# The Influence of the Properties of the Material Used for Obtaining Geopolymers on Their Structure and Compressive Strength

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*This article presents the influence of the properties of the materials in the geopolymeric mixture, ground granulated blast furnace slag (GGBFS) + wheat straw ash (WSA) + uncalcined red mud (RMu), and ground granulated blast furnace slag + wheat straw ash + calcined red mud (RMc), over the microstructure and mechanical properties of the synthesised geopolymers. The activation solutions used were a NaOH solution with 8M concentration, and a solution realised from 50%wt NaOH and 50%wt $Na_2SiO_3$. The samples were analysed: from the microstructural point of view through SEM microscopy; the chemical composition was determined through EDX analysis; and the compressive strength tests was done for samples tested at 7 and 28 days, respectively. The SEM micrographies of the geopolymers have highlighted a complex structure and an variable compressive strength. Compressive strength varied from 24 MPa in the case of the same recipe obtained from 70% of GGBFS + 25% WSA +5% RMu, alkaline activated with NaOH 8M (7 days testing) to 85 MPa in the case of the recipe but replacing RMu with RMc with calcined red mud, alkaline activated with the 50%wt NaOH and 50%wt $Na_2SiO_3$ solution (28 days testing). This variation in the sense of the rise in compressive strength can be attributed to the difference in reactivity of the materials used in the recipes, the curing period, the geopolymers structure, and the presence of a lower or higher rate of pores, as well as the alkalinity and the nature of the activation solutions used.*

*Keywords: geopolymers, ground granulated blast furnace slag, microstructure, compressive strength*

In recent years, the rise in waste production has been associated to a number of environmental issues and so the necessity of sustainable development regarding the management of waste has become a main priority. Recycling has always been a positive approach of solving the issue of waste, thus reducind pollution and protecting natural resources. In this context, geopolymeric types binder materials represent a new class of binders materials which can be synthesised from a variety of industrial by-products such as ground granulated blast furnace slag [1-5], ash [6-10], bauxite red mud [11,12] or certain recycled waste [13-15]. The term „geopolymer” has been associated, by French researcher, J. Davidovits, in 1972 [16], to a new class of binding materials formed with industrial by- products alkaline activated. The process through which geopolymerisation is achieved has been divided into three phases by H. Wang [17]. These phases are: the dissolution of silico-aluminates from different materials which promote the formation of silica and aluminum hydrates, followed by the rearrangement of the chemichal formed species and then the polycondensation/gelling, thus forming the tridimensional network of the silica-aluminous gel under the action of the alkaline activation solution used.

Geopolymers are silico-aluminous mineral materials [18] generally amorphous in structure, which can be obtained at atmospheric pressure and a temperature of about 100 °C [19-21]. This type of material presents low microporosity because in the geopolymeric type binder system there is a very smoll amount of water absorbed due to the amorphous or semicristaline structure resulting from synthesing, which causes high values of compression strength. Through recent studies [22, 23], it was revealed that the raw materials of the synthesized geopolymers plays a significant role in the geopolymerisation reactions and largely influences the mechanical properties and structure of the final product. The alkaline activators generally used in the synthesing of a binder geopolymer type are caustic alkalis or mineral alkalis. Their classification according to their chemical composition was designed by Glukhovsky [24]. Among all known alkali activators, NaOH and $Na_2SiO_3$ were selected for this study because they are available on the market at a low cost and it has been demonstrated throughout previous studies [25, 26] that by alkaline activating of some industrial by-products have been obtained very good results.

In this study it was investigated the possibility of using ground granulated blast-furnace slag, wheat straw ash, uncalcined red mud, and calcined red mud, resulted from Bayer process, in order to obtain construction material through the geopolymerisation. Also, it was determined the optimal combination of materials, which would allow the use of uncalcined or calcined red mud at 600°C, in mixture with ground granulated blast-furnace slag and wheat straw ash and an alkaline activator, that would lead to a geopolymeric binder with specific physical, chemical, and mechanical properties.

