Recycling of dam sediment and granulated slag wastes for the production of geopolymers materials

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ABSTRACT

The objective of this experimental work is the design of geopolymer materials, used in the field of civil engineering and presenting similar performance to Portland cement.
This work concern the optimization of the geopolymer formulation based on granulated slag (GS) and recycled calcined sediment (RCS) as source materials rich in aluminosilicate. For that different mixtures of geopolymer (100% RCS, 100% GS and 50% /50% RCS and GS) were prepared.
Several characterization tests have been carried out in order to evaluate the physical and mechanical performance of designed geopolymers such as spreading, setting times, shrinkage and compressive strength.
The obtained results show that slag paste is considered as fluid paste because of the spread value which is equal to 12cm. The setting time increases with increasing slag percentage in all mixture. The lowest linear shrinkage value is obtained for specimens based on the mixture containing 50%/50% RCS and GS. Good compressive strength of 39.5 MPa and 22.3 MPa were obtained for mixtures based on granulated slag and sediment/slag respectively.

Keywords: Geopolymers, Alkaline activation, Sediment, Slag, Waste, Aluminosilicate.

INTRODUCTION

The manufacture of Portland cement consumes significant amounts of energy. For several decades, studies have been undertaken with the aim of developing binders that consume less energy and are more suitable for the environment [1]. Among these new binders, compounds known as geopolymers have been discovered. The synthesis of geopolymers consists of an alkaline activation of aluminosilicate materials at the room temperature or slightly elevated. They are today interesting materials of the makes use in field of civil engineering. The aluminosilicate raw materials commonly used for their synthesis are fly ash, certain industrial wastes or metakaolin [2-6]
Scientists have recently investigated the durability of specimens stabilized using geopolymers. Yuan et al [7] looked at how low-calcium geopolymer concrete increased its resistance to freezing. They concluded that polyvinyl alcohol fiber improves the mechanical characteristics and cold resistances of class F fly ash geopolymer concrete the most.
Another work of Mohammad Ali et al [8] study the effects of geopolymer manufactured from recycled glass powder on the strength and durability of clay soils and found that, adding recycled glass powder to the soil enhanced the unconfined Compressive strength of the soil. Because the weight percentage of glass powder had an ideal value of 15%, using weight values less or more than this value results in

lower unconfined Compressive strength. For the unconfined Compressive strength test, the ideal molarity of the activating ingredient was 3%.

Dam sediments constitute a material whose percentage in Al_2O_3 and SiO_2 can allow their use for the synthesis of geopolymer materials.

Annually huge quantities of sediment are evacuated from the dams in order to ensure their good functioning, however these considerable quantities generate an ecological problem, The valorization of rejected sediments is essential as a solution to the difficulties of storage and the nuisance of the environment for this reason various researches have proposed the recovery of this waste in the field of civil engineering.

Ben Allal. L et al [9], studied the recovery of dredged sediments from the ports of Tangier and Larache (Morocco) in mortars by substituting sand. The compressive strength values obtained confirmed that a partial substitution of sand by dredging sediments from the ports of Tangier and Larache in the mortars is satisfactory for a dosage of 20%.

Achour R [10], studied the durability of two concrete blocks based on marine sediment. Another work of Bourabah et al [11] studied the mixture of 30% natural sediment and 70% sand in road construction and concluded that the addition of a granular material showed an improvement in dry density and a reduction in optimal moisture content, and concluded that the treated sediment can be used as a foundation and base layer. Gueffaf and al [12] investigate the use of sediment dam for the elaboration of stabilized earth block. The obtained results show that elaborated bricks with sediment have good thermal insulation with thermal conductivity of 1.06 W/m.k at 15% cement.

The main objective of this work is the development of a geopolymer material based on recycling dam sediments and granulated blast furnace slag, after design the physical and mechanical performances of specimens were studied.

Materials used

Recycled sediment

The sediment used in this experimental study comes from the KADDARA dam (figure 1) located in north Algeria. It is an earth dam whose height of the dike is 106m over a length of 468m, it is intended mainly for the city of Algiers.

Fig. 1: Photography of KADDARA dam sediment

All the quantity taken by sampling from the Kaddara Dam is selected homogenized, dried in open air, crushed and reduced to powder, sifted through a 1mm sieve then washed to eliminate unwanted organic matter, after 24 hours we recover the material which precipitates.

The treated sediment is calcined for 3 hours at a temperature of 750°C. After that, calcined sediment is crushed and sieved to 40µm.

All stages of treatment and preparation of sediment are represented in figure 2.

