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MATERIAL COMPOSITION AND PROPERTIES OF RED MUD COMING FROM DOMESTIC ALUMINA PROCESSING PLANT

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Abstract
Red Mud from domestic alumina plant contains high excess alkaline components and traces of many toxic heavy metals. The results showed that Red Mud contains as main components hematite, goethite, residual aluminium oxide-hydroxides, desilication products (DSP), TiO_2 and does not contain radioactive elements such as Uranium (U), Thorium (Th). U and Th in Red Mud are less than the average concentration values of Uranium (4ppm) and Thorium (12 ppm) in the earth´s crust. Physical structural composition of domestic Red mud can be classified by an equilateral triangle iron oxide/hydroxide-sandy components-clays (23.2÷53.7% iron oxide/hydroxide; 34.1÷63.8% sandy components; 7.3÷34.1% clays). Chemical composition of red mud is, by weight (%w/w): Fe_2O_{3Tot} in the range 31.02 to 47.04; Na_2O: 2.45÷5.39; CaO: 3.01÷20.86; K_2O: 0.02÷0.07; MgO: 0.05÷ 0.73; Al_2O_3: 10.97÷22.78; SiO_2: 1.09÷8.28; TiO_2: 2.59÷ 5.76; Loss of Ignition (LOI): 13.03÷ 38.75. Red Mud also contains traces of some heavy metals and valuable metals (Cr, Mn, Zn, V, Ga, Gd, Sc, etc.) and has the pH in the range 13.0 ÷ 11.9 (when it is fresh) and 9÷10 (when it is aged) respectively. The search results are an important basis to select the proper treatment and recovery this waste as secondary resources for ceramic materials production (ceramic foams).

Keywords: *alumina plant, chemical analysis, material properties, red mud, wastes*

Introduction
The red mud is the solid waste generated in the Bayer process alumina production (Al_2O_3) from high bauxite grade ore, which has fine, micronized to colloidal texture and a chemical composition that varies due to the many types of bauxite processed over the time in Romania. The red mud contains mainly elements such as Fe, Al, Si, Ti, Na, Ca, traces components (V, Ga, Sc, Mg, Ba, K, Cu, Pb, Zn, Cr, Ce, Gd etc.) and has an excessively alkaline pH. The high alkalinity level is due to the residual non-recoverable soda content; however it is acceptable for storage as non-hazardous waste (01 03 09), (Council Directive 91/689/EEC 1991). The US Environmental Protection Agency has rated red mud as a residue based on 4 risk characteristics: corrosively, reactivity, ignition capacity and extraction procedure toxicity but does not classify it as hazardous waste. However, large quantities generated globally at world level (between 0.8 and 2.0 Mt/Mt Al_2O_3) could be a problem for the environment due to deposits and possible leaching under severe environmental

conditions. At the same time, red mud pits can lead to impacts and risk perceived by the population in the areas surrounding these deposits by: landscape changes and visual discomfort, air pollution, surface water pollution, soil fertility changes and biocenosis composition on the land of adjacent landfills. Removal from the natural or economic circuit of land for red mud deposits is a process that can be considered temporary but which, in terms of the concept of "sustainable development", extends over at least two generations if the planning periods are summed up (1-3 years), exploitation period (about 15-30 years), ecological recovery and post-monitoring (15-20 years). In terms of biodiversity, such a deposit means removing about 30-300 species / ha from the affected area, without considering the microbiological population of the soil. In addition, the biocenosis in the vicinity of the landfill are changing in the sense that vegetal associations become dominate by the ruderal species specific to the polluted areas, and some mammals, birds or insects leave the area. These features require precautionary storage and intense concerns for finding alternative solutions (a pre-treatment to reduce the level of alkalinity might be beneficial) and recovery (for example, as a secondary resource for production of building materials, special cements, adsorbents and catalysts for wastewater treatment, recovery of useful elements, and more recently the obtaining of ceramic oxide materials), (Çengeloğlu 2003; Paramguru 2005; Sglavo 2000; Yalcin 2000; Patent RO131328 (A2) 2016). The composition and properties of Red Mud in the world were presented by many researchers in their publications (Snars 2009). The results from this work showed that: Red Mud generated from domestic alumina plants contains a high content of oxides, such as: Fe_2O_3, Al_2O_3, SiO_2, TiO_2, Na_2O, CaO, K_2O, MgO, and sometimes a high concentration of valuable metal elements (V, Ga, Sc, Gd, etc.) and does not contain radioactive elements such as U, Th, are less than the average concentration values of Uranium (4 ppm) and Thorium (12 ppm) in the earth´s crust (Galateanu 1976; Hodgman 1960). The physical-structural composition of the domestic Red Mud is based on three classes of components: iron-oxide/hydroxides-sandy components-clays. However, due to the bauxite origin and the technology utilized by the alumina processing plant, the composition and properties of Red Mud are very different. Currently, the alumina processing plant, from Romania, is a big and modern plant with a capacity 600,000 Mt of alumina annually (Vitmeco Alum 2017). The Romanian processing plant uses bauxite from the Sierra-Leone mine and other types of bauxite and Bayer technology for alumina production. A second alumina processing plant, which mainly used boehmite-diaspore and boehmite-gibbsite bauxite ore, was closed several years ago, but there are still red mud deposits in its vicinity. Investigation of the composition and properties of Red Mud from domestic alumina processing plant in order to turn its use towards the obtaining of ceramic oxide materials (foams) is a main objective of the project "WATOPREM" PN-II-PT-PCCA-2013-4-177, Ctr. 78/2014.