## Experimental part

### *Solid materials preparation*

The development of a geopolymer implies a series of operations that leads to obtaining them. To prepare the raw materials some operation are need to reduce the volume through grinding, sieving, and in particular for red mud water removal through calcination. Grinding of ground granulated blast furnace slag was achieved using ball mill PM 400 type, this grinding mill has zirconium oxide containers, with 5 mm diameter balls from the same material. The ratio between the quantity of slag and the quantity of balls was 8:1, and the grinding speed was 200 rpm for 4h. To avoid the powder adhering to the balls surface a quantity of 1 mL of ethanol was added. The

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powder obtained was preserved in airtight containers of polypropylene. Reducing the particle size of red mud and straw wheat ash was done with the Retsch mill rotor type ZM100 which has a system of grinding and sieving incorporated. The powder obtained was brought to the average diameter of 80μm. Red mud calcination was carried out at a working temperature of 600°C in the calcination oven NABERTHERM type, for 5h with a heating rate of 10°/min and cooling was done in the same time with the oven.

*Solution preparation for the alkaline activation*

The alkaline solutions used in the study were prepared in the laboratory, 24h before being used, to ensure good mixture and dissolution of chemical species. The maturation and the cooling of NaOH (98% purity) solution has been carried out in the laboratory at a temperature of 20°C and approximately 50% humidity. The solution obtained by mixing of sodium silicate ($Na_2SiO_3$) and sodium hydroxide solution (NaOH) in 50 wt%, was kept in an oven at 60°C in order to avoid the separation of sodium silicate. Before using of the solution of NaOH/ $Na_2SiO_3$ ,(50/50 wt%) it has been left to cool down at the room temperature.

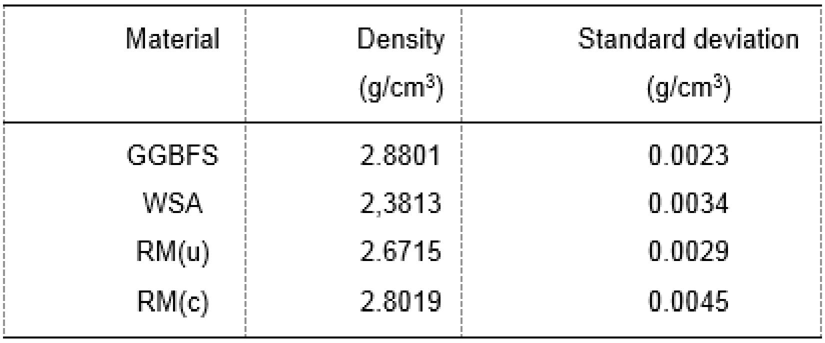
*Experimental procedure*

Preparation of the samples was done as follows: in the first instance the solid phase (s) was mixed with the liquid phase (l) at a ratio of 0.4 (s/l) until a compact paste was obtained. The paste was then placed into plastic cubic shaped molds measuring 2.5 x 2.5 x 2 mm fitted with a lid. Filled shapes were sat on a vibration table for 5 min in order to remove the residual air and to produce a compact structure. For the samples analysed with SEM was required an supplementary preparation, each sample has been coated with a conductive layer of Au-Pt to facilitate analysis. To ensure reproducibility of the data each sample was performed in triplicate.The samples were stored at room temperature until the time of analysis.

## Results and discussions

The physic properties of raw materials analised were: density, the average diameter of the particles and the surface area in order to observe the chemical, morphological and structural characteristics. Thus, ground granulated blast furnace slag used in the study had a density of 2.88 g/cm$^3$, a specific area of 4.2653 g/cm$^2$, and an average diameter of particles of 45.2 mm. Wheat straw ash had a density of 2.38 g/cm$^3$ and a specific area of 4.1187 g/cm$^2$, with an average particle size of 22.06 ìm. Red mud, uncalcined had a density of 2.77 g/cm$^3$, a specific area of 7.3087 g/cm$^2$, and an average size particles after calcination of 14.86 ìm, and the density increased to 2.80 g/cm$^2$, the surface area was 7.3679 g/cm$^3$, and the average size of particles by 10.28 ìm. To determine the density of the materials used in the study, the Micromeritics Accupyc 1330 was used, the values recorded from the analysis and standard deviations are shown in table 1.

