

RADIOLOGICAL AND PHYSICOCHEMICAL PROPERTIES OF RED MUD BASED GEOPOLYMERS

by

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A significant amount of red mud generated as a by-product of the Bayer process in the aluminum industry may cause environmental problems if appropriate treatment is not carried out. The presented research dealt with the possibility of application of red mud as a pigment or as raw material for use in the construction material industry. In relation to the aim of this work, the physicochemical characterization was performed and the natural radioactivity of red mud as an industrial waste and a geopolymer sample based on it was determined. The presented research is a contribution to the potential solution for environmental protection through the synthesis of possible construction material based on red mud. The radiological hazard originating from ²²⁶Ra, ²³²Th, and ⁴⁰K in the samples was assessed through the absorbed dose rate and the annual effective dose rate, calculated in accordance with the UNSCEAR 2010 report. Physicochemical characterization of all samples was conducted using X-ray diffraction and diffuse reflectance infrared Fourier transform spectroscopy.

Key words: amorphous material, microstructure, pigment, red mud, spectroscopy

INTRODUCTION

Geopolymers are an aluminosilicate three-dimensional network structure that is formed from the reaction between an aluminosilicate-rich precursor and an alkaline activated solution. The process of making a geopolymer sample is performed in three stages: the process of dissolution of an aluminosilicate precursor to liberate aluminate and silicate species that hydrolyze and produce monomers; condensation where the monomers are rearranged by co-sharing the oxygen atoms to produce an oligomers gel and create larger networks and the last stage occurs when the mixture becomes oversaturated with the aluminosilicate gel which increases the connectivity of the network and the gel starts to harden [1]. Waste materials like red mud are utilized as the precursors, because of the presence of unreacted, silica and alumina. The activator solution contains alkali, silica, and water. Red mud does not have enough Si and Al content, so it is necessary to enrich it using metakaolin as their source. These two precursors (red mud and metakaolin) are mixed with an activator solution that causes a reaction

which produces a disordered alkali aluminosilicate gel. Red mud (RM) is a reddish suspension-like waste product obtained from the Bayer process in the aluminum industry with an average specific density higher than 2.9 gcm⁻³. The RM is mainly made up of oxides of iron, aluminum, silicon, and titanium. The high alkali content (pH up to 12) comes from the nature of the Bayer process where a high residual percentage of the sodium alkaline solution remains in a waste product (RM) [2]. Although the RM is usually considered as an inert waste material, depending on the process automation, RM can exhibit high alkalinity as well as a corrosive nature [3]. Because of its alkaline nature its disposal is an important environmental issue. In the cases when the dump or basins for the RM disposal are not equipped with the adequate geo-membrane protection the potential leaking into the soil or ground waters can be one of the most urgent environmental issues. It is reported that 0.8-1.5 tons of red mud is generated per ton of alumina produced [4]. A lot of research has reported on the potential re-use of RM in the brick industry, the industry of construction materials or roads construction. The presented research here is another contribution to the potential solution of this environmental issue through the synthesis of potential construction

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material based on RM (geopolymers). In the process of geopolymerization the RM is used as the aluminosilicate source which is alkali activated creating a 3-D structure similar to the polymers. Because of its upgrading properties (mechanical properties, corrosion resistance, resistance to aggressive environments, resistance to high or low temperatures), a geopolymer offers a lot of application possibilities in the construction industry. The full understanding of its nature is of utmost importance for further processing.

The content of naturally occurring radionuclides in manufactured building material products [5-7] is important from the standpoint of radiation protection. Gamma radiation of the primordial radionuclides (^{40}K and members of the uranium and thorium series) increases the external gamma dose rate [8, 9].

In recent years, considerable research and development work for the comprehensive utilizations of red mud have been put into practice all over the world, but to date very few technologically and economically viable solutions have been found [10]. Studies on the utilization of red mud for making efficient and low-priced building materials namely ordinary Portland cement [11], clay-based ceramics [12], bricks [13] and glass-ceramics [14, 15] have been reported. However, more and more precedence is being given to limiting the radiological dose of building materials on the population these days. Since RM contains radioactive elements like ^{226}Ra and ^{232}Th , this may be another key problem for the further utilization of red mud.