Materials and Methods

Fresh Red Mud (RM2, RM7, RM8, RM9 red mud samples) were collected from red mud deposits located in vicinity of alumina processing plant from east of Romania, in April, 2015;

Older Red Mud (up to 1 year aged), RM10, RM11, RM12, RM13, RM14 red mud samples, were collected from a red mud deposit near the closed alumina processing plant from west of Romania, in February 2013.

Determination of basic physical and chemical characteristics of Red Mud

Physical texture of Red Mud was determined using dry sieving-classified particles method according to the methods SR EN 244971994; SR EN 12192-1 2003.

Red mud samples dissolved by hot acid HCl 1:1, norm up 250mL, or through dissolution in HNO_3 + HCl acids while the solid residues were digested by fusing Na_2CO_3 and K_2CO_3 in a platinum dish at 1,000^{0}C. The solutions were used to determine the percent concentration (%w/w) of the oxides and heavy metals.

Fe_2O_3, Al_2O_3, SiO_2, TiO_2, MgO, trace elements: Cu, Cr, Ni, Zn, Zr, Ba, Ce, Gd, Sc, U, Th were determined by the spectrometric methods SR EN 14242 2005; ASTM E1479 2016, using an ICP-OES AGILENT 725 equipment;

Na_2O, CaO, K_2O, trace elements Ga, Cd and Pb were analysed by atomic absorption spectrometry using AAS ZEEnit 700 Analytic Jena AG firm, Germany equipment according to STAS 3223/2- 1980; STAS 3223/1 1992; STAS 1269/17 1982; STAS 1269/7 1983; STAS 1269/3 1987, IEC 61010-2-061, 2015.

Loss on Ignition (LOI) was determined by chemical analysis method according to SR 9934-2 1998 standard.

pH of Red Mud sludge (in water and saline suspensions – mass/volume) was determined according to the methods SR 7184-13 2001; SR EN 60746-1 2004; SR EN 60746-2 2004.

Mineral composition analysis

The mineral composition of red mud has been determined by X-rays diffraction method Bragg-Bretano Θ-2*Θ on BRUKER diffractometer XRD D8 Advance with CuKα radiation and software DIFFRAC^{PLUS} XRD Commender – BRUKER AXS, EVA12, 2006. X-ray diffraction angle varied from 4^0 to 74^0, U=40kV, I=40mA.

Determination of radioactive level

The radioactive level have been analysed by dose rate measuring method covering the range of the environmental back-ground up to the dose rate range of 200μSv·h^{-1} - 50μSv·h^{-1} on SCINTO dose rate meter instrument with Scintillation Detector (NaI) 1.5x1" for the measurement of Gamma-radiation and X-rays, Germany according to EN 55 011 (Class B); EN 61326-A1; EN 61326-1(Class B). By means of the scintillation detector it is possible to perform measurements in the energy range from approx. 25keV up.

Results and Discussion

Physical texture of Red Mud

Analysing results of physical texture of Red Mud was shown in Table 1, Box plots from Fig. 1 and Fig. 2.