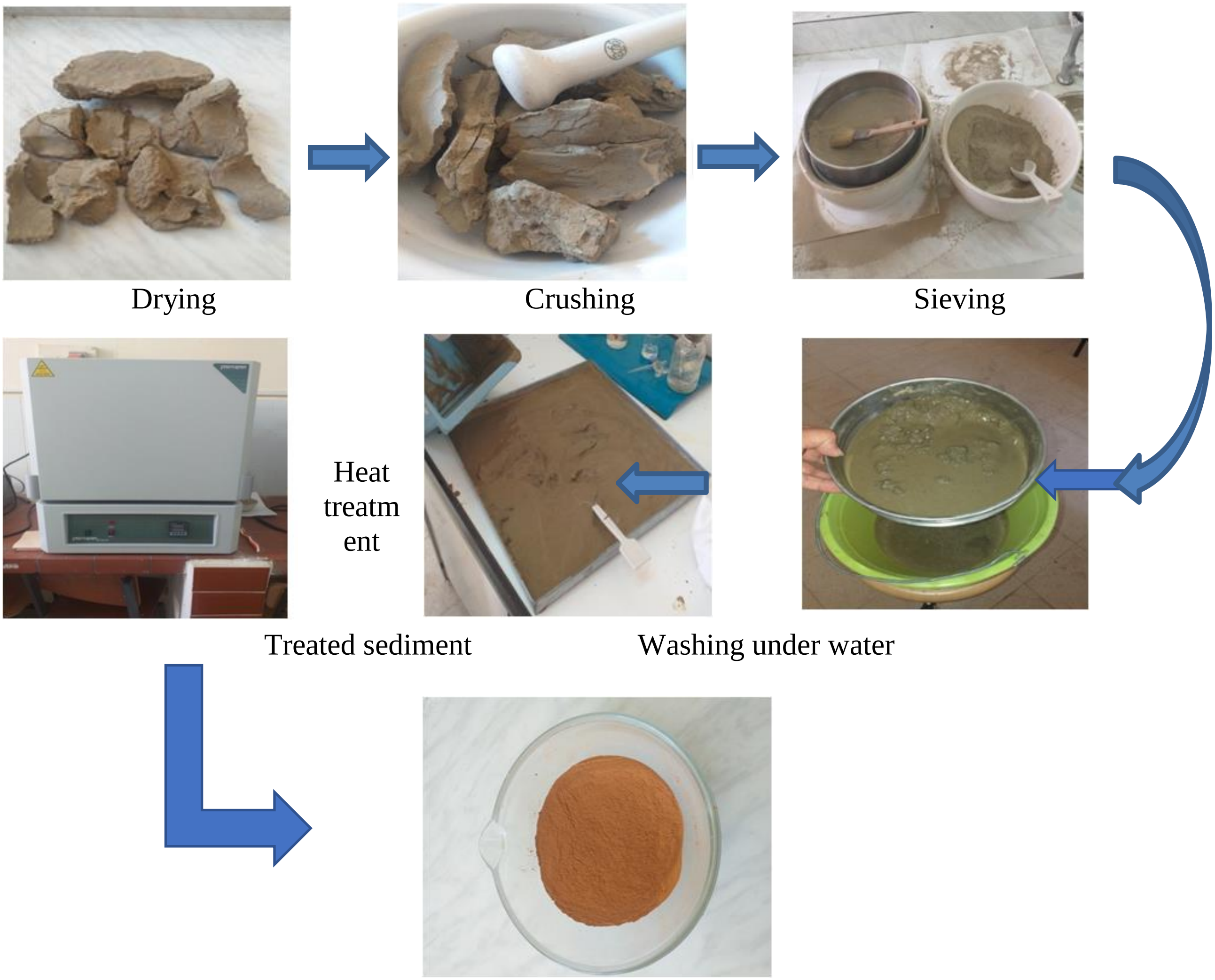


Fig. 2: Treatment and preparation stages of sediment.

Granulated blast furnace slag

The granulated slag used in this work is in the form of light gray sand, with a grain size of 0 to 5 mm. The granulated blast furnace slag was ground by a ball mill and then sieved to 40μm.

Fig. 3: Granulated blast furnace slag

Alkaline Solution

The alkaline solution used in this work results from the dissolution of a quantity of sodium hydroxide in distilled water to obtain an aqueous solution of sodium hydroxide with a concentration (12 M) to be used as an alkaline activating solution.
The solution obtained was left at room temperature (21 ± 3°C) in the laboratory for at least 24 hours before use, so that there is better ionization.

Formulation of geopolymers pastes

Three formulations of geopolymer pastes have been synthesized depending on the nature of the raw material:
- Recycled calcined sediment samples noted by (RCS): Composed of 100% recycled calcined sediment.
- Granulated slag samples (GS): Composed of 100% granulated slag.
- Calcined sediment and granulated slag samples (RCS-GS): Composed of a mixture of 50% calcined sediment and 50% granulated slag.

These formulations are compared to a reference sample composed of 100% cement noted by (C).
For all formulations the solution/ Binder (S/B) ratio was kept constant: S/B= 0.4.
For the preparation of specimens, all materials were firstly homogenized for 5 minutes. Then the alkaline solution was added and the mixture between powder and solution was thoroughly homogenized using a mixer for 10 minutes. The obtained paste was used to fill cylindrical PVC test specimens (diameter = 40 mm and height = 80 mm).Once the specimens were covered and stored in the laboratory environment at a temperature of 20° ±1°C. The demoulding is carried out after a period of 24 hours and the samples are kept in the laboratory to be used for different characterization tests.