The aim of this research was to investigate the possible influence of polymerization processes on the natural radioactivity of alkali-aluminum materials synthesized by metakaolin and red mud as a byproduct of the Bayer process of obtaining alumina from the Aluminum Factory, Podgorica. Calcium hydroxide is added due to a formation of the calcium silicate hydroxide gel (CSH) under low alkalinity conditions. The presence of CSH can boost the mechanical properties, especially compressive strength. Through the understanding of the role that calcium plays in the geopolymer we can also explain the product's radiological and physicochemical properties. FTIR and XRD analysis were used to monitor the forming of new chemical bonds during the synthesis, and specific activities of natural radionuclides (^{226}Ra , ^{232}Th , and ^{40}K) were determined by gamma spectrometry measurements.

EXPERIMENTAL RESEARCH

Raw materials

Metakaolin (MK) was obtained by thermal treatment of kaolinite from the welding electrode production process in the Company "Piva", Montenegro. In the solid main raw material metakaolin, the red mud as

a by-product of the Bayer process of obtaining alumina from the Aluminum Factory, Podgorica, is added.

The raw mixtures (solid part) were used in this research: metakaolin (80 wt. %), red mud (15 wt. %) and $\text{Ca}(\text{OH})_2$ (P. A. purity) (5 wt. %). Si/Al ratio is 1.48 (GPRM class of specimens).

The alkaline solution was prepared from sodium silicate (Merck, $\text{Na}_2\text{O} : \text{SiO}_2 = 3.4, \text{Na}_2\text{O} 7.5-8.5 \%$, $\text{SiO}_2 25.5-28.5 \%$, and $d = 1.347 \text{ gcm}^{-3}$). and 4 mol dm^{-3} NaOH (analytical grade, Merc). A volume ratio of $\text{Na}_2\text{SiO}_3/\text{NaOH}$ was 2.5. The reference geopolymer (GPRM) was formed from metakaolin/red mud and the alkaline solution (solid/liquid ratio (S/L) was 1.45).

Methodology of alkali treatment activation of metakaolin/red mud

The specimens were prepared according to the following procedure:

- sieving of raw-mixture components through a 250 μm sieve,
- homogenization of pulp by mixing of the solid and liquid components of the raw mixture,
- molding into cylindrical containers dimensions ($d = 33 \text{ mm}, h = 17 \text{ mm}$),
- keeping specimens closed at 25 °C for the duration of 3 hours,
- drying of the specimens at 60 °C for the duration of 2 hours, and
- air aging of the specimens closed for the duration of 24 hours and opened for 7, 14, 21, and 28 days.

Materials characterization

XRD methods

All samples were characterized by X-ray diffractometry (XRD) by using an Ultima IV Rigaku diffractometer, equipped by with $\text{Cu K}\alpha_{1,2}$ radiation, with generator voltage 40.0 kV and generator current 40.0 mA. The range of 5°-80° 2θ was used for all powders in a continuous scan mode with a scanning step size of 0.02° at a scan rate of 2 °/min.

DRIFT spectroscopy

Diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS) is a cheap, fast and non-destructive way of evaluating clay minerals and their products [16]. Drift spectra were obtained using the Perkin-Elmer FTIR spectrometer. Approximately 5 % samples were dispersed in oven-dried spectroscopic grade KBr with the refractive index of 1.559 and particle size of 5-20 μm . Background KBr spectra were obtained and spectra rationed to the background. The spectra were scanned at 4 cm^{-1} resolution and collected in the mid-IR region from 4000 to 400 cm^{-1} .

Gamma spectrometry

The specific activities of naturally occurring radionuclides in the samples were determined by gamma spectrometry. The samples were mechanically prepared and measured in PVC cylindrical containers (125 cm³). Samples were sealed in order to reach radioactive equilibrium between ²²⁶Ra and its daughter products. A radiological analysis was performed by means of a coaxial semiconductor high purity germanium (HPGe) detector (Canberra GC5019, with 50 % relative efficiency and 1.8 keV resolution for ⁶⁰Co at the 1332 keV line) associated with standard beam supply electronics units. A certified solution of mixed gamma-emitting radionuclides (²⁴¹Am, ¹⁰⁹Cd, ¹³⁹Ce, ⁵⁷Co, ⁶⁰Co, ¹³⁷Cs, ²⁰³Hg, ¹¹³Sn, ⁸⁵Sr, and ⁸⁸Y), purchased from the Czech Metrology Institute (CMI) and traceable to BIPM, was used for the preparation of standards for the energy and efficiency calibration of the spectrometer in accordance with IAEA recommendations [17]. Obtained efficiencies were corrected for the coincidence summing effect using the correction factors obtained by EFTRAN software [18]. All spectra were recorded and analyzed using the Canberra Genie 2000 software; net areas of the peaks were corrected for the dead time, background and coincidence summing effects. The measurement times were 60 000 s. The obtained specific activities are given in tab. 1. Artificial radionuclide ¹³⁷Cs was below detection limits. Quoted uncertainties (the confidence level of 2σ) were calculated by the error propagation calculation. The combined standard uncertainties included the efficiency calibration uncertainty and the statistical uncertainties of the recorded peaks.

