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GEOPOLYMERS OBTAINED WITH RED MUD FROM ALUMINA MANUFACTURING

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ABSTRACT

Getting reusable aluminosilicate materials, based on the geopolymerization reaction mechanism of some physical and mechanical material particular, has been imposed in the recent years in building materials.

This paper presents the physico-chemical and structural characteristics of the red mud from alumina (Al_2O_3) manufacturing through the Bayer process, material that can be investigated in order to obtain geopolymeric materials. Geopolymers made from red mud are eco- friendly on the one hand because their synthesis reuses waste from industries as raw materials, and on the other hand, because the conditions through which they can be obtained are more economics and cheap.

KEYWORDS: geopolymers, residue, red mud, construction materials

1. Introduction

Considering the problem of pollution in the entire planet, the demand for cement used in construction of the momentum buildings, getting Portland cement and other types of building materials using concrete, are a source of pollution with a strong impact on the environment. Both in terms of technology and production of the raw material, large-scale production of cement, namely a building material by conventional technologies, poses a threat. Therefore, to minimize the negative effect was taken into account obtaining other materials which fulfill the same role at the same time as environmentally friendly as the technology of production and layout while.

After analyzing some specialty papers, it was concluded that the creation of waste by the geopolimerization process, a building material with a minimum of cost of raw material and production technology is an intelligent and effective reuses of these types of waste. This article deals with the process of getting geopolymer cement with solid waste as raw material (clay, ash) or semi-solids such as sludge, in this case, the red sludge resulting from the Bayer process, the basic requirement for achieving this material being a high content of crystalline aluminosilicate phase.

In the cement industry, in an attempt to protect the environment was already partially replaced the amount of solid aggregate, non-renewable raw materials with ash from power plants that run on fossil fuels [1-3], or slag from the manufacture of iron [4-6] with very good results, so that the properties of the obtained geopolymers may be comparable to that of Portland cement. Being highly successful in this regard, researchers attempts were not limited to these conditions but we tried to replace the unit with other solid waste. In this sense, changing the source of aluminosilicate was achieved with red mud, but because there are no known conditions that ensure its maximum properties, thorough research are needed.

1.1. The concept of geopolimer

The term of geopolimer assigned as a building material was first introduced in the academic language by the French researcher J. Davidovits in 1979, [7] and was defined as a "polymeric mineral made by geochemistry or geosyntesis "aluminosilicate materials dissolved in the basic medium. In this context, the term refers to a inorganic three-dimensional geopolymeric structure with a high content of aluminosilicates which is structurally similar with natural aluminosilicates, respectively zeolites. The general formula for a geopolymer is: [8] $\cdot\mathbf{Mn[-(SiO_2)_z\text{-}AlO_2]_n\cdot wH_2O}$ in which **n** is the degree of polycondensation or polymerization; **z** is the ratio Si/Al and may be 1, 2 or 3; **M** is a cation such as Na^+ or K^+, and **w** is the number of water molecules in the geopolymeric structure. The inorganic polymeric material formed by SiO_4 and AlO_4 tetrahedral units is arranged in a three-dimensional structure and the link between these units is balanced by alkali ions.

Geopolimerization reaction mechanism involves the following steps [9]: the dissolution of aluminosilicates in alkaline solution refocuses ions in the network and generates the formation of geopolymer. Figure 1 shows the block diagram for obtaining a geopolymer.

Fig. 1. *The block diagram for obtaining a geopolymer*

1.2. Geopolymers made from recyclable materials

In the recent years, teams of researchers have highlighted the importance of reuse of industrial by-products that are generated in large amounts as in the case of red mud.

Researcher Liu X.and his team [10] analyzed the red mud samples with a length of three years, resulting from the calcination of bauxite ores. The samples were previously subjected to drying in an oven at 100 °C they comminuted to obtain a powder which was subjected to calcination in an electric furnace at a temperature between 400 °C and 900 °C. The powder was obtained by ball milling with an average diameter of about 23 µm. They mixed a quantity of 50% calcined red mud with 45% clinker and 5% gypsum and obtained a mortar which has undergone testing according to current standards, and the results are satisfactory and comparable to those of Portland cement.