Fig. 4: Designed géopolymére specimens

Results and discussion

Characterization of raw materials

The Physical characteristics and the chemical compositions of the raw materials such as calcined sediment and granulated slag are summarized in the table 1 and 2, respectively.
The calcined sediment used is essentially constitute of silicium oxide SiO_2 and aluminium Al_2O_3 which are the majority oxides, an acceptable content of Fe_2O_3, CaO and MgO, this material is also made up of TiO_2, Na_2O, SO_3 and MnO with a small amount. The SiO_2/Al_2O_3 mass ratio is about 2.92.
Granulated slag contains a large amount of silicon oxides SiO_2 and calcium CaO, these are two elements which are the majority oxides essentially in siliceous mineral phases. An acceptable content of Al_2O_3, MgO, Fe_2O_3, and MnO.

Table. 1: Physical characteristics of sediment and granulated slag

	Calcined sediment	Granulated slag
Specific gravity (g/cm^3)	2.40	2.71
Blaine specific surface (cm^2/g)	6574	6082

Table. 2: Chemical composition of raw materials

Constituents	SiO_2	Al_2O_3	Fe_2O_3	CaO	K_2O	MgO	TiO_2	Na_2O	K_2O	SO_3	MnO
Calcined sediment	58.5	20.0	7.46	5.85	3.91	2.05	0.85	0.63	/	0.34	0.14
granulated slag	41.01	6.38	2.45	41.5	3.91	2.05	0.85	/	0.63	1.55	2.26

Mineralogical analyzes by XRD:

The results of the X-ray diffraction analyzes of the calcined sediment and granulated slag, give us an idea of the main crystalline minerals by the existence of the characterizing lines (figure 5).
Granulated slag is rich in Quartz (SiO_2) with prominent peaks, followed by vaterite and less calcite ($CaCO_3$).
for the calcined sediment, the results indicate the presence of Quartz (SiO_2) with significant peaks, followed by calcite ($CaCO_3$) and a good proportion of alumina (AL_2O_3) represented in the form of Kaolinite (AL_2O_3 $2SiO_2$ $2H_2O$) and the illite, clinochlor, albite (Na Al Si_3O_8) and orthoclase are in low proportions.

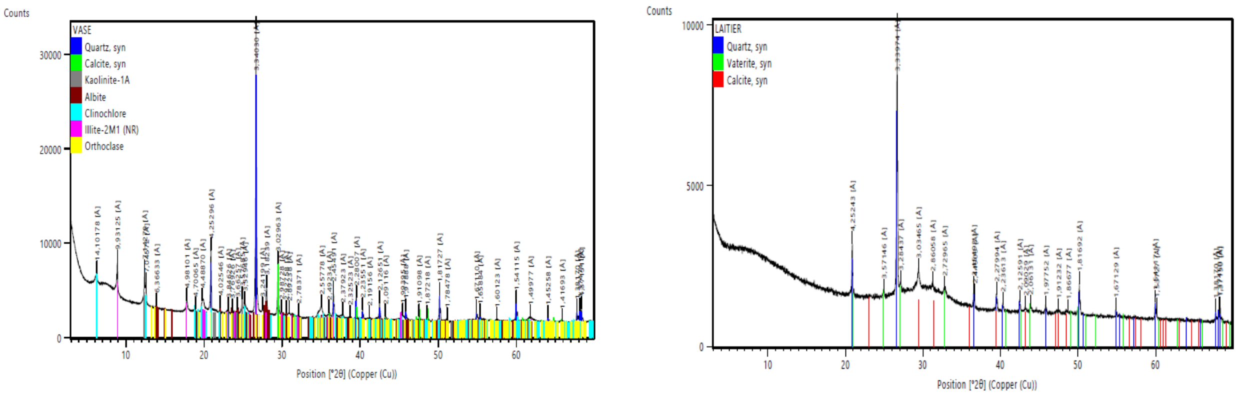


Fig. 5: Mineralogical analysis by XRD of calcined sediment and granulated slag.

Differential Scanning Calorimetry (DSC)

Figures 6 and 7, shows the obtained results by DSC anaysis of granulated slag and sediment respectively. The DSC curve of the granulated slag (Figure 6) represents the polymorphic transformations of silica towards 667.9°C representing the transformation of Quartz, and towards 1276°C, a peak representing the transformation into tridymite.

