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Synthesis and Characterization of Slag-Sludge-Based Eco-Friendly Materials – Industrial Implications

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ABSTRACT

Currently in Morocco, wastewater treatment plants (WWTPs) generate huge quantities of sludge from wastewater treatment. This sludge is a real concern for the environment. No regulatory text takes into account the future and management of sludge, its use in agriculture is not officially authorized and the solutions currently implemented for its elimination or recovery are done on an ad hoc basis and pose a number of difficulties for operators. The objective of the present study is to investigate the stabilization and solidification of a geopolymer based on sewage sludge and blast furnace slag (slag), as well as the possibility to use this geopolymer as a thermal insulation material. The sludge used comes from the AIN AOUDA-activated sludge type WWTP, the collected sludge is dehydrated and stabilized by slaked lime ($Ca(OH)_2$) in the sludge treatment process. The blast furnace slag (slag) came from the plant in France. Four samples were prepared by substituting the slag with quantities (10 to 40%) of limed sludge, a quantity of sand, and a solution of sodium silicate and sodium hydroxide used as an alkaline activator. The effect of limed sludge on the physicochemical and microstructural properties of the synthesized geopolymers was evaluated using several analytical techniques, such as compressive strength, P-wave velocity, density and porosity tests, and X-ray diffraction. The results showed that the addition of 10 to 40% of limed sludge resulted in a progressive decrease in the compressive strength of the synthesized geopolymer and an increase in the thermal conductivity, which allows the use of the synthesized geopolymer as a material.

Keywords: geopolymer mortar; non-incinerated sewage sludge; blast furnace slag; construction materials.

INTRODUCTION

Wastewater sludge, a by-product of wastewater treatment, is a "special" category of waste. The increase in the production of these sludges has pushed researchers to find recovery solutions in several areas [Andreea Gherghel et al. 2019]. These applications offer alternative methods for sludge recycling and long-term resource savings. From a broad review of the literature, numerous published research on the application of sewage sludge and incinerated sludge ash in building materials [Cieślik and Konieczka, 2017], such as the production of eco-cement from the use of dry municipal sewage sludge as a partial substitute for 5% to 15% of traditional raw materials in the production of Portland cement [Rezaee et al., 2019]. Some studies have been conducted on the use of sewage sludge ash as brick material [Hassan et al., 2022], for the manufacture of ceramic products [Panepinto et al., 2016, Amin et al., 2018] or to produce lightweight aggregate (LWA) [Franus et al., 2016, Mañosa et al., 2021]. Another application of sewage sludge ash that has attracted a lot of research interest is its use in cement-based materials, the sludge ash is added as supplementary cementitious material to partially replace cement in mortar or concrete [Li et al., 2017, Świerczek et al., 2021, González et al., 2021, Vouk et al., 2018]. It is obvious that the use of sewage sludge ash alone as supplementary cementitious material in concrete production gives low mechanical strength due to the very slow hydration reaction, however, the addition of several pozzolanic minerals such as metakaolin [Istuque et al., 2016, Chen et al., 2021], Blast furnace slag and quicklime, improve the performance of sewage sludge ash mortar or concrete. In addition, the mechanical properties would be further improved by adding lime to the pozzolanic mineral mixture [Chakraborty et al., 2017]. This study focuses on the use of recycled sewage sludge ash combined with quicklime and blast furnace slag as a cementitious material to control the physical and mechanical performance of mortar, the addition of QL and the optimal amount of BFS in the SSA significantly improves the physical and mechanical properties of the mortar. In a study by Chen et al. [Chen et al., 2018], blast furnace slag (GGBS) was used to make geopolymer pastes in which sludge ash was used as a precursor. The results showed a maximum compressive strength of 32.8 MPa at 28 days.

All the above studies were performed on the preparation of geopolymers with sludge ashes (waste obtained from the incineration of sewage sludge). However, this study aimed at investigating the geopolymerization of mixed geopolymers based on dewatered sludge stabilized with lime.