Geopolymers obtained in laboratory by researcher A. Van Riessen *et al.* [11] have been made in order to reuse the solution resulting from the Bayer process alumina production. They compared the geopolymeric cement paste that was obtained with pure NaOH solution and the solution of synthetic red mud processed (Bayer solution) in order to determine the effectiveness of the Bayer alumina source solution and at the same time, the quality of the cement alkaline source, respectively of a resulting slurry. There have been geopolymeric pulp samples in which the source of alkaline substituted, NaOH at various concentrations, with the following chemical composition: Al_2O_3 (15%), Na_2O (24%) and H_2O (61%). After analyzing the different methods, they concluded that the Bayer liquore did not influence the solution properties of cement paste made with Bayer solution compared to pure NaOH solution and the solution made synthetically.

To obtain a binder for geopolimeric materilas of construction debris mixed with ash class F, a team of researchers led by Saeed Ahmari [12] analyzed the effect of mineralogy to specify the amount of waste, the concentration of the alkaline solution and the amount of calcium effect on the binder composition. Since in the structure of geopolymer, besides the quantity of calcium hydrate and silica hydrate, there are calcium and aluminum, it has been shown that increasing the amount of calcium compound based on the base material may improve the mechanical properties of geopolymer. In his work, L. Reig *et al.* [13] have used waste (red brick) from the demolition of buildings, an impressive amount that can be reused to make a cement paste and mortar. In this case, alkali activators used were sodium hydroxide and sodium silicate. It has been found that a mixture of the 45/6.0/1.6 mole ratio is one that has shown the best mechanical properties. The samples obtained were dried at a working temperature of 65 °C for 7 days, and were used to compare the geopolimeryzation waste material resulting from the initial to the difference between the initial properties and the subsequently obtained properties. Thus it can be stated that physical characterizations of geopolymers obtained from different sources of alkali activated aluminosilicate in different quantities and chemical and mineralogical characterization provides valuable information on how to obtain the best mechanical properties not only thereof.

Thus, it has been found in many studies that in order to obtain a diffusion of the aluminate and silicate waste from the original source, it is necessary that the source has a high degree of fineness, the reactivity of the mixture increases in proportion to changes in particle size, [14] activation of the concentration of the solution of the formulation [15, 16] and the amount of water in the system. Also, knowing the exact chemical composition is advantageous for adjusting the recipe to get those elements and chemical species without which you can not achieve cementitious material properties required for use in construction. Mineralogical composition provides information about existing minerals and their influence on the structure of geopolymers prescription.

2. Red mud

The use of red mud filler material as a material for obtaining various geopolymeric materials is comparable to the quality of the materials obtained by extraction from ore an easy process, both from the point of view of therapy and financial processing. Because aluminum demand is very high in Romania and elsewhere in the world by producing one ton of aluminum resulting approximately two tons of wet waste, ie red mud, which amount exceeds its reuse. [17]. Therefore, it is imperative that the waste to be used in other directions, thus solving some of the issues raised after storage in the open landfill (transport of particulate matter infiltration of substances into groundwater, etc.).

Red mud is a semi-solid waste produced during the extraction of alumina from bauxite ore by the Bayer process. They use several terms that define the same structure ie bauxite waste; tailings resulting from the Bayer process; or tailings resulting from the processing of bauxite. The investigations concluded that the waste due to the high content of aluminosilicate is a source of raw material that can replace some, or all solid units that go into a building material.

Generation of red mud in the production of alumina takes place in the first stages by means of acid or alkaline leaching of the sinter ore or bauxite, or other materials with a high content of alumina, which are generally in the form of aluminosilicates, such as nefelinele, oil shale, subjected to physico-chemical process for extracting aluminum hydroxide or other aluminum compounds which ultimately will result in ignition alumina [18]. In Figure 2 is shown schematically how it is obtained that the alumina and red mud from bauxite by the Bayer process. Tailings recovered from the filtering process called red mud are collected, washed and directed to storage area which can be: a lagoon, a large area, disused mines or landfills.