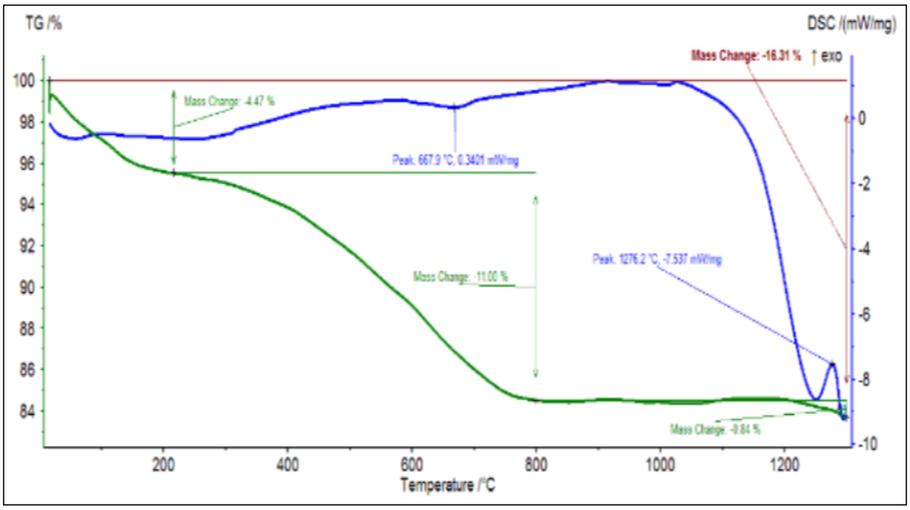


Fig. 6: Curves differential scanning calorimetry of granulated slag.

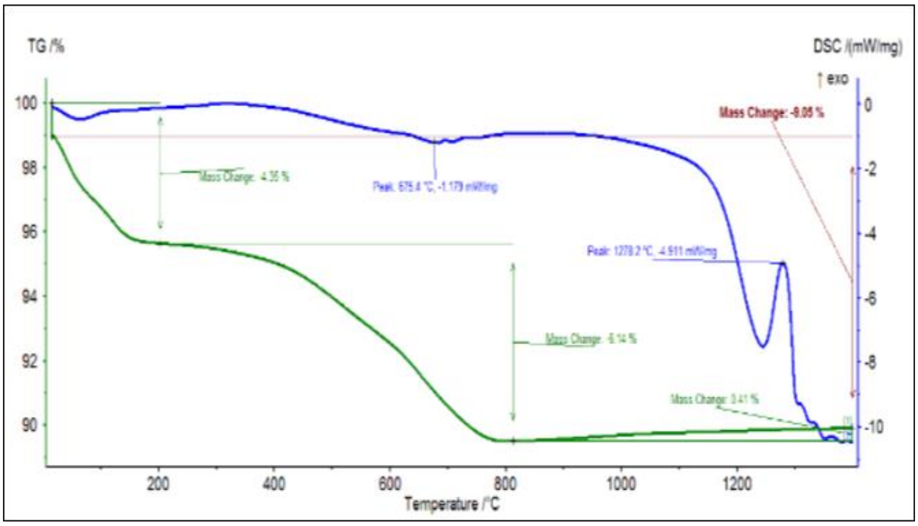


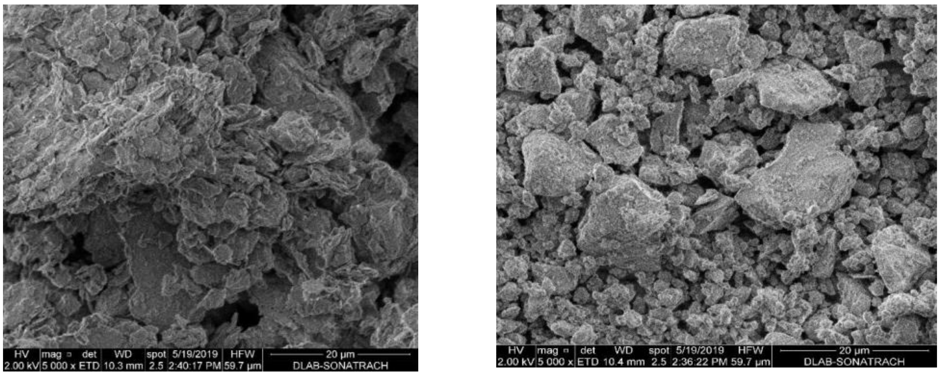
Fig. 7: Curves Differential scanning calorimetry of sediment.

The thermograms of figure 7 reveal the presence of three common peaks including two endothermic and one exothermic. The first endothermic peak, located between 60 – 120°C, is attributed to a loss of mass linked to the departure of 4.47% hygroscopic water, initially present in the clay fractions.
The second endothermic peak at 675.4°C, located between 530 and 750°C, related to the dehydroxylation of kaolinite and its transformation into metakaolinite. The considerable mass loss observed at 810°C is equal to 6.14% corresponding to the elimination of the Kaolinite constitution water.
The last common exothermic peak, located between 960°C and 1000°C, is linked to the structural reorganization of the metakaolinite, with a very low loss variation of 0.41%.

Scanning electron microscope (SEM) analysis

Scanning electron microscope observations show that the sediment is essentially made up of agglomerates particles. These particles are themselves formed from a stack of hexagonal plates (Figure 8.a). The dimensions of the plates vary from a micron to around ten microns.