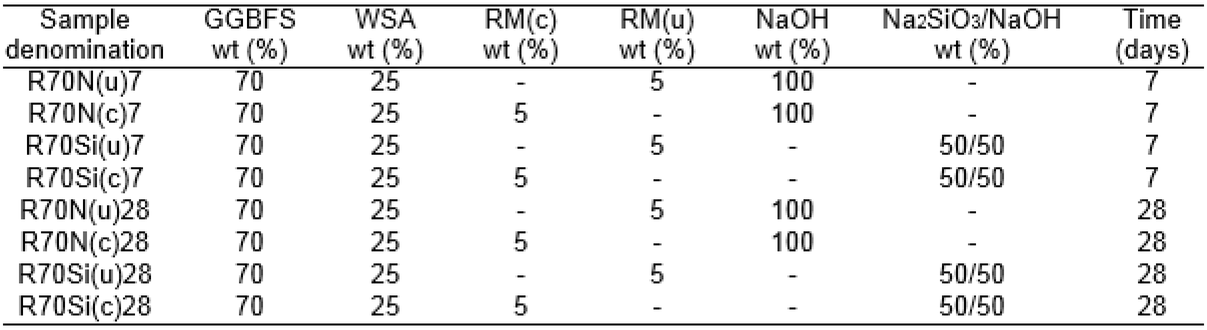
In the case of red mud, the difference between the particle sizes of the calcined and uncalcined is due loss of water related to various physical and chemical reactions occurring during calcination [27]. The assessment of average particle sizes was needed because geo-polymerization as a process is influenced directly, as the particle sizes are smaller then reactivity of the material increases, which is confirmed by the high level of compressive strength [28-30]. In the table 2 the sample denomination, raw materials and alkaline activators quantity and time of the analysis was noted. Thus, by symbol R70N(u)7 was noted that the sample was made up of 70% of ground granulated blast-furnace slag + 25% wheat straw ash + 5% uncalcined red mud and alkaline activated with sodium hydroxide solution with 8M concentration. The samples were stored under laboratory conditions at ambient temperature for a period of 7 days, after which the analysis were carried out. With the symbol R70Si(c)28, for example, was noted that a test has been carried out with 70% ground granulated blast furnace slag + 25% wheat straw ash + 5% calcined red mud, which was enabled by an alkaline solution made by mixing the solution of NaOH 8M concentration with sodium silicate solution in 50 wt%. In addition, samples were stored at ambient temperature for a period of 28 days, until the time of the tests.



| Material | Density (g/cm$^3$) | Standard deviation (g/cm$^3$) |
|---|---|---|
| GGBFS | 2.8801 | 0.0023 |
| WSA | 2,3813 | 0.0034 |
| RM(u) | 2.6715 | 0.0029 |
| RM(c) | 2.8019 | 0.0045 |

**Table 1**
BASE MATERIAL DENSITY VALUES

**Table 2**
SAMPLE DENOMINATION, RAW MATERIAL AND ALKALINE ACTIVATOR QUANTITY AND TIME OF THE TESTS



| Sample denomination | GGBFS wt (%) | WSA wt (%) | RM(c) wt (%) | RM(u) wt (%) | NaOH wt (%) | $Na_2SiO_3$/NaOH wt (%) | Time (days) |
|---|---|---|---|---|---|---|---|
| R70N(u)7 | 70 | 25 | - | 5 | 100 | - | 7 |
| R70N(c)7 | 70 | 25 | 5 | - | 100 | - | 7 |
| R70Si(u)7 | 70 | 25 | - | 5 | - | 50/50 | 7 |
| R70Si(c)7 | 70 | 25 | 5 | - | - | 50/50 | 7 |
| R70N(u)28 | 70 | 25 | - | 5 | 100 | - | 28 |
| R70N(c)28 | 70 | 25 | 5 | - | 100 | - | 28 |
| R70Si(u)28 | 70 | 25 | - | 5 | - | 50/50 | 28 |
| R70Si(c)28 | 70 | 25 | 5 | - | - | 50/50 | 28 |