The objective of this research is to study the effect of limed sludge without incineration, on the physico-mechanical and microstructural properties of geopolymers based on blast furnace slag. From a methodological point of view, a chemo-mineralogical characterization of the two wastes (unincinerated sludge (LSS) and blast furnace slag (BFS)) was first carried out using XRD and ICP analysis. A Series of geopolymers were then synthesized by substituting the slag with a growing quantity of limed sludge, the geopolymers thus obtained were subjected to a series of physicomechanical (compressive strength (Rc), bulk density (D), and porosity (P)) and microstructural (XRD,) characterizations in order to study the mechanical properties of the obtained materials. The results obtained were discussed in light of the numerous previous studies to identify the physical and mechanical properties of the materials obtained.

In addition, this work aims to valorize the sludge only stabilized by lime without incineration in the field of geopolymers. Its objective is to identify the alternatives of valorization, which allow better management of the sewage sludge, and consequently our work presented falls under the optics of sustainable development and environmental preservation.

EXPERIMENTAL PROGRAM

Materials

Blast-furnace slag

Blast furnace slag is a by-product of the steel industry. They are generated during the steel production process, during the iron ore smelting stage. The blast furnace slag used in this study comes from the French company ECOCEM.

Sewage sludge

The sewage sludge used in this study comes from the Ain Aouda wastewater treatment plant (a city in Morocco about 30 km from the country's capital). The wastewater treatment system of this plant is of the low-load activated sludge type. The Ain Aouda wastewater treatment plant has a composite sludge treatment process. The diagram in Figure 1 shows the sludge treatment stages of the Ain Aouda wastewater treatment plant. The dewatered and limed sludge is sent to three (3) sludge storage silos. This sludge is stored at the WWTP for disposal in the public landfill which poses an environmental problem. The sampling of the limed sludge used in this work was carried out just after the dehydration phase by centrifuge and liming.

Activating solution

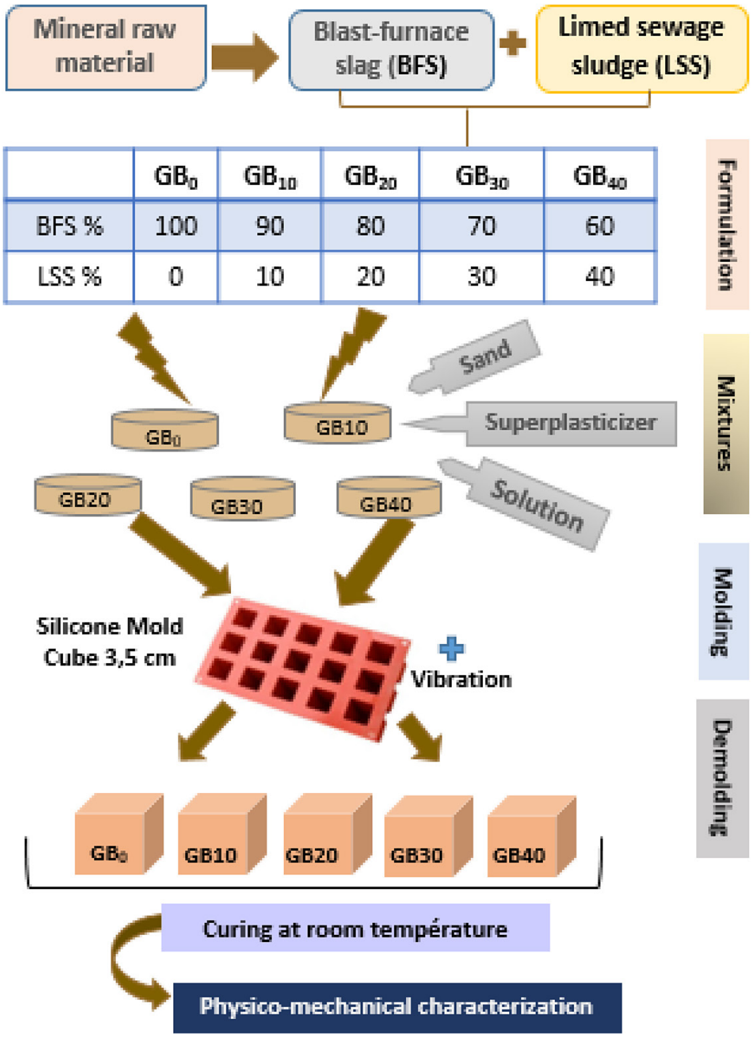
A NaOH solution with a molarity of 8M and a sodium silicate gel was used as activating solutions in the present study. The NaOH solution was prepared by dissolving NaOH pellets (98%) in distilled water and mixed with a sodium silicate gel composed of 25.7% SiO_2, 10.2% Na_2O, and 64.1% H_2O with a $NaOH/Na_2SiO_3$ ratio = 1. The activating solution is prepared 24 hours before the manipulations.