For the SEM photos of the granulated slag, we observe grains of micrometer size up to grains close to thirty micrometers are particles of angular appearance with sharp edges.



a) Calcined sediment **b) Granulated slag**

Fig. 8: SEM photography of raw materials

Spreading test

In order to estimate the properties in the fresh state of the different geopolymers mixture, we study the spreading of paste using mini-cone test (figure 3). According to the obtained results (Figure 9), it is noted that the value of the spreading of the composition with calcined sediment is very low (8 cm) compared to the reference sample which confirms a firm paste (bad flow absence of workability), for the mixture of 50 % calcined sediment and 50% granulated slag the value of the spread is equal to 10 cm, so it is a plastic paste and for the activated granulated slag paste it is considered a fluid paste because of the value of the spreading which is equal to 12 cm close to the value of reference paste. For the low spreading value of the paste, the calcined sediment and granulated slag mixture with (plastic paste) results from the combination between two materials rich in silicon SiO_2, which ensures an increase in the quantity of amorphous phase. The more the amount of amorphous phase of the aluminosilicate material is abundant, the more the demand for water increases. The granulated slag paste spreads well and is workable compared to the other two types of paste because of its vitreous structure.

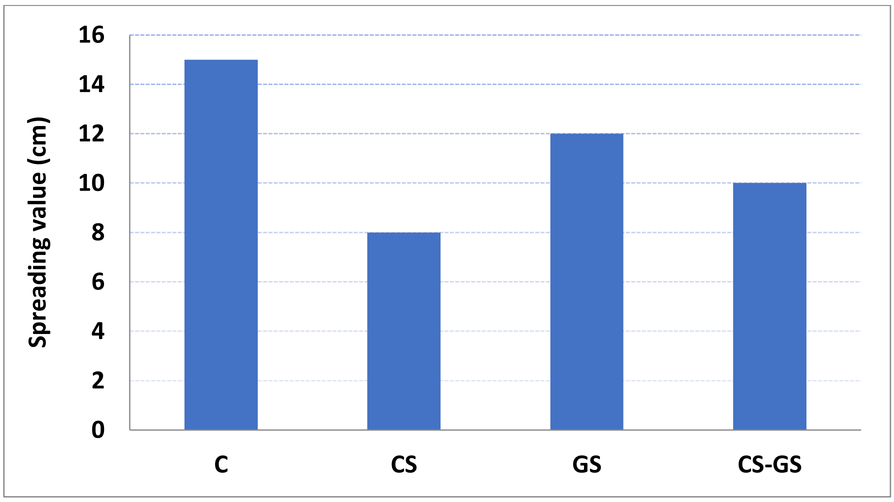
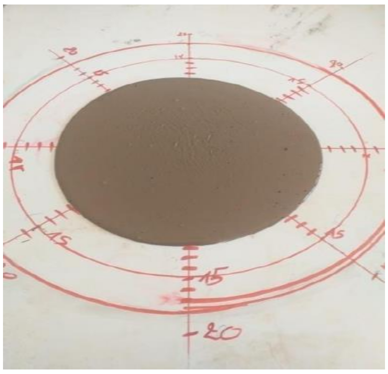
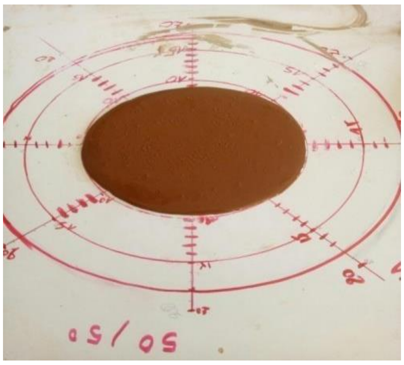


Fig. 9: Spreading of geopolymers formulations



a) (GS) paste



b) (CS-GS) paste

Fig.10: Spreading of pastes

Setting time test

The fluidity of geopolymer pastes decreases with the increase in the percentage of calcined sediment, which makes it possible to reduce the start and end of setting time in the two cases of geopolymers based on (100% calcined sediment) and based on a mixture (50% calcined sediment and 50% granulated slag), the high fineness to cause an affinity to the activating solution thus causing a reduction in the setting time. A high concentration of NaOH=12M increases the viscosity of the alkaline solution, hence a reduction in the onset of setting time.
On the other hand, the setting time of geopolymers based on (100% granulated slag) is higher because of their vitreous structure, the reduced fineness compared to the samples of calcined sediment, therefore a low water demand, a high CaO content. The setting time is high when the percentage of CaO in the aluminosilicate material is high [13, 14].

Table 3: Setting time of the different geopolymer pastes

	Samples	C	RCS	RCS-GS	GS
Setting time (min)	Start of setting	135	139	128	143
	End of setting	183	256	242	300

Density

The density values for the geopolymer samples are presented in Figure 11.
The essential parameters which influence the value of the density of the samples are the fineness which plays a very important role on the compactness of the materials and the mass ratio alkaline solution /binder (water content). The slag-based geopolymer sample presents the highest density values because the slag develops the best geopolymerization reactions, therefore better densification of the structure and greater compactness.