*FTIR analysis*

Analysis with Fourier transforms infrared spectroscopy aimed to establish the chemical structure of the synthesized geopolymeric materials along with the assessment of the chemical composition analysis by EDX. FTIR spectra have been used to determine the changes in the structure of the obtained samples, using different activation solutions. The spectra of the analysed samples were marked and shown in figure 1 where the vibrations intensity corresponding to certain chemicals species were observed. Vibration intensity in the case of uncalcined red mud samples (R70N(u)7, R70Si(u)7, R70N(u)28, and R70Si(u) 28 of around 3200 $cm^{-1}$ and 3650 $cm^{-1}$ was assigned to the stretching vibrations of the H-OH bonds which are assumed to have been caused by hydrate formations in the system [31]. Vibrations due to links between H-OH were also observed at approximately 1650 $cm^{-1}$ for all spectra. In case of uncalcined red mud samples the vibration was more likely due to the presence of water or presence of $Al(OH)_4$ [32]. Absorption band at around 1460 $cm^{-1}$ is present in all spectra of the samples, and was associated with carbonates in the system, the way the O-C-O of carbonates links spread [33]. This absorbtion band iti is more highlighted in the sample noted R70N(c)7, which implies a high quantitie of carbonates and lower values of compressive strength. Also, a difference in the vibration absorbtion waves around values 1390 $cm^{-1}$ and 980 $cm^{-1}$ was noticed, which was associated with the chemical composition of the binder type geopolymeric material, present in the studied samples R70Si(c)7 and R70N(c)28 [34]. Absorption band around 690 $cm^{-1}$ present in all samples and especially in the R70Si(c)28 was due to the stretching vibration of the link group of a tethraedal T (where T is Al or Si) [35]. The peacks around 800 $cm^{-1}$ in all samples may indicate the presence of small quantities of Si-O-Si stretching in quartz [36].

The presence a sharp peack of around 890 $cm^{-1}$ can be explained by the transformation of the $Al^{3+}$ octahedral structure in tetrehedral structure due to the influence of the alkaline activation solution used [38]. Also the distinct peacks between 450 $cm^{-1}$ and 480 $cm^{-1}$ can be due to bending mode of the O-Si-O in $SiO_4$ tethraedra [37].

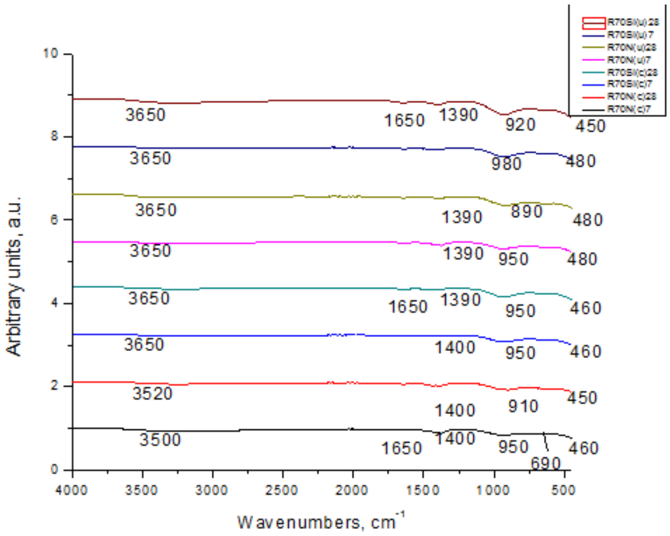


Fig.1. FT-IR spectra for studied samples

*Compressive strength tests*

The values resulted from the testing of geopolymers by compressive strength are given in table 3, and are in line with the SEM images described in the next section. The testing results shows that geopolymer noted sample R70N(u)7 obtained the smolest value of 24 MPa, and for 28 day of testing, the compressive strength test result had the value of 28 MPa. The highest values were obtained for sample geopolymer noted R70Si(c)7 with 68 MPa at 7 days of testing, and 85 MPa at 28 days of testing.