Mortar's preparation

Synthesized geopolymers

The method of preparation of the geopolymers in this study is shown in Figure 2. The sludge is spread on a tray in a single layer, dried in an oven at 105 °C for 24 hours, and kept in a

Figure 1. Treatment stages of sludge from the AIN AOUDA wastewater treatment plant



	GB_0	GB_{10}	GB_{20}	GB_{30}	GB_{40}
BFS %	100	90	80	70	60
LSS %	0	10	20	30	40

Figure 2. Methodological scheme of the geopolymer synthesis in this study

Table 1. Mix design

Sample	Sand (g)	Blast-furnace slag (g)	Limed sewage sludge (g)	Superplasticizer (g)	Solution (g)
GB_0	150	100	0	1	70
GB_{10}	150	90	10	1	70
GB_{20}	150	80	20	1	80
GB_{30}	150	70	30	1	90
GB_{40}	150	60	40	1	100

closed bag. Next, the sludge was powdered using a ball mill and sieved to obtain very fine powders (particle size <63 μm). The sand was sieved to 200 μm. These constituents were mixed according to the formulations displayed in Table 1. A total of 5 mixtures are produced using sand and slag which will be substituted by different fractions of limed sludge. The slag-based geopolymer composites are synthesized by incorporating different percentages of limed sludge (LSS) 10% to 40% in a 10% step. The activating solution (NaOH (8-M) and Na_2SiO_3), previously prepared, was added to the mixture, and finally, to compensate for the loss of workability, a Sika-type superplasticizer was used. The mixtures were homogenized and then the resulting slurries were poured into 3.5 cm cubic molds. The filled molds were placed on a vibrating plate to eliminate the air bubbles inserted in the slurries, then left in the ambient air of the laboratory 24 hours later, the samples were removed from the molds and stored in airtight bags until the age of tests. The mixture was homogenized and then poured into 3.5 cm cubic molds. The filled molds were placed on a vibrating plate to remove air bubbles inserted in the mixture, then left in the ambient air of the laboratory 24 h later, and the samples were removed from the molds.

Characterization methods

ICP-MS analysis

Inductively Coupled Plasma Source Mass Spectrometry (ICP-MS) will make it possible to determine the chemical composition of the sludge (mainly heavy metals), evaluate their degree of pollution, and ensure their conformity for a possible valorization.

XRD analysis

The slurries and the prepared geopolymers were examined by X-ray diffraction using a powder X-ray diffractometer “Labxxrd-6100 SHIMADZU”. It allows the determination and identification of unknown phases, minerals, organic or inorganic.

Scanning electron microscopy

Scanning electron microscopy analysis is capable of producing high-resolution images of the surface of samples down to a few nanometers. It can also perform a chemical analysis using an EDS (Energy DipersiveX-ray Spectroscopy) detector.

Compressive strength test

The compressive strength (Rc) of the synthesized geopolymers was measured, according to ASTM 1231 (2010), on cubic samples (3.4 cm Ø) after 14 days of curing using a hydraulic press (RP25 ATF-ASTM) at a loading rate of kN/s. For each formulation, at least three replicate specimens were tested, and the average mechanical strength was taken as the representative value.

P-wave velocity measurement

The P-wave velocity measurement on geopolymers was performed after 28 days of curing, using a TICO-type (NFP 94-411), composed of an ultrasonic wave generator (0.1-6500 s; 1 pulse/s) and associated with two piezoelectric transducers. The P-wave velocity (longitudinal waves or P-waves) is a non-destructive analysis method to evaluate the solid matter compaction and physical continuity.

Measurement of bulk density, porosity, and water absorption

The bulk density, porosity, and water absorption of the synthesized geopolymers were determined, according to ASTM C642-13, at a curing age of 28 days. For each geopolymer sample, at least three tests were performed and the average rate was considered the representative value.

