respectively. Therefore, it can be summarized that the fineness of the two samples was very similar.

The chemical and mineralogical composition and the particle size distribution of red mud samples are different. This is a reason for the different properties of the two kinds of red mud.

Radiolgical analysis of red mud samples

The presence of the natural radionuclides, members of the uranium and thorium radioactive series, in red mud can represent an important issue in further utilization of this material.

As it was said before, radiological analysis of two different red mud samples was performed by using the gamma ray spectrometry system. Naturally occurring radionuclides, members of the uranium and thorium series and ^{40}K were detected. The presence of artificial radionuclides was not detected. The content of ^{137}Cs was below the detection limit (<0.3 $Bqkg^{-1}$). Obtained specific activities expressed in $Bqkg^{-1}$ of dry mass, as well as those from previously published papers, are given in tab. 3.

The obtained results for ^{226}Ra and ^{232}Th were mostly within the ranges that can be found in the literature for this kind of sample, and significantly higher than the world average for building materials. Measured specific activities of ^{40}K were mostly below quoted values, and much lower than the world average for building materials [21-23].

Table 3. ^{232}Th, ^{226}Ra, and ^{40}K activity concentrations in the red mud samples

Red mud samples	^{232}Th	^{226}Ra	^{40}K
	Measured in this study [Bqkg^{-1}]		
Sample A (Almasfuzito red mud)	364 18	185 10	17.5 1.5
Sample B (Ajka red mud)	298 15	473 32	24.8 1.6
	Mean value (range), previous studies [Bqkg^{-1}]		
Almasfuzito, Hungary [20]	103 (47-212)	294 (102-506)	103 (47-212)
Ajka, Bayer red mud, Hungary [20]	292 (285-380)	360 (150-700)	48 (5-101)
Bayer red mud from Guizhou, China [21]	404 (360-475)	302 (125-620)	113 (67-247)
Sintering red mud from Guizhou, China [21]	389 (340-450)	276 (95-570)	87 (58-164)
World average for building materials [22]	50	50	500

Regarding radioactivity, tab. 3 shows that the previously investigated red mud type Almasfuzito [20] has significantly (more than 3 times) lower contents of thorium, but a higher content of ^{226}Ra than the one measured in this study. The biggest difference compared to results from [20] was for the ^{40}K content – more than five times.

The differences in the measured activity of the sample of Ajka red mud in this and the previous study [20] are not so pronounced.

The observed differences in measured red mud samples indicates a different type of aluminum ore - bauxites. Some of the Hungarin bauxites contain significant amounts of monazite as accessory minerals, which always have a Th content [24]. The bulk bauxite samples posses 10-100 ppm U and Th contents [25].

The obtained calculated values of radium equivalent activity, external radiation hazard index, absorbed dose rate from outdoors terrestrial gamma radiation and the annual effective dose rate are given in tab. 4.

By comparing parameters Ra_{eq}, H_{ex}, D, and D_E with parameters related to soil investigation of Nenadovic *et al*. [26], it can be concluded that higher values were obtained.

CONCLUSIONS

According to the measurements and discussion both kinds of red mud (Almasfuzito and Ajka red mud) have the possibility of large scale application in the production of cement mixtures, but further investigation is necessary to study the long-term behaviour. Compared with the previously analyzed samples from Hungary in other studies, the measured samples have a comparable concentration of ^{226}Ra and similar concentration of ^{232}Th, but a significantly lower concentration of ^{40}K. Also, it can be concluded that both sintered red mud and Bayer red mud have higher values of radium and thorium concentrations than the world average for building materials, whereas for ^{40}K the opposite conclusion can be made.

Also, the possible use of red mud as a pigment in the construction materials industry (or any other where it represents a certain percentage of the final product) is restricted to adding a small percentage of this material in the final product, in order not to increase the activity of the final building materials. At the same time it has been noticed that there is a difference in the mineral composition which can affect to the differences relating the natural radioactivity of the sample.

Table 4. Radium equivalent activity, external radiation hazard index, absorbed dose rate and annual effective dose

Samples	Ra_{eq} [Bqkg^{-1}]	H_{ex} [Bqkg^{-1}]	D [nGyh^{-1}]	D_E [mSv]
Sample A	706.87	1.91	306.06	0.375
Sample B	901.05	2.43	399.55	0.490

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AUTHORS' CONTRIBUTIONS

Red mud sampling, sample preparation, chemical analysis, XRD and PSD measurement were carried out by G. Mucsi and F. Kristaly. Measurement of natural radioactivity and interpretation of results were carried out by I. Vukanac. M. Mirković interpreted results of XRD measurements. M. Nenadović, Lj. Kljajević and S. Nenadović performed literature research, theoretical analysis, and discussion of the presented results together with the other authors.

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ФИЗИЧКО-ХЕМИЈСКА, МИНЕРОЛОШКА И РАДИОЛОШКА СВОЈСТВА УЗОРАКА ЦРВЕНОГ МУЉА КАО СЕКУНДАРНОГ СИРОВИНСКОГ МАТЕРИЈАЛА

Главни циљ истраживања био је прелиминарно испитивање могућности примене црвеног муља (мађарске депоније Almasfuzito (узорак А) и Ајка (узорак Б)) као пигмента или као сировине за употребу у индустрији грађевинског материјала. Такође, циљ овог рада био је карактеризација црвеног муља као индустријског отпада који је произведен Бајеровим процесом у индустрији алуминијума – који може узроковати проблеме у животној средини ако се не третира на одговарајући начин. Главне минералне фазе оба црвена муља су хематит (Fe_2O_3), калцит ($CaCO_3$) и гибсит ($Al(OH)_3$). Они се састоје од честица средње величине 2.1 μm (узорак А) и 2.5 μm (узорак Б) карактеристичне су црвене боје, што је био разлог за њихово тестирање за употребу у индустрији грађевинских материјала као пигмента у стандардним мешавинама бетона. Садржај радионуклида у узорцима одређен је гама спектрометријски, а радиолошки ризик услед изложености ^{238}U, ^{232}U, ^{40}K присутних у узорцима, процењен је преко радијум еквивалент индекса и екстерног индекса радијационог ризика. У раду су приказани резултати процене јачине апсорбоване дозе (D) и годишње ефективне дозе (E), чије су вредности израчунате у складу са извештајем UNSCEAR 2010.

Кључне речи: гама-спектрометрија, црвени муљ, НОРМ, минерални састав, физичко-хемијска својства