**Table 3**

COMPRESSIVE STRENGTH VALUES

| Sample ID | Compressive strength values (MPa) |
|---|---|
| R70N(u)7 | 24 |
| R70N(c)7 | 30 |
| R70Si(u)7 | 51 |
| R70Si(c)7 | 68 |
| R70N(u)28 | 28 |
| R70N(c)28 | 33 |
| R70Si(u)28 | 79 |
| R70Si(c)28 | 85 |

The values registered after the compressive strength tests are varied due to: the different curing time, the utilization of uncalcined or calcined red mud into geopolymer synthesis and different alkaline activating solutions used. Thus, regarding the different curing period it influence the compressive strength in the possitive way because the characteristic geopolymers chemical bonds are reorganising and consolidating in time, fact that was observed in other studies [38,39]. Another factor that positively influenced the compressive strength of synthetised geopolymers was the calcination of red mud. Thus, the physical bonded water was eliminated [36], and new phases are formed that facilitated the geopolymers formation [37]. Also, another factor taht positively influenced the compressive strenght was the use of the solution made by Na-silicate and sodium hydroxide in equal proportions. An important amount of silicon was added, which was the key element in the geopolymers tridimensional structure formation [38]. Thus, a change in the silicon quantity determined the rising of geopolymers sample compressive strength values, like in the twoo studied recipes. In sample noted R70N(u)7 alkaline activated with NaOH, the Si quantity from the system is unchanged, and for the sample noted R70Si(u)7, in which the Si is added from alkaline activating solution used in geopolymer synthesis. Silicone which is contained in sodium silicate, plays an important role because it is the one that initiates the geopolymerisation reactions which allows the rapid and complete dissolution of the material. A higher concentration of silica leads to formation of silica species with a complex chemical structure which allows the three-dimensional polymeric network to grow. The silicate solution favors polycondensation, although a higher initial concentration has cracks on the surface of the samples [43].

*SEM and EDX analysis*

The micrographies of the synthesized geopolymer samples which appear in Figures (2÷9) were done by electron microscopy scaning (SEM), and the elementary chemical composition corresponding to these samples shown in table 4 was determined by the EDX analysis. The microstructures of alkaline activated samples with NaOH (fig. 2, 4, 6 and 8) were observed to have a rougher surface, showing some agglomeration of particles, with holes caused by water removal, entrapped air, or as a result of the compressive strength test.

Also, for the samples alkaline activate with a solution obtained by mixing the solution of NaOH and Na-silicate (50/50 wt%) from figure 3, 5, 7 and 9, it was noted that

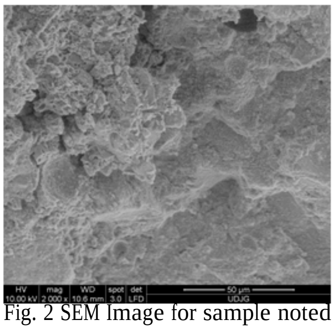


Fig. 2 SEM Image for sample noted R70N(c)7 (2000x)

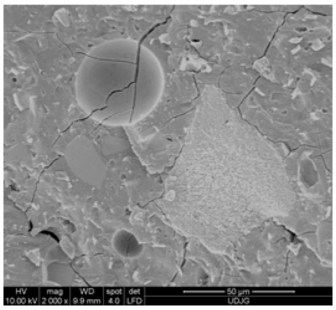


Fig. 3 SEM Image for sample noted R70Si(c)7 (2000x)