RESULTS

Characterization of raw materials

Blast-furnace slag

Chemical analysis by XRF revealed that the slag is mainly composed of 37.4% SiO_2, 10.8% Al_2O_3, 43.7% CaO, and 6.5% MgO (Table 2). Qualitative and quantitative mineralogical characterization showed that the blast furnace slag used in this work is a nearly amorphous material composed of 98.4% of the amorphous phase (Figure 3), indicated by the high intensity of the amorphous phase halo between 20° and 40° in 2Θ, and 1.4% of the crystalline phase in the form of traces of quartz and calcite.

Sewage sludge

The results of the analysis of the solution in the atomic absorption spectrometer (ICP) are presented in Figure 4. It was noted that the limed

Table 2. Chemical composition of blast furnace slag determined by FX

Chemical composition	SiO_2	Al_2O_3	CaO	MgO	Fe_2O_3	TiO_2	Na_2O	K_2O	SO_3	LOI
Blast-furnace slag (%)	36.9	10.2	44.1	6.3	0.39	0.53	0.19	0.37	0.12	0.03

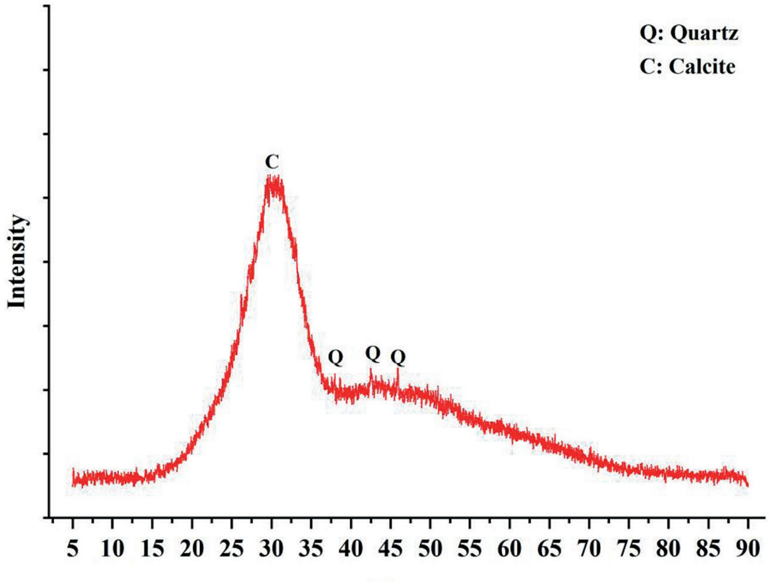


Figure 3. X-ray diffractogram of blast furnace slag

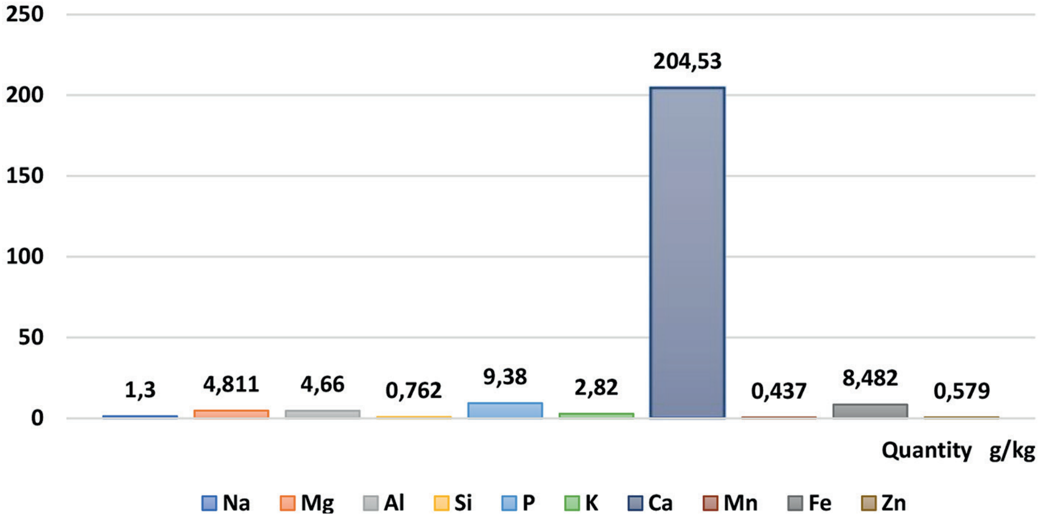


Figure 4. Results of the analysis of the solution in the atomic absorption spectrometer (ICP)