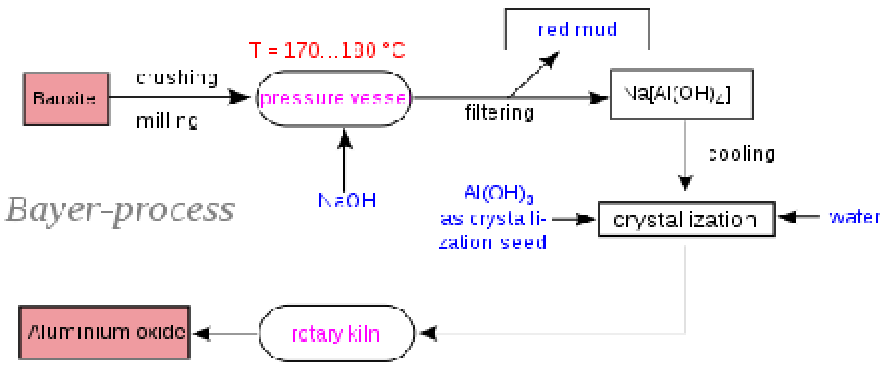

***Fig. 2.** The Bayer process* [19]

Regardless of ore with a high aluminum content processing and extraction technology used alumina, red mud result raises environmental issues locally and globally. Re-use of the red mud is based on the following: the origin of the ore, the treatment, the water content, the amount of radioactive metal slurry composition, the alkalinity of the particle size, mineralogy. There are several technologies for obtaining of red mud geopolymers, each depending on both the amount of red mud is used and the future direction of the use thereof. In construction, the red mud is used in large quantities together with other wastes, in the form of binder [20-22], mortar, [23], reinforced concrete [24, 25] concrete [26, 27], amendment of pollutants in solid and liquid phases [28], building bricks [29], bricks [30] aggregate in building materials [31], or recipes for the manufacture of ceramics [32, 33], glazes [34], in the extraction of heavy metals [35, 36], as an adsorbent for the removal of H_2S from industrial emissions [37]. In Figure 3 are shown the potential applications of red mud.

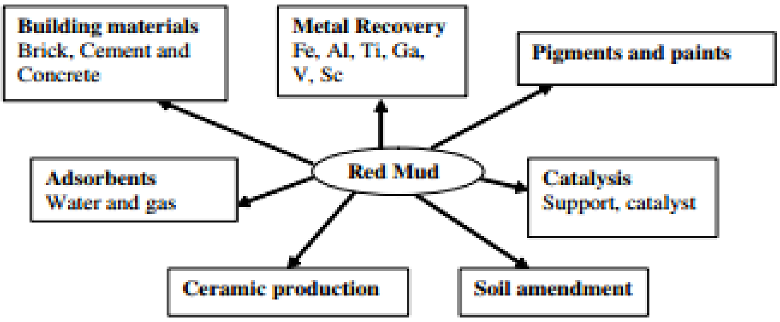

***Fig. 3.** Potential applications of red mud* [38]

3. Physico-chemical and structural properties of red mud

3.1. Physico-chemical properties

Bauxite tailings semi-solid material has a brick-red color due to the high amount of iron oxide, iron sulfate, silicoaluminates, titanium dioxide, etc. The red mud particle size is characteristic of each type of ore bauxite and varies between limits below 100 mm and 200 micrometers. Red mud is a mixture of mineral and chemical composition which depends on the bauxite ore (diaspore, boehmite, gibbsitic) containing alumina, in which the solid phase is about 20% to 80% of the total quantity of sludge. Minerals with the highest weight are, in order, the following: Hematite (α-Fe_2O_3), Goethite (α-FeOOH), Magnetite (Fe_3O_4) as iron oxides with an average of 40.9%; followed by the Boehmite (AlOOH-γ), Gibbs, (γ-Al $(OH)_3$), Diaspora (γ-AlOOH), aluminum oxides as an average of 16.3%. The silicon oxide content averages 9.6%, respectively: Sodalite ($Na_6[Al_6Si_6O_{24}]$2NaOH, Na_2SO_4]), Cancrinite ($Na_6[Al_6Si_6O_{24}]2[CaCO_3]$-$O[H_2O]$), Quartz ($SiO_2$) and other, Illite, Muscovite. Titanium oxides are embedded in smaller quantities, 8.8% minerals form: Rutile (TiO_2) Anatase (TiO_2), Perovskite ($CaTi^{iv}O_3$), Ilmenite, ($Ti^{IV}Fe^{ii}O_3$). Other minerals are small amounts of oxides from sodium and calcium. Also found in traces the next: K, Cr, V, Ni, Ba, Cu, Mn, Pb, and Zn [39].