RESULTS AND DISCUSSION

Metakaolin and red mud precursors (solid phase) are mixed with an activator solution (liquid phase) that causes a reaction which produces a disordered alkali aluminosilicate gel – red mud based

Table 1. Activity concentration (in Bqkg⁻¹) of natural radionuclides in RM and GPRM with associated measurement uncertainties (*k* = 2)

Radionuclides	Activity concentration [Bqkg ⁻¹]		Activity ratio	
	RM	GPRM	GPRM/RM	
¹³⁷ Cs	<0.3	<0.5		
²¹⁰ Pb	90 30	33 3	0.37	0.13
²¹⁴ Pb	156 8	54 3	0.35	0.03
²¹⁴ Bi	158 9	50.0 4.6	0.32	0.03
²²⁶ Ra	157 9	52 4	0.33	0.03
²³⁸ U	170 40	44 5	0.26	0.07
²³⁵ U	9.9 0.6	1.69 0.14	0.17	0.02
²³² Th (²²⁸ Ac)	430 22	86 4	0.20	0.01
⁴⁰ K	80 5	440 20	5.50	0.43

geopolymer. Physicochemical characterization was conducted using XRD and DRIFTS while radiological analysis was done by gamma spectroscopy.

XRD analysis

Figure 1 shows the XRD pattern of RM and GPRM. The XRD patterns of the phase are referred from the ICDD base of the PDXL2 software. Mineralogical composition of the red mud depends on the mineral composition of the source material – bauxite a multiphase ore that may contain more than a hundred minerals of various grinding fineness and decomposition [19]. The minerals aluminum, iron, silicon, titanium, calcium, magnesium are essential constituents. Depending on the type of mineral deposits, the amounts of the essential and accessory minerals may vary within wide ranges [20]. From fig. 1 it can be seen that the main components are hematite (Fe₂O₃) (ICDD No. 01-085-0599), gibbsite (Al(OH)₃) (ICDD No. 01-070-2038), and rutile (TiO₂) (ICDD No. 01-075-1750). In addition to these minerals, some research has shown the presence of other minerals: calcite (CaCO₃) and cancrinite (Na₆Ca₂Al₆Si₆O₂₄(CO₃)₂), which are not found in red mud of Montenegrin origins [20].

The geopolymer is formed by the alkaline activation of the solid phase (metakaolin and red mud) with an alkaline activator. The characteristic halo that appears in the geopolymer in the range of 2θ from 20 to 35 is poorly expressed. Figure 1(b) shows that the main crystal phases of geopolymer are quartz (SiO₂) (ICDD No. 01-089-8936), muscovite (KAl₂(AlSi₃O₁₀)(F,OH)₂) (ICDD No. 00-002-0058) and a minor crystal phase (grossular (Ca₃Al₂(SiO₄)₃) (ICDD No. 01-072-1491) appeared in the sample obtained during the process of making the geopolymer. Wider research on red-mud-based geopolymers points also to the potential presence of muscovite [21] and quartz [22]. Generally the content of red mud depends on bauxite quality and