The results presented in Table 1 and Fig. 1 and 2 showed that, the physical texture of Red Mud classified by an equilateral triangle iron oxide/hydroxide-sandy components-clays are: 23,2÷53,7% iron oxide/hydroxide; 34,1÷63,8% sandy components and 7,3÷34,1% clays for fresh and aged Red Mud.

Table 1. Physical texture of red mud

Mean aperture of passing/ retaining sieves x_i, (mm)	Fresh red mud				Aged red mud			
	Cumulate distribution $Q_3(x_i)$	Density distribution $q_3(x_i)$, [1/mm]	Cumulate weight (%) passing	Average particle size P80 (mm)	Cumulate distribution $Q_3(x_i)$	Density distribution $q_3(x_i)$, [1/mm]	Cumulate weight (%) passing	Average particle size P80 (mm)
0.2	0.0238	0.0031	97.76	0.038	0.1161	0.0155	93.26	0.049
0.125	0.0347	0.0056	93.80		0.1158	0.0187	85.64	
0.063	0.6316	0.2746	54.43		0.7478	0.3251	25.46	
0.040	0.3099	0.0775	39.99		0.0201	0.0050	10.03	

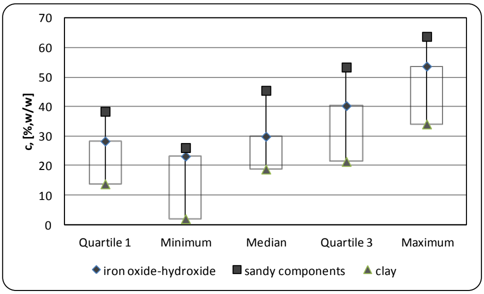

Figure 1. Box plots, physical texture of Red Mud

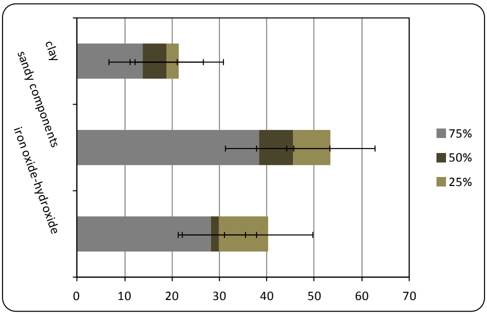

Figure 2. Box plots, physical texture of Red Mud

Particle size distribution in Red Mud showed a fine physical texture of solid sludge: > 93.80 % particles smaller than 0.125 mm size in fresh Red Mud and respectively, higher 85.64 percent of particles with lower size of 0.125 mm in aged Red Mud.

The chemical composition of the red mud samples expressed as distribution of oxides

The chemical composition of the fresh and aged red mud samples expressed as distribution of oxides is presented in Table 2. In high-grade bauxite ore, iron is commonly found as minerals such as: Hematite (α-Fe_2O_3), Goethite (α-$FeOOH$), Magnetite ($FeO.Fe_2O_3$), clay minerals, (Patterson 1986; Borra 2016).

Table 2. Concentration of oxides in Red Mud, fresh and aged samples, (%w/w)

Sample		Fe_2O_3	Al_2O_3	SiO_2	TiO_2	Na_2O	K_2O	CaO	MgO	L.O.I.
Fresh mud	RM2	37.97	16.79	5.56	3.56	2.97	0.06	4.15	0.10	28.75
	RM7	47.04	14.92	7.48	3.07	4.08	0.03	3.36	0.05	17.67
	RM8	42.89	18.19	8.28	2.77	5.39	0.02	3.30	0.05	17.47
	RM9	44.46	19.83	7.51	3.27	4.81	0.02	3.01	0.07	16.46
Aged mud	RM10	43.18	11.50	2.34	5.76	2.45	0.02	16.94	0.73	18.30
	RM11	31.02	11.26	0.84	2.80	2.84	0.04	23.10	0.41	26.52
	RM12	35.31	10.97	1.09	2.85	2.98	0.04	20.86	0.38	24.98
	RM13	35.03	22.78	1.56	5.04	4.93	0.02	9.97	0.35	17.51
	RM14	33.60	21.57	2.01	2.59	4.87	0.07	5.35	0.28	26.76