sludge sample is rich in calcium (204.53 g/kg) with low levels of phosphorus (9.38 g/kg), aluminum (4.66 g/kg), and magnesium (4.81 g/kg). Si, Zn, and Mn are present in trace amounts.

Characterization of synthesized geopolymer mortars

Macroscopic aspect

Macroscopic observation of the synthesized geopolymer mortars (Figure 5) shows that the geopolymer mortar samples have unchanged shapes and heterogeneous colors that change with the addition of limestone slurry until a light color is obtained. Furthermore, it was found that the macroscopic porosity increases and the hardness decreases with the increase of the slurry content from 0% to 40% with the appearance of cracks on the surface of the GB_{40} sample containing 40% of the slurry. The deposition of the efflorescent layer was not observed in all samples.

Optical microscope

The microstructure of the synthesized mortars was studied under an optical microscope. The micrographs presented in Figure 6 show that the mortars have a good distribution of sand grains in the geopolymer matrix. Moreover, it was observed that the increase of the limed slurry content from 0% to 40% significantly increases the apparent porosity of the geopolymer matrix with the appearance of microcracks in the sample containing 40% limed slurry.

Compressive strength

The compressive strength (Rc) of the synthesized geopolymers was measured, according to

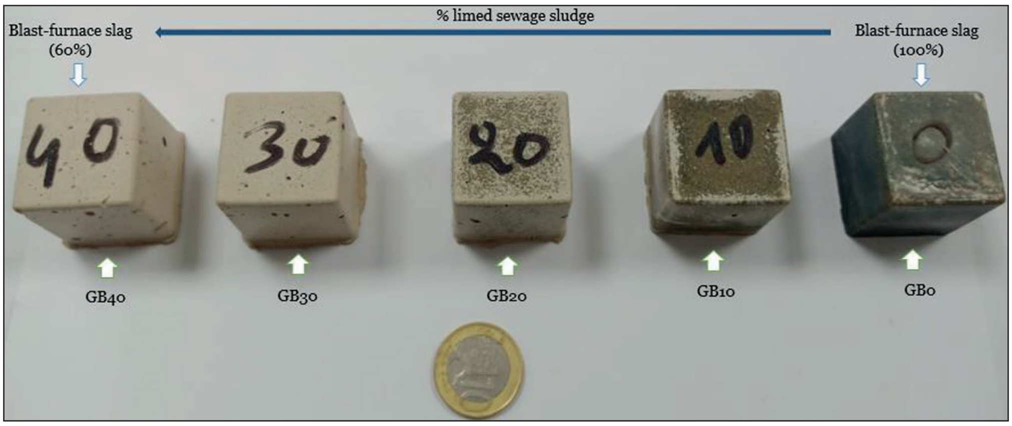


Figure 5. Typical photographs of synthesized geopolymers coded GB_0, GB_{10}, GB_{20}, GB_{30}, and GB_{40}