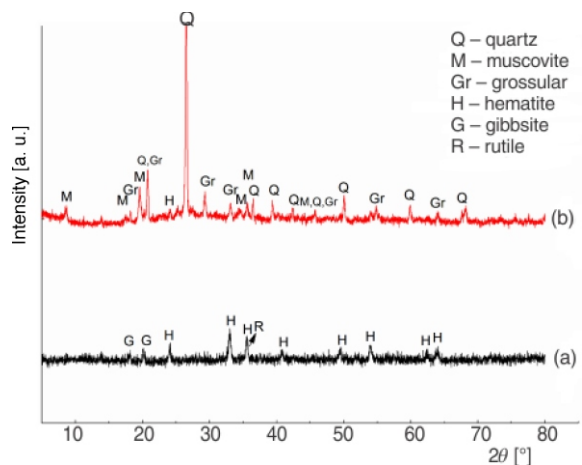


Figure 1. XRD pattern of red mud (a) and the geopolymer based on red mud (b)

content. What is particular for the Podgorica red mud is quite a significant presence of hydrate Fe oxides. Grossular is a calcium-aluminum species of the garnet group of minerals. In part, the calcium may be replaced by Iron (II). Also, the aluminum from grossular may be replaced by Iron (III) which is presented in red mud. Peaks corresponding to hematite found in red mud overlap with peaks that correspond to other crystalline phases in the geopolymer sample.

DRIFT spectra of red mud and geopolymer

DRIFT is more applicable to powders but is limited to some extent by the Reststrahlen effects where the particle size and the incident IR wavelengths create interference effects. These effects appear toward the low-frequency region, normally below 1000 cm^{-1} . Such effects are minimized by mixing the sample with KBr at the 5 % level [16].

The DRIFT spectra of RM and GPRM are shown in fig. 2. Figure 2(a) shows small but sharp bands that occurred around 3619 and 3529 cm^{-1} and small diffuse bands at 3290 and 3096 cm^{-1} . These may be due to the stretching vibrations of O-H bonds and H-O-H bending vibrations of the interlayer adsorbed H_2O molecule [23]. Stretching vibrations of C=O are found at 1451 cm^{-1} confirming the presence of carbonate groups [24]. The main reason being the presence of chemisorbed CO_2 in RM or which might be formed from the remaining unreacted activator solution and CO_2 [25]. The Si-O stretching band is located in the range of 1000 - 1200 cm^{-1} , whilst the Si-O bending bands are found at 800 cm^{-1} and in the range of 890 - 975 cm^{-1} . The band at approximately 1100 cm^{-1} is assigned to the Si-O stretching in tetrahedrons [26-28]. Characteristic bands of Si-O and O-Si-O group are observed at 1007 cm^{-1} confirming the presence of silicate groups. A minor stretching vibration of Fe-O is observed in the region around 470 cm^{-1} .

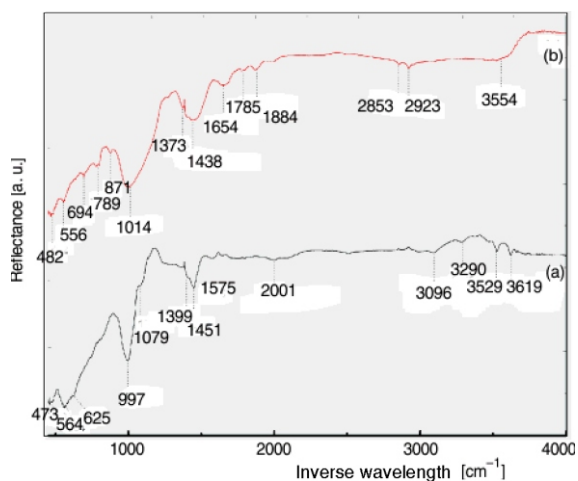


Figure 2. DRIFT spectra of red mud (a) and geopolymer samples (b)

As a result of the alkaline activation process of the metakaolin/red mud precursor a geopolymer sample was obtained. The Si-O-Al asymmetric stretch band overlaps with peaks which correspond to the Si-O or Si-O-Si stretching vibrations. The strong and broad peak is centered around 1014 cm^{-1} fig. 2(b). The peak at 1654 cm^{-1} corresponds to the H-O-H bending vibration.

A peak at 1438 cm^{-1} belongs to the O-C-O stretching vibration of the carbonate phase which is shifted in relation to the red mud. The peaks at 1100 - 473 cm^{-1} in relation to Si-O-Si, Al-O-Si are asymmetric and symmetric stretching and bending vibrations. As a result of red mud addition some peaks are shifted to a smaller or larger wavelength (473 to 482 cm^{-1} , 564 to 565 cm^{-1}). It shows that slight differences in newly formed phases can be observed due to red mud addition. However, the most significant change was detected concerning the carbonate peak which was shifted from 1451 to 1438 cm^{-1} , at the same time the peak is broadest probably due to red mud addition indicating the change in the concentration of carbonate compounds.