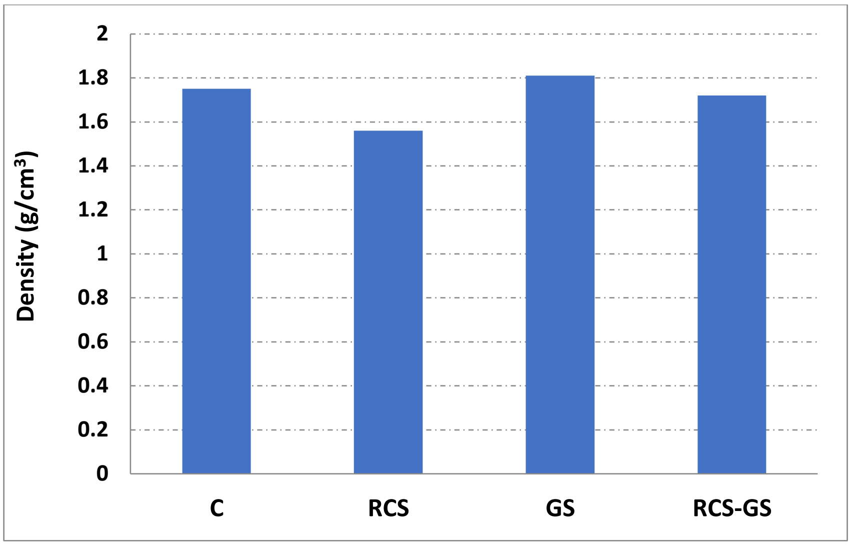


Fig. 11: Density of all géopolymes samples

Linear shrinkage

The obtained results for the linear shrinkage test are represented in the figure 12.
The lowest linear shrinkage value is (7.5%) is obtained for geopolymers based on a mixture (50% calcined sediment and 50% granulated slag) it is noted that the specimens have not undergone any change after 28 days. On the other hand, the highest value compared to the control is (21.25%) corresponding to geopolymers based on calcined sediment, the linear shrinkage of geopolymers based on granulated slag decreases slightly with a value of (12.5%).

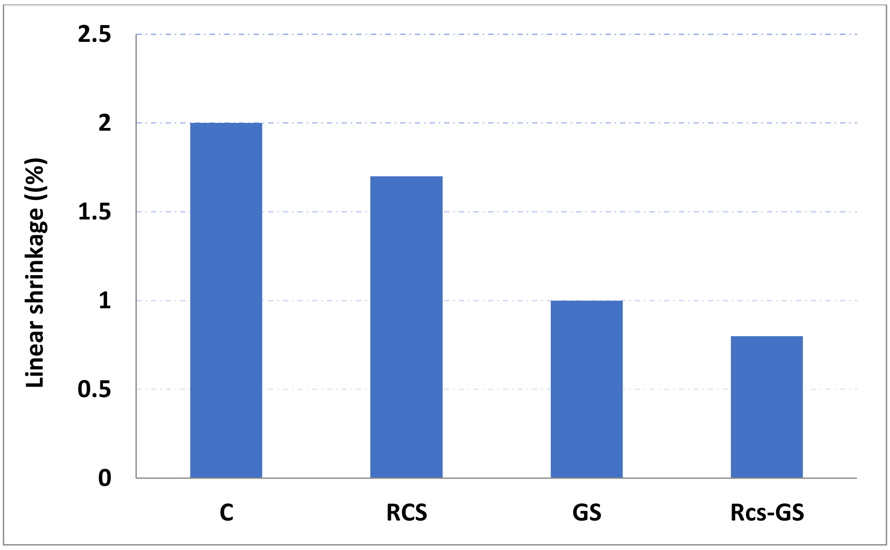


Fig.12: Shrinkage test of geopolymers samples

Water absorption and porosity

Obtained results show an increase in the water absorption and the porosity for the geopolymer samples based on calcined sediment, it can be said that it results from the elimination of hygroscopic water, the constitutions of the sediment and a rate of low amorphous phases; low densification reduces the compactness between the grains. The elimination of these waters and the migration of the alkalis outside the specimen cause a modification of the microstructure of the material, which probably causes pores to appear. The low values of water absorption and the porosity for the samples of geopolymers based on a mixture (50% calcined sediment and 50% granulated slag) and based on (granulated slag) we can say that its results from the vitreous structure of slag and their density of solid particles which has a high density which ensures good densification between the grains therefore a very compact and less porous material.