3.2. Materials and methods

Red mud used in this study was made from bauxite manufacturing by the Bayer process, ALUM Tulcea, namely dried red mud dumped in the related plant. Firstly raw red mud dried was sieved using a set of sieves grain of 125 µm. For the experiment were weighted 3.5 g of red mud grain of 125 µm with an analytical balance. A sample was subjected to calcination in a furnace-type Lenton Thermal Design at a calcination temperature of 600 °C, at a heating rate of 10 °C/minute, a hold time of 45 minutes. Following the calcination process, resulted 2.1 g of red mud, which is a weight loss by eliminating natural water of about 60%.

3.3. Analytical methods

Uncalcined an calcined samples, were subjected to thermogravimetric analysis (TGA) and (DTG), differential scanning calorimetry analysis (DSC), and analysis to determine the structure and chemical composition by electron microscopy (SEM).

It is very important to know the thermal behavior of red mud because according to this process can specify a future use of its as a construction binder or other geopolimeric type material.

4. Experimental data and literature

The reaction temperature is very important for obtain a geopolimer from any source of aluminosilicates materials, the red mud has been characterized from the point of view of the thermal effects, physical and chemical processes taking place in state of uncalcined sample and calcined sample at 600 °C. Differential scanning calorimetry analyzes were performed in the laboratory of UDJG nanostructures, the analyzer Q20 found in the laboratory, which is provided with a platinum crucible, the temperature is in the range of 20 °C-400 °C, with a heating rate of 10 °/min in an inert atmosphere (nitrogen). The sample was weighed and then subjected to analysis.The resulting curves are shown in Figure 4a and in Figure 4b.

The DSC technique, differential scanning calorimetry curves that determined the changes of heat and mass due to physical and chemical degradation of red mud compounds entering the structures analyzed. With this technique were evaluated faster interactions between components based on changes of endothermic and exothermic peaks of the device. The literature mentions the main minerals contained in red mud [39], and temperatures at which phase transformations occur [40]. The analyses made the following considerations: in the temperature range of 40 °C to a maximum temperature of 68 °C it appears to be little loss of water due to the hygroscopic nature of the calcined and uncalcined sample. Also, in the temperature range of 230 °C there is another peak which is explained by loss of chemically bound water of the peak maximum at around 279 °C or transformation of the silica into tridymite lower Cristobalite (metastable phase) at around 275 °C which further will change under the temperature action in higher Cristobalite [40]. Other exothermic changes after analyzing samples are the reduced of magnetite to iron and with CO and forming CO_2 with a maximum peak at 321 °C [41]. For the corresponding temperature peak of 380 °C, the appearance of the DSC curve is assigned to the formation of sodium carbonate, Na_2CO_3, after the interaction between NaOH and CO_2.

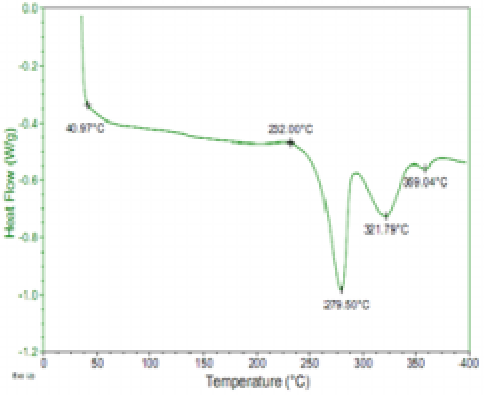

Fig. 4a. *DSC curve of a non-calcined sample*

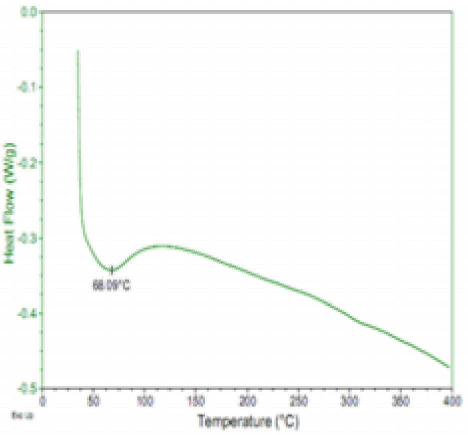

Fig.4b. *DSC curve of a calcined sample*

Thermal gravimetric analysis curves were made with thermogravimetric analyzer, Q5000 IR with air purified 5.0, under an inert atmosphere of nitrogen, with 12.5 mg of the uncalcined sample weight and 13.33 mg of calcined sample.