Radiological analysis

Results of gamma spectrometric analysis are given in tab. 1, and based on these values the dose calculations were performed.

Measurement results showed that specific activities of the synthesized geopolymer were mostly significantly lower than those in the RM sample. For the members of the uranium series the activity ratio was statistically the same. This ratio was slightly lower for ^{232}Th and ^{235}U , while for ^{40}K it has a comparably high value.

In previous studies Puertas *et al.*, [29] confirmed on a series of samples that the activity concentration of the hydrated or activated end product by unit of mass is slightly lower than in the anhydrous material because of the presence of water. The extent of the decline closely parallels the proportion of hydration of water/water hydration (40-50 %). However, in our research we monitored the change in radioactivity after the polymerization process, *i. e.* the geopolymer synthesis.

A definite conclusion regarding the influence of polymerization processes on the natural radioactivity of a red mud based geopolymer cannot be deduced due to the fact that RM was added to a kaolinite in a certain amount. At the same time, ^{40}K specific activity in the end product has increased notably, which could be expected due to the fact that kaolinite is derived from clay, which is (in most cases) rich in potassium, and therefore with the ^{40}K isotope. In summary, a total specific activity of RM is more than two times higher than the total specific activity of the geopolymer based on it. This conclusion could be affirmative concerning the

potential solution for environmental protection, through the synthesis of the geopolymer based on RM as a possible construction material.

Dose calculations

In order to estimate the possible health effects due to the exposure to natural radionuclides present in the measured samples the radium equivalent activity, Ra_{eq} [$Bqkg^{-1}$], the external hazard index, H_{ex} [$Bqkg^{-1}$], absorbed gamma dose rate D ($nGyh^{-1}$), and annual effective dose rate EDR ($mSvy^{-1}$) were calculated and presented in tab. 2. All the calculations were performed under the assumption that investigated matrices will be used as construction materials.

The radium equivalent activity was used to estimate the hazard associated with materials that contain ^{226}Ra , ^{232}Th , and ^{40}K . The external radiation hazard index reflects the external radiation hazard due to the emitted gamma radiation. The radiation hazard is insignificant if its value is less than unity, which corresponds to $370 Bqkg^{-1}$ of radium equivalent activity. The values of these indicators of exposure were calculated according to eqs. (1) and (2), respectively, [30]

$$Ra_{eq} = A_{Ra} + 1.43A_{Th} + 0.077A_K \quad (1)$$

$$H_{ex} = \frac{A_{Ra}}{370} + \frac{A_{Th}}{259} + \frac{A_K}{4180} \quad (2)$$

where A_{Ra} , A_{Th} , and A_K denotes specific activities of ^{226}Ra , ^{232}Th , and ^{40}K in $Bqkg^{-1}$.

The gamma radiation absorbed dose rate, D , in the air due to radionuclides ^{226}Ra , ^{232}Th , and ^{40}K in the measured samples was calculated using eq. (3) [31]

$$D [nGyh^{-1}] = 0.92C_{Ra} + 1.1C_{Th} + 0.08C_K \quad (3)$$

The annual effective dose rate, EDR [$mSvy^{-1}$], was estimated utilizing a conversion coefficient of $0.7 SvGy^{-1}$ to convert the absorbed dose in the air into the effective dose in the human body, assuming that the annual exposure time is 7000 h and using the eq. (4), [9, 31]

$$EDR [mSvy^{-1}] = 0.7 [SvGy^{-1}] 7000 [h] D [\mu Gyh^{-1}] \quad (4)$$

Table 2 presents the calculated values of the radium equivalent activity (Ra_{eq}), radiation hazards (H_{ex}), absorbed dose rate (D), and annual effective dose rate (EDR) due to radionuclides present in red

Table 2. Calculated radium equivalent activity, external radiation hazard index, external absorbed dose rate and annual effective dose rate

Sample	Ra_{eq} [$Bqkg^{-1}$]	H_{ex} [$Bqkg^{-1}$]	D [$nGyh^{-1}$]	EDR [$mSvy^{-1}$]
Red mud (RM)	778.06	2.104	623.84	3.057
Geopolymer (GPRM)	208.86	0.578	177.64	0.870