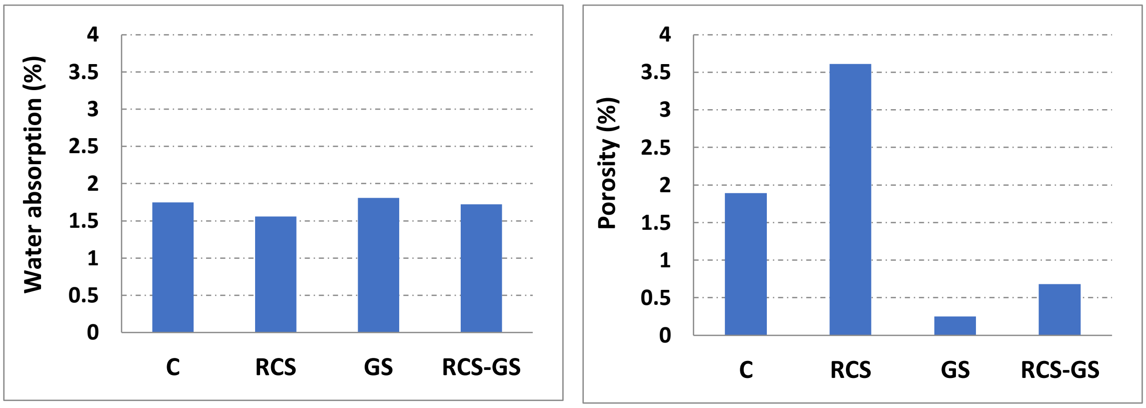


Fig.13: Water absorption and porosity of specimens

Compressive strength

In order to estimate the mechanicals properties in the hardened state of the different geopolymers mixture, we study the compressive strength of pastes. According to the obtained results (Figure 14, 15 and 16), it is noted that the value of compressive strength of geopolymers based on a mixture of 50% calcined sediment and 50% granulated slag (RCS-GS) are higher than those of the geopolymer values based on 100% calcined sediment (RCS) and lower than the geopolymer values based on 100% granulated slag (GS).
We note also that the strength in the short term (3, 7) days is very low, also in the long term (28) days there is an increase.
The high values of the compressive strength of geopolymers based on granulated slag that have been obtained are the result of good geopolymerization, due to their mineralogical and chemical composition which is close to that of Portland cement, the slag hydration process is then similar to that of Portland cement as explained by Le Chatelier [15].

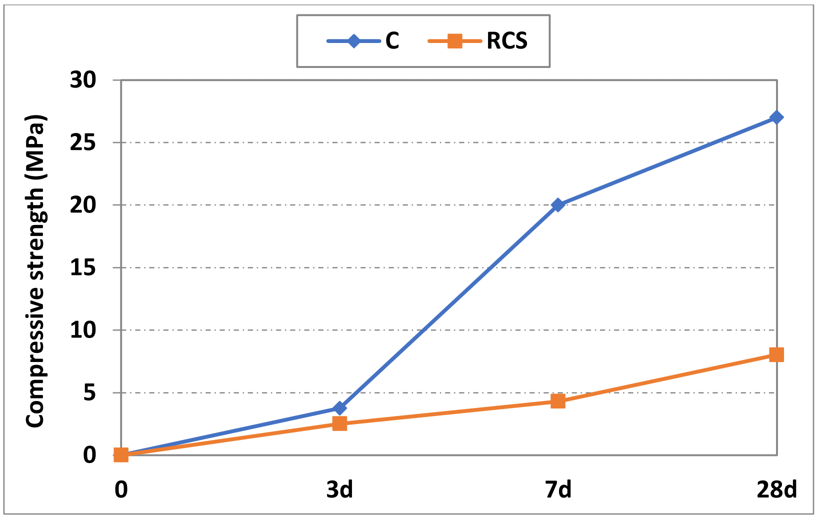


Fig.14: Compressive strength of RCS specimens compared to the reference specimens