The thermogravimetric curves are shown in Fig. 5 a) and Fig. 5 b), and they highlight the changes that occur as a result of phase transformation or formation or dissociation of chemical combinations in the mass of the sample.

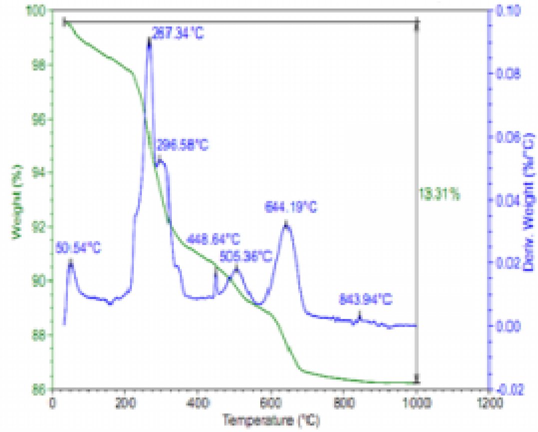

Fig. 5a. *TGA, DTG curves of a non-calcined sample*

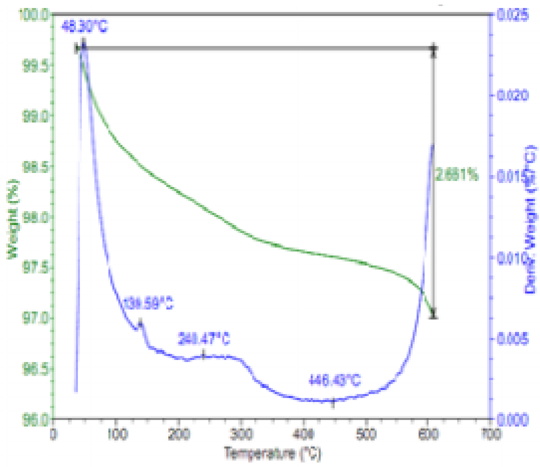

Fig. 5b. *TG , DTG curves of a calcined sample*

As the temperature in sample increases linearly with time, mass loss occurring due to physical or chemical processes in red mud as a result of temperature increase and the decrease of mass is initially 2.66% for the calcined sample and 10.96% for the uncalcined sample. As observed in the DSC curve and the DTG, there is a little peak around the temperature of 50 °C which demonstrates the removal of water of hydration and water of crystallisation at temperatures above 170 °C with a maximum peak of 267 °C in DTG curve. And for the thermogravimetric curves occurring specific peaks of the phase transformation of silica from tridymite into inferior Cristobalite, but in this case, phase transformation is evidenced by the temperature about of 296 °C [40] and also the conversion of the magnetite into Fe with the releasing of CO_2 [41] in the temperature about of 300 °C. It is noted that the peaks which appear at temperatures superior to 400 °C, temperatures to which other already started process occurs or are continued if necessary (such as cristobalite which has continued its transformation up to temperatures approaching of 1000 °C) [40]. Thus, at temperatures above 450 °C there is a bit that can be attributed to the formation of sodium carbonate (Na_2CO_3). At temperatures above 550 °C begins the calcination of aluminum hydroxide into γAl_2O_3. Also in the temperature range of 600 °C can take place the decomposition reaction of calcium carbonate ($CaCO_3$) from the red mud.

Thus, is resulted from the analysis of diagrams, that many of exothermic reactions presented on scanning calorimetry curves and on the thermogravimetric analysis curves are due, in general, to the chemical and physical losses of bound water; phase transformations or other chemical processes between the red mud components, or to the processes of decomposition of the components.

4.1. SEM analysis

Scanning microscopic structure analyses, SEM were made in the laboratory UDJG, the type QUANTA analyzer 200, which is provided with an EDX analyzer type. These tests were carried out on samples annealed at 600 °C and non-calcined samples in order to emphasize the structural changes that occur as a result of the heat treatment applied.

In Figures 6a and b are shown SEM images of the non-calcined samples with particle size of 12.5 µm in the sizes of 500x, 5000x and Fig. 7a or b are shown SEM images of the sample annealed and their respective sizes 500x and 5000x.