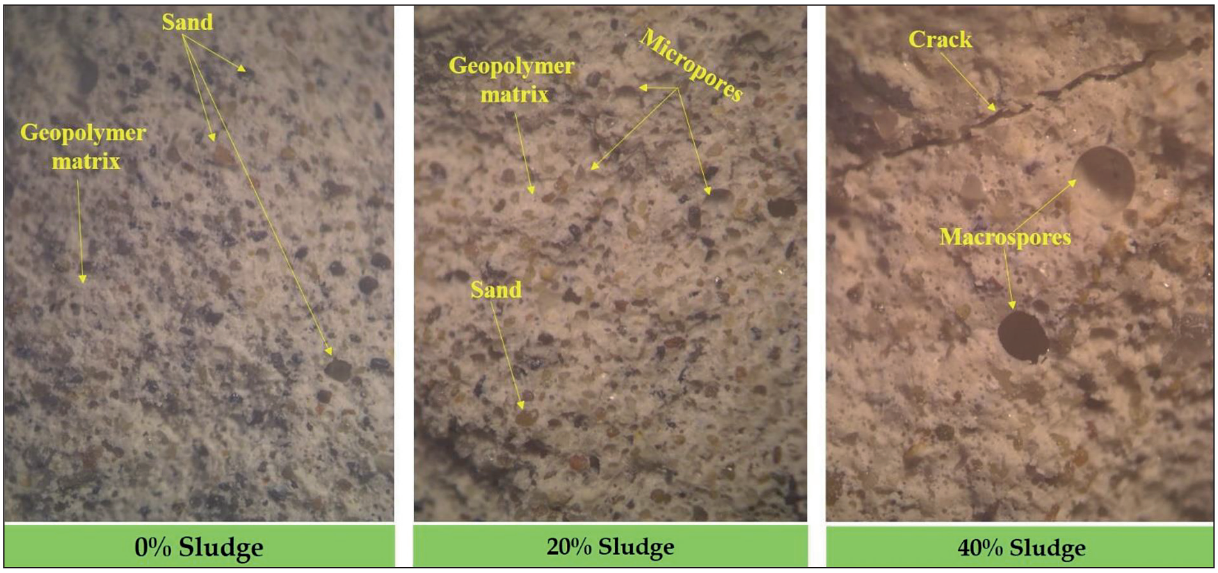


Figure 6. Optical micrographs of geopolymers

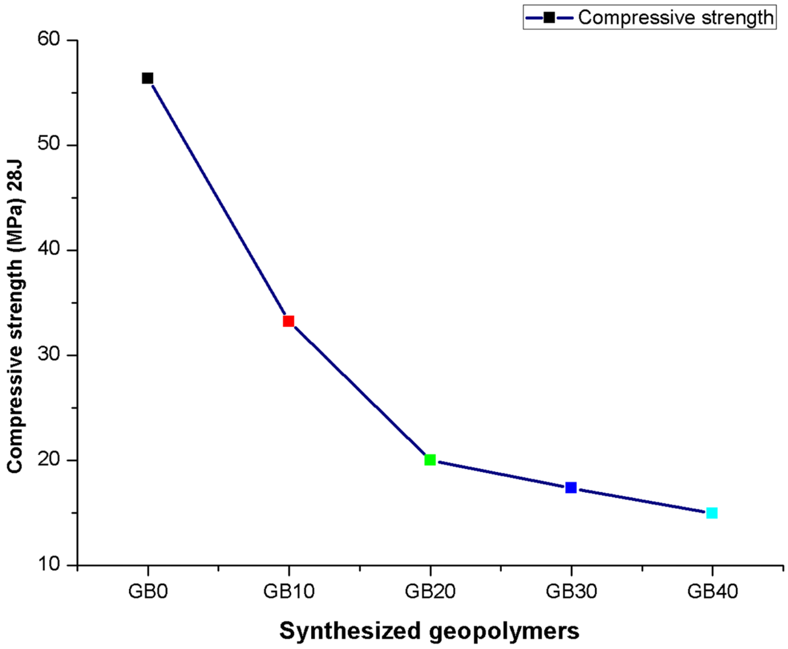


Figure 7. Effect of slag substitution by limed mud on the compressive strength of synthesized geopolymers at 28 days

ASTM 1231 (2010), on cubic samples (3.5 cm) after 14 days of curing using a hydraulic press (RP25 ATF) at a loading rate of 0.5 MPa/s. Figure 7 summarizes the effect of the slurry used on the compressive strength of the mortar. There is a reduction in compressive strength with an increasing amount of slurry added. The results showed that the addition (10%) of limed slurry decreased the compressive strength of the synthesized geopolymer from 56.352 MPa to 33.208 MPa. On the other hand, the progressive addition of sludge from 20 to 40% significantly impacted the compressive strength of the geopolymers. The Rc values are considered optimal since the addition of more than 40% slurry resulted in the cracking of the geopolymers.