Table 3. ^{40}K , ^{226}Ra , and ^{232}Th activity concentration of the RM samples

Red mud sample	Radionuclide concentration [$Bqkg^{-1}$]		
	^{226}Ra	^{232}Th	^{40}K
Bayer red mud from Guizhou, China [32]	302	389	113
Bayer red mud from Hungary [33]	360	292	48
Bayer red mud from Montenegro	157	440	80
Geopolymer, Montenegro	52	80	440
World average of building materials [9]	40	30	400

mud and geopolymer samples. As can be seen from tab. 2, the dose assessment indicates that the synthesized material is considerably more suitable from the radiological point of view for a promising construction material.

Several authors have measured the content of naturally occurring radionuclides in RM and their results, in comparison with the world average for building materials and those obtained in this study are presented in tab. 3.

Compared with the samples from China and Hungary, our samples have a relatively low concentration of ^{226}Ra and high concentration of ^{232}Th . However, compared with the world average for building materials, the values of radium and thorium specific activities in red mud are significantly higher, so their use in the building industry should be restricted in accordance with the requirements of radiological protection.

CONCLUSIONS

XRD analysis of the red mud and geopolymer sample shows the present phase of the red mud that was used as a monomer in the polymerization process and presents a new crystal phase in the geopolymer sample. By using XRD and DRIFT analysis the process of polymerization after 28 days and formation of a new bond in the geopolymer sample were followed.

In these investigations the ratio of natural radionuclides during the polymerization process is monitored. Using the obtained results, one can conclude that after alkaline activation of red mud, the decrease of specific radioactivity was measured/detected in comparison to red mud. So, the lowest values of the specific activity of naturally occurring radionuclides were measured in inorganic polymers (geopolymers). From the aspect of the natural radioactive obtained material (geopolymers) it can be recommended as a potential building material. This research confirmed that during the polymerization process the natural radioactivity is reduced, *i. e.* a process of an activation of red mud has influence on the natural radioactivity of the precursor.

Despite these promising results, the mixtures composed of red mud and other matrix components that are used in building material factories must be investigated, since certain components can have an effect on one another, which in turn can cause a potentially harmful final structure. Data obtained in this research recommend the use of red mud as an ecological material for the synthesis of future promising building materials.

Also, when metakaolin is used alone in the process of geopolymerization natural radioactivity is reduced after the geopolymerization [34]. Due to the potential application of these kinds' of materials in the construction industry this presents very important information.

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AUTHORS' CONTRIBUTIONS

Red mud sampling and sample preparation were carried out by I. V. Bošković and M. A. Vukčević. Measurement of natural radioactivity and interpretation of results were carried out by I. S. Vukanac. N. G. Stanković and J. M. Luković interpreted results of XRD measurements. S. S. Nenadović and Lj. M. Kljajević performed literature research, theoretical analysis and discussion of the presented results together with the other authors.

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РАДИОЛОШКЕ И ФИЗИЧКО-ХЕМИЈСКЕ КАРАКТЕРИСТИКЕ ГЕОПОЛИМЕРА НА БАЗИ ЦРВЕНОГ МУЉА

Значајна количина црвеног муља која се генерише као нус-производ Бајеровог процеса у индустрији алуминијума може узроковати проблеме у животној средини ако се не предузму одговарајући третмани. Приказано истраживање бавило се могућношћу примене црвеног муља као пигмента или као сировине за употребу у индустрији грађевинских материјала. У вези са циљем овог рада, урађена је физичко-хемијска карактеризација и одређена природна радиоактивност црвеног муља као индустријског отпада, као и геополимера на бази црвеног муља. Приказано истраживање представља допринос потенцијалном решењу заштите животне средине кроз синтезу могућег грађевинског материјала – геополимера на бази црвеног муља. Радиолошка опасност која потиче од ^{226}Ra , ^{232}Th , и ^{40}K у узорцима процењена је кроз апсорбовану јачину дозе и годишњу ефективну јачину дозе, израчунату у складу са извештајем UNSCEAR 2010. Физичко-хемијска карактеризација свих узорака спроведена је помоћу рендгенске дифракције и DRIFT спектроскопије.

Кључне речи: аморфни материјал, микроструктура, пигмент, црвени муљ, спектроскопија