The results in Table 2 showed that in fresh Red Mud the iron content as Fe_2O_3 was 37.97÷44.46% which is 1.2 times higher than in the aged Red Mud. A similar trend is noticed for Al_2O_3 content, which in fresh red mud was in average 17.43%, slightly higher than in aged red mud. Silicon, which is the second element as percentage after oxygen in the earth crust, is commonly found in soil as Quartz (SiO_2). In Red Mud, Silicon can be found also as Quartz and clay minerals (desillication products – DSP, Cancrinite $3Na_2O·3Al_2O_3·5SiO_2·5H_2O·Na_2CO_3$, Cancrisillite/Sodalite $Na_4Al_3Si_3O_{12}·Cl$; Katoite $Ca_3Al_2(SiO_4)(OH)_8$)). The results in Table 2 showed that SiO_2 concentration in fresh red mud was in average 7.21%, higher than in aged red mud (0.84÷2.34%). Meanwhile the concentration of TiO_2 concentration was higher in the aged red mud (average concentration 3.81%), than in fresh red mud (average concentration 3.17%). These differences in the chemical compositions are related to the bauxite ore sources for the two types of technologies and corresponding emerging deposits. Analysing the results in Table 2 showed that the residual non-recoverable soda content Na_2O in fresh red mud is comparable to the values obtained at the analysis of the aged red mud. Potassium element, both present in fresh and aged Red Mud (0.02÷0.07%) was lower than K content in the Earth´s crust. The estimated average Potassium (K) concentration in the Earth´s crust is in the order of 1.84% to 2.3%, (O´Brien 2008). Most of this K is bound in primary minerals or is present in the secondary clay minerals, which largely make up the clay fraction of the soil of particle size less than 2 micrometre (O´Brien 2008). Exchangeable K^+ is electrostatically bound to the surface of clay minerals and humus substances present in Red Mud. CaO concentration in fresh red mud was 3.1÷4.15% which was much lower than the CaO content in the aged red mud (which has suffered during depositing a subsequent treatment with lime). The same behaviour was noticed for MgO content in fresh mud which was 0.5÷1% lower than MgO content in aged red mud (0.28÷0.73%).

The distribution of trace elements (heavy metals and rare elements) in indigenous Red Mud

The results of the trace analysis for heavy metals and rare elements concentration in fresh and aged Red Mud are given in Table 3.

Table 3. Concentration of heavy and rare metals in Red Mud from domestic alumina processing plants, (ppm)

Metal	Fresh Red Mud				Aged Red Mud				
	RM2	**RM7**	**RM8**	**RM9**	**RM10**	**RM11**	**RM12**	**RM13**	**RM14**
V	28	150	22	240	300	23	200	190	300
Ga	50	61	61	67	56	50	46	64	77
Cd	15	22	17	15	43	31	32	35	33
Cu	34	90	100	94	96	81	86	82	100
Cr	73	665	845	912	900	2,400	3,400	1,000	700
Ni	45	90	85	88	28	350	33	46	50
Pb	56	20	15	21	270	7,900	1,240	180	70
Zn	32	37	34	65	96	660	1,000	59	62
Ba	10	13	20	110	260	1,600	1,500	480	330
Zr	40	18	120	130	1,100	570	590	250	750
Ce	10	10	13	21	628	282	204	250	520
Gd	15	19	25	32	65	44	43	53	32
Sc	20	38	43	38	120	62	64	70	52
U	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0
Th	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0

According to the results in Table 3, both fresh and aged Red Mud contain trace concentrations of many heavy metals and rare elements (V, Ga, Cd, Cu, Cr, Ni, Gd, Sc). Heavy metals may become hazardous for environment (soil, water, air) by successive accumulation. The content of Uranium and Thorium in the investigated Red Mud was lower than the average content of Uranium (4 ppm) and Thorium (12 ppm) in the Earth´s crust (Table 3). From the broad composition of red mud, some of the heavy metals and rare elements such as: Fe, Ti, V, Ga, Sc, and Gd can be extracted as valuable economic resources.

pH of Red Mud sludge

The pH values of the investigated Red Mud samples in aqueous suspension (w/w= liquid: solid ratio =10:1) are displayed in Table 4.