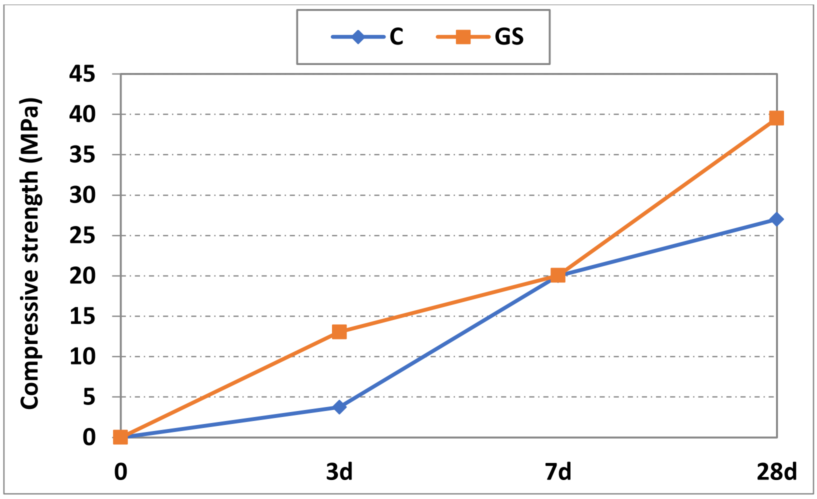


Fig.15: Compressive strength of GS specimens compared to the reference specimens

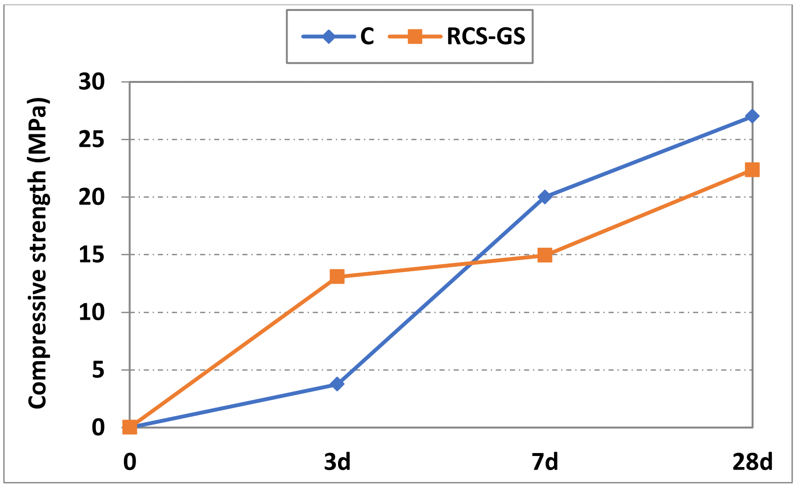


Fig.16: Compressive strength of RCS-GS compared to the reference specimens

The figures 17 and 18 show the mineralogical analysis by x-ray diffraction of the two geopolymer samples, sediment/slag mixture and granulated slag.

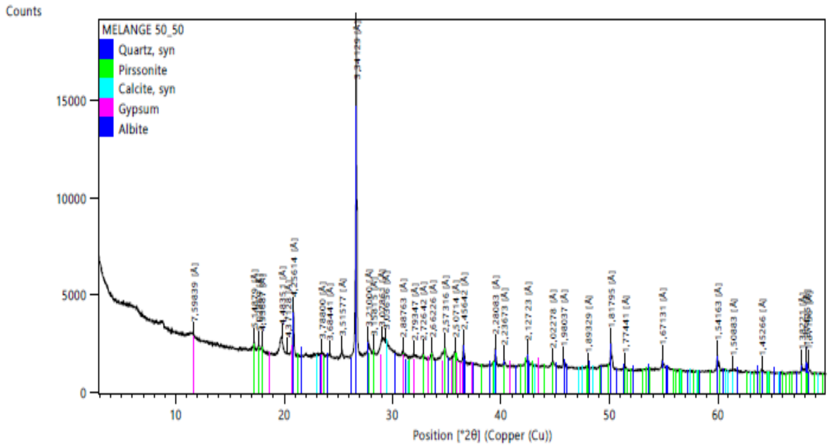


Fig. 17: X-ray diffraction analysis of geopolymer specimens (RCS-GS)

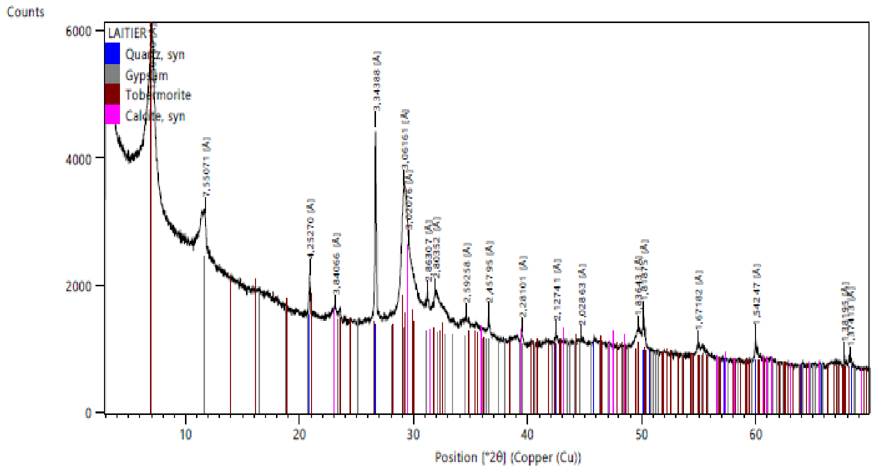


Fig. 18: X-ray diffraction analysis of geopolymer specimens (GS)

Conclusion

This work concerns the optimization of geopolymer formulation based on granulated slag and recycled sediment as source materials rich in aluminosilicate. The various analyzes carried out show that the size of the particles and their specific surface influence significantly the properties of geopolymer materials

For geopolymers based on 100% granulated slag and based on a mixture, the specimens did not undergo linear shrinkage after 28 days, for geopolymers based on calcined sediment, the linear shrinkage increases in time for all the specimens with a low value compared to the control specimen.

The fluidity of geopolymer pastes decreases with the increase in the percentage of calcined sediment, which makes it possible to reduce the setting time for the geopolymers based on (100% calcined sediment), the high fineness to cause an affinity with the solution activator thus causing a reduction in setting time.

The geopolymers based on 100% granulated slag achieves a higher compressive strength than cement which will allow us to obtain a binder of acceptable class.

The increase in fineness positively influences the polymerization reaction, the best performance has been obtained for granulometry of 40 microns for all formulations.

Finally, through this study it can be said that the activation of sediment dam could be more successful if we control the heat treatment process and increase the rate in the amorphous phase.

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