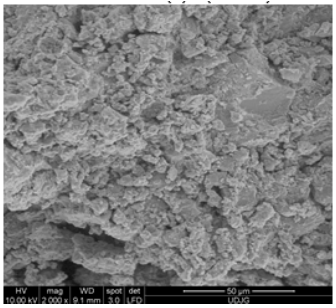


Fig. 4. SEM Image for sample noted R70N(u)7 (2000x)

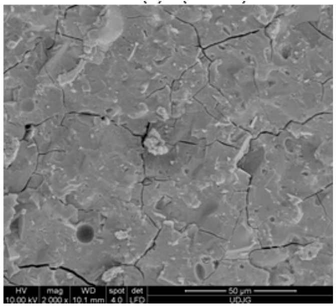


Fig. 5. SEM Image for sample noted R70Si(u)7 (2000x)

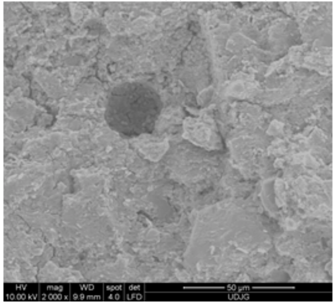


Fig. 6. SEM Image for sample noted R70N(u)28 (2000x)

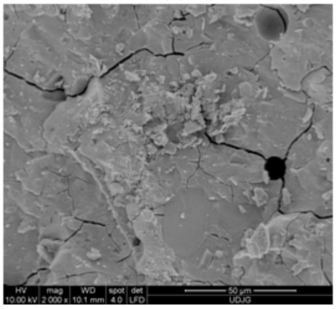


Fig. 7. SEM Image for sample noted R70Si(u)28 (2000x)

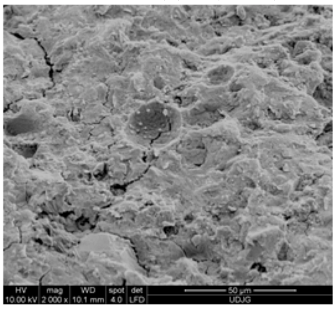


Fig. 8. SEM Image for sample noted R70N(c)28 (2000x)

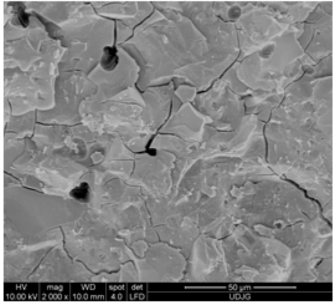


Fig. 9. SEM Image for sample noted R70Si(c)28 (2000x)

they have smooth microstructures and polymer gel is continuous, even if in some cases the cracks appeared. It was also noted that for all the samples in which calcined red mud was used, figures 2, 3, 8 and 9, the geopolymeric gel surface is bigger and shows cracks. Considering the fact that the samples taken for SEM analysis were collected after the compressive strength tests, it can be determined that these cracks may be due to the effect of compression testing, and in small amount due to the phenomenon of evaporation of water through drying. The area containing the continues gel structure supposedly is made of pure geopolimeric binder and the porous area is not just geopolimer, but there may be some microcrystals of NaOH, or $Na_2CO_3$. This may be due to a high alkalinity when some of the crystals of NaOH can precipitate due to water, which evaporates during drying. On the other hand, dissolved NaOH may also react with $CO_2$ from the atmosphere to form $Na_2CO_3$, which also may precipitate in the form of microcrystals after evaporation of the water. The formation of these microcrystals can be avoided by optimizing the conditions of drying/hardening of the obtained samples [44].