Table 4. pH value of Red Mud sludge

No.	fresh Red mud	pH_{H2O}	pH_{KCl}	No.	aged Red Mud	pH_{H2O}	pH_{KCl}	No.	aged Red Mud	pH_{H2O}	pH_{KCl}
1	RM2	11.30	10.87	5	RM10	9.10	8.74	9	RM14	9.96	9.97
2	RM7	11.60	10.97	6	RM11	9.35	9.13	-	-	-	
3	RM8	11.90	10.20	7	RM12	9.48	9.02	-	-	-	
4	RM9	11.98	10.25	8	RM13	9.92	10.18	-	-	-	

The results in Table 4 showed that the pH of fresh Red Mud was 1.22% higher than for the aged Red Mud, and it decreased with the increase of the storage time. Na dominated among the soluble cations, but the concentration of soluble Na, decreased with increasing duration of storage time as a result of leaching (natural weathering). Ca was the predominant exchangeable cation in fresh Red Mud but the concentration of exchangeable Ca markedly decreased in aged Red Mud, which was dominated by exchangeable Na. This fact suggests that in the fresh red mud a higher amount of Ca is found as soluble species, while in the aged red mud which has also suffered a subsequent treatment with lime Ca was mainly converted to non-soluble species. Therefore, cation exchange capacity also decreased with increasing period of storage time. It could also be assumed that this fact could be due to a decrease in pH causing a reduction in negatively charged sites on the red mud particle. A high pH of Red Mud suspension may have a strong environmental impact on soil and surface waters.

Mineral composition of Red Mud

Mineral composition of research Red Mud (fresh and aged mud) generated from domestic alumina processing plant has been determinate by X-ray diffraction method is showed in Table 5.

Table 5. Mineral composition of Red Mud determined by X-ray diffraction method

Compound		Hematite	Titano-Hematite	Goethite	Gibbsite	Boehmite	Diaspore	Calcite	Katoite Silicatian	Amesite	Cancrinite	Cancrisilite	Hidroan-	Quartz	Anatase/	Ilmenite
Formula		Fe_2O_3	$Fe_{1.696}Ti_{0.228}O_3$	$FeO(OH)$	$Al(OH)_3$	$AlO(OH)$	$AlO(OH)$	$CaCO_3$	$Ca_3Al_2(SiO_4)(OH)_8$	$(Mg_{4.15}Al_{1.85})(Si_{2.15}Al_{1.85}O_{10})$	$Na_{0.262}(CO_3)_{0.932}Al_6Si_6O_{24}$	$Na_7(Al_5Si_7O_{24})CO_{3.3}H_2O$	$Ca_3Fe_2Si_{1.15}O_{4.6}(OH)_{7.4}$	SiO_2	TiO_2	$Fe^{+2}TiO_3$
Red Mud (fresh)	RM2	10.0	-	34.0	29.0	-	-	5.0	-	-	14.0	-	7.0	-	1.0	-
	RM7	14.8	-	25.5	8.0	3.1	-	4.8	12.2	-	-	9.2	-	7.3	1.5	13.7
	RM8	9.1	-	17.6	25.4	1.9	-	7.5	7.0	-	-	6.8	-	17.5	1.1	-
	RM9	12.1	-	16.8	35.0	2.4	-	13.1	-	-	-	7.3	-	4.7	1.5	7.1
Red Mud (aged)	RM10	-	46.8	-	5.5	-	5.2	23.4	7.0	-	12.0	-	-	-	-	-
	RM11	-	26.5	1.8	5.5	-	8.2	39.2	5.2	3.2	10.4	-	-	-	-	-
	RM12	-	27.8	3.4	2.2	-	5.7	37.6	5.7	4.9	12.7	-	-	-	-	-
	RM13	-	19.0	4.2	12.3	2.8	8.1	15.1	10.1	4.0	20.0	-	-	-	4.3	-
	RM14	-	10.0	19.9	20.4	2.2	4.8	17.2	5.9	-	9.6	-	-	7.2	2.8	-

According to Table 5, almost all the components in the investigated Red Mud component are found as microcrystal states which can be determined by X-ray diffraction patterns. The degree of crystallization of the red mud increased with increasing duration of storage. Main mineral components determined by X-ray diffraction patterns were Hematite (α-Fe_2O_3), Goethite [α-FeO(OH)], Gibbsite [γ-$Al(OH)_3$], Boehmite [γ-AlO(OH)], Calcite ($CaCO_3$), Quartz (SiO_2) and new synthetic minerals with chemical formula Cancrinite ($Na_{0.262}(CO_3)_{0.932}Al_6Si_6O_{24}$), Cancrisilite ($Na_7(Al_5Si_7O_{24})CO_{3.3}H_2O$), Katoite Sillicatian [$Ca_3Al_2(SiO_4)(OH)_8$]. Hematite can occur partially from the dehydration of the Goethite, as a phase called "proto-Hematite" in literature date, which has although the structure of the hematite but is deficient in iron and still retains hydroxyl groups ($Fe_{2-x/3}O_3H_x$). Goethite has diffraction lines slightly offset to values of smaller interplanar spacing suggesting the formation of solid solutions with Diaspore, Goethite aluminian [(Fe,AlO(OH)]. Boehmite AlO(OH), Gibbsite $Al(OH)_3$, Calcite $CaCO_3$ and Rutile/Anatase TiO_2 do not appear to form solid solutions. Cancrinite with the ideal formula $Na_6Ca_2Al_6Si_6O_{24}(CO_3)_2$ knows the great variations due to possible substitutions:

$$CaCO_3 \leftrightarrow NaHCO_3, \quad (1)$$
$$CaCO_3 \leftrightarrow Na_2CO_3 + vacancies, \quad (2)$$
$$Ca^{2+} \leftrightarrow Mg^{2+}, \text{ in a small proportion}, \quad (3)$$
$$Si^{4+} \leftrightarrow Ti^{4+}, \text{ in a small proportion}, \quad (4)$$
$$Na^{+} \leftrightarrow K^{+}, \text{ in a small proportion}, \quad (5)$$
$$Al^{3+} \leftrightarrow Fe^{3+}, \text{ in a small proportion}, \quad (6)$$

Cancrisilite with ideal formula $Na_7Al_5Si_7O_{24}(CO_3)\cdot 3H_2O$, has the same structure as Cancrinite and shows variations due to similar substitutions.
Amesite with the ideal composition $(Mg_2Al)(AlSiO_5)(OH)_4$ belongs to the kaolinite group and also accepts a series of isomorphic substitutions:

$$Mg^{2+} \leftrightarrow Fe^{2+}, \text{ in large proportions}, \quad (7)$$
$$Mg^{2+} \leftrightarrow Ca^{2+} \leftrightarrow Mn^{2+}, \text{ in small proportion}, \quad (8)$$

Measurements of radioactive level of Red Mud
Table 6 shows the radioactive level of the investigated Red Mud samples generated from domestic alumina processing plants were determined by Gamma radiation measurement method. The resulting range of annual average effective absorbed doses in air and received by adults were estimated in according to United Nations Scientific Committee on the Effects of Atomic Radiation (UNSCEAR 2000).
According to the standard for residents, the limit of the total annual effective dose of gamma radiation is 1mSv. Annual effective dose due to natural gamma fond, according to United Nations Scientific Committee on the Effects of Atomic Radiation to the General Assembly, is 0.48 mSv (UNSCEAR 2000). The results in Table 6 showed that the effective dose of gamma radiation of the fresh and aged Red Mud from domestic alumina processing plants varied in a small range from 72 $nSv\cdot h^{-1}$ to 79 $nSv\cdot h^{-1}$ and, subsequently the annual effective dose of gamma radiation was estimated in the range 0.505 - 0.554 mSv, which is fairly close to the average effective annual dose due to gamma radiation natural fond (0.48 mSv).

Table 6. Effective gamma radiation dose of Red Mud samples, comparative values with natural fond

Sample	Effective dose, [nSv·h^{-1}]	Effective annual dose(estimated), [mSv·a^{-1}]
RM2	72	0.505
RM7	78	0.547
RM8	73	0.512
RM9	72	0.505
RM10	75	0.526
RM11	76	0.533
RM12	78	0.547
RM13	79	0.554
RM14	75	0.526
Natural fond	75	0.526
Standard limit/year, [mSv·a^{-1}]	-	<1.0

Conclusions

Considering the three main categories of compounds (iron oxides-hydroxides, sands and clays) generating the textural properties of the red mud generated from both Romanian alumina processing plants, it may be inferred that these materials could be utilized to generate ceramic foams.

However, due to the fact that these residues have a high free alkalinity, a pre-treatment process for lowering the Na soluble content level is imperiously necessary before their utilization in ceramic foams synthesis.

Taking into account that both types of red mud contain insignificant amounts of radioactive elements their further processing does not present environmental risks.

Another direction of red mud valorisation could be the recovery of Fe, Ti, V, Ga, Sc, Gd by extraction. This method would be important especially in the case of Ga, Sc, Gd, which are critical metals with specific properties and rarely encountered in natural ores.

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