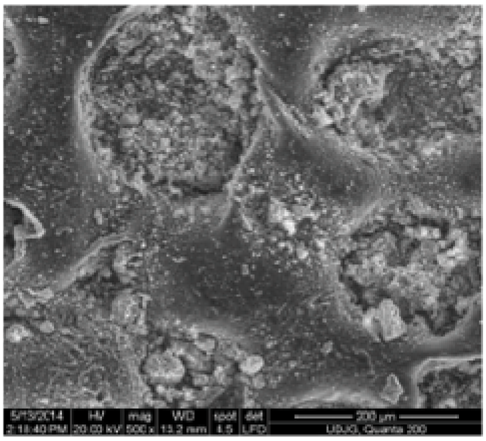

Fig. 6a. *SEM image of the non-calcined sample 500x*

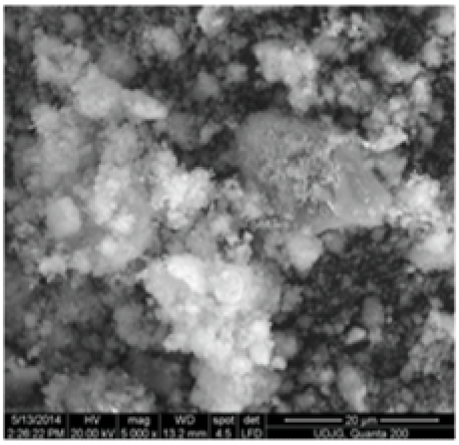

Fig. 6b. *SEM image of non-calcined sample 5000x*

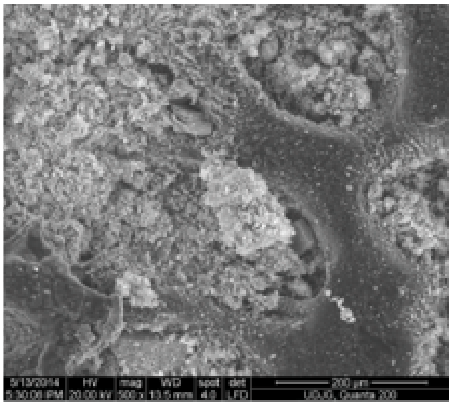

Fig. 7a. *SEM image of the calcined sample 500x*

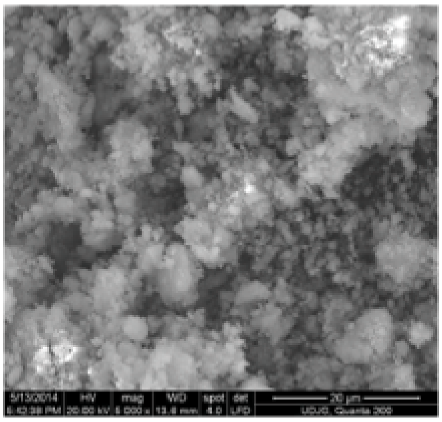

Fig.7 b. *SEM image of calcined sample 5000x*

Figures 8a and 8b shown the non-calcined sample spectra and calcined sample, respectively, and Fig. 9a and 9b maps the layout elements of an non-calcined and calcined samples.

In Table 1 and Table 2 are shown the elemental composition of uncalcined and respectively calcined sample.

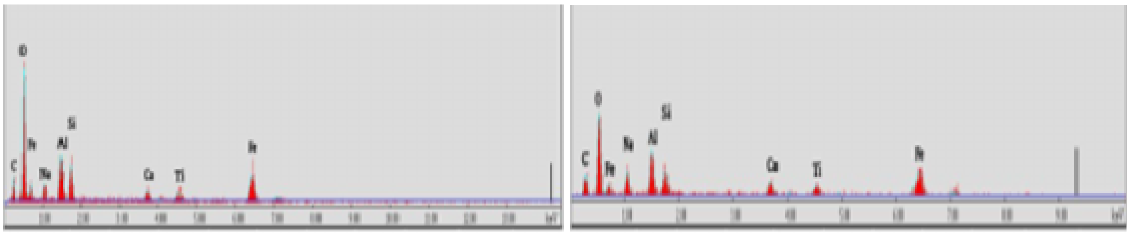

Fig. 8. *a) Non-calcined sample spectrum; b) Calcined sample spectrum*

Tabel 1. *The elemental composition of non-calcined sample*

Element	Wt%	At%
C	21.38	33.30
O	40.47	47.33
Na	3.97	3.23
Al	6.66	4.62
Si	5.56	3.70
Ca	2.43	1.13
Ti	2.58	1.01
Fe	16.96	5.68