In respect of EDX analysis, table 4 shows the chemical compositions of synthesized geopolymers. By studying them, it was observed that for samples R70N(c)7, R70N(u)7, R70Si(c)7, and R70Si(u)7, elements that have a high weight are: $O_2$, C, and Na and the quantities of Si and Al are small compared to other samples. The explanation consists in the fact that they formed a few Si-O stable links, or an even more unstable connections of $Na_2CO_3$, and due to the fact that curing time was very short (only 7 days) which led to low compressive strength values. In samples in which red mud was used as such (uncalcined), R70N(u)7, R70N(u)28, R70Si(u)7, and R70Si(u)28 a higher carbon content was observed, especially for the alkaline activated samples with NaOH, respectively R70N(u)7, and R70N(u)28. Although the curing time was different, several unstable connections of $Na_2CO_3$ have formed, which is in agreement with low values resulting from carrying out the test of compressive strength compared to the resulting values for samples obtained with calcined red mud.

The highest wigth in Si, respectively 18.18% is contained in the sample R70Si(c)28, resulting in the best value of the compression strength test (85 MPa) in relation to a quantity of 17.47% Ca and 2.40% Al. Although the sample noted R70Si(u)28 are a significant content of Si (17.17%), Ca (18.52%), and Al (3.23%) but the presence of a greater quantity of C (11.35%) resulted in lower values of compressive strength test, probably due to the formation of $Na_2CO_3$ in the structure of the material.

**Table 4**
ELEMENTAL CHEMICAL COMPOSITION DETERMINED THROUGH EDX ANALYSIS OF THE SYNTHESISED SAMPLES

| Sample ID | Elemental compozition (wt%) | | | | | |
|---|---|---|---|---|---|---|
| | Carbon (C) | Oxygen (O) | Natrium (Na) | Silicon (Si) | Aluminium (Al) | Calcium (Ca) |
| R70N(u)7 | 13.66 | 40.60 | 6.27 | 13.39 | 2.35 | 14.69 |
| R70N(c)7 | 16.56 | 37.85 | 9.34 | 16.71 | 1.72 | 7.31 |
| R70Si(u)7 | 13.92 | 35.39 | 5.83 | 17.78 | 2.57 | 13.13 |
| R70Si(c)7 | 8.10 | 32.42 | 5.59 | 15.91 | 2.90 | 12.47 |
| R70N(u)28 | 16.69 | 38.63 | 8.92 | 12.75 | 2.39 | 10.99 |
| R70N(c)28 | 9.81 | 37.18 | 7.86 | 14.77 | 2.58 | 13.12 |
| R70Si(u)28 | 11.35 | 33.42 | 5.46 | 17.17 | 3.23 | 18.52 |
| R70Si(c)28 | 8.64 | 30.33 | 6.20 | 18.18 | 2.40 | 17.47 |

## Conclusions

Two geopolymer recipes from ground granulated blast furnace slag, wheat straw ash, and uncalcined /calcined red mud were made. The chemical composition was analyzed, as well as the microstructure, and compressive strength tests. Based on the experimental results obtained and the comparison between the two geopolymers, the following conclusions were drawn:

-under optimal synthesis conditions, geopolymeric materials based on slag/ash/uncalcined red mud or slag/ash/calcined red mud respectively, had good and very good compressive strength. These values were attributed to the formation of geopolymeric gel, which has bonded the undissolved particles from raw materials. The presence of geopolymeric gel has also been observed in the SEM analysis.

-geopolymers noted R70Si(c)28 obtained from 70% GGBFS + 25% WSA + 5% RM(c) alkaline activated with solution made by mixing NaOH solution with Na-silicate solution in 50 wt%, demonstrated the best mechanical properties with 85 MPa compressive strength value, for 28 testing days;

-in microstructures of geopolymers noted R70N(c)7, R70N(u)7, R70Si(c)7, and R70Si(u)7, which showed low values of compressive strength, the phases with small undissolved particles, porous surfaces and cavities of various shapes were highlighted;

-results of the basic properties of synthesized geopolymers shows that these can be used in the construction sector. Depending on the purpose of the final product, other tests are needed as well as analysis from the economic point of view.

-through the process of geopolymerization, this can significantly contribute to the conservation of the environment, bearing in mind that there is a possibility of using of large amount of existing industrial wastes.

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