Tabel 2. *The elemental composition of a calcined sample*

Element	Wt%	At%
C	21.10	34.44
O	33.03	40.49
Na	6.78	5.78
Al	8.62	6.26
Si	4.76	3.32
Ca	3.28	1.61
Ti	3.78	1.55
Fe	18.65	6.55

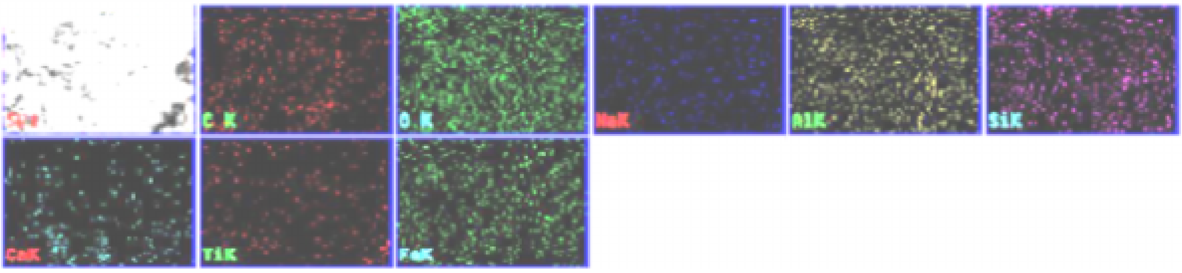

Fig. 9a. *The distribution map of the main chemical elements in the non-calcined sample, 5000 X*

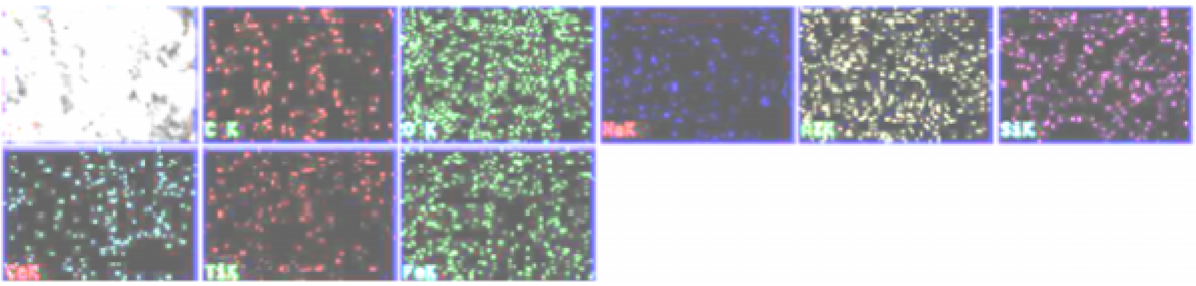

Fig. 9b. *The distribution map of the main chemical elements in the calcined sample, 5000 X*

From the analysis of scanned images by scanning electron microscopy it was observed that the metallographic structure of uncalcined sample reveals fewer crystallized phases compared to the sample that was subject to the calcination treatment, which shows that after calcination took place and transformations phase, passage of silica from tridymite into inferior Cristobalite, or of the amorphous phases into crystalline phases.

5. Conclusions

Geopolymers are inorganic polymers, alkali-activated aluminosilicates, from different materials characterized by a high content of oxides of aluminum, silicon, calcium, and their complexes, which are employed in various industrial fields. Red mud is one of these sources of aluminosilicate (among other materials) used to obtain geopolymers.

Due to the great demand for aluminum and the very high amount of red mud generated , the demands for building materials, the solution to reuse of this waste is the best way to solve both environmental issues and socio-economic issues related to obtaining cheap and durable buildings materials.

In order to know the properties of a material it is important to know the chemical composition, physical properties, namely the structure of the material. Therefore in this study were performed chemical analysis, thermal, and structural for the uncalcined samples and calcined samples. From thermal and structural analysis has emerged a picture of the behavior of the temperature variation of the red mud. Thus, by analyzing DSC, DTG and TGA was observed a mass loss due to removal of physically bound water or crystallization water and other chemical processes or specific material phase transformations.

The SEM analysis showed a difference in structure and surface morphology of the uncalcined sample compared to the calcined sample, difference is explained by the fact that when the heat treatment is applied, crystalline phases occurring are important in the reuse of the red sludge for the various materials used in construction.

Acknowledgements

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