Density, porosity, and water absorption

Figure 8 shows the evolution of the porosity as a function of the addition of the sludge. In comparison with the synthesized geopolymer (GB_0), the increase in the quantity of limed sludge added to the mixture influences the porosity. The substitution of 10%, 20%, 30%, 40%, gives

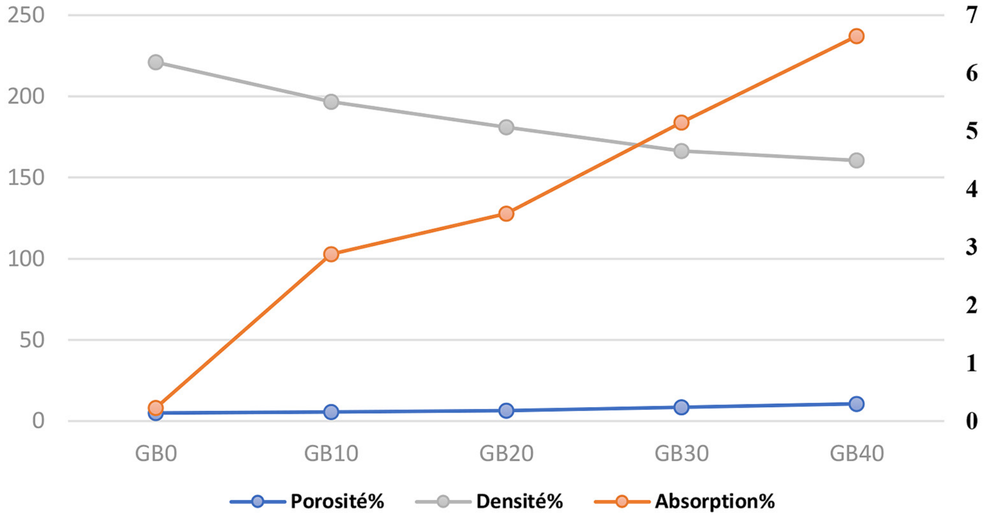


Figure 8. Evolution of porosity, density, and absorption depending on the addition of mud

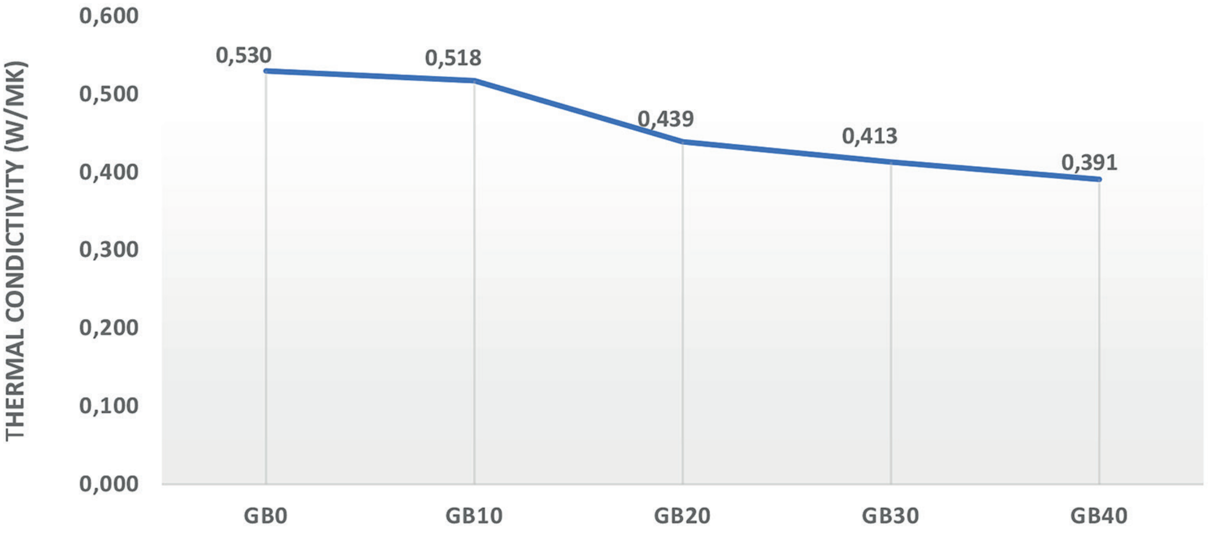


Figure 9. Thermal conductivity values of synthesized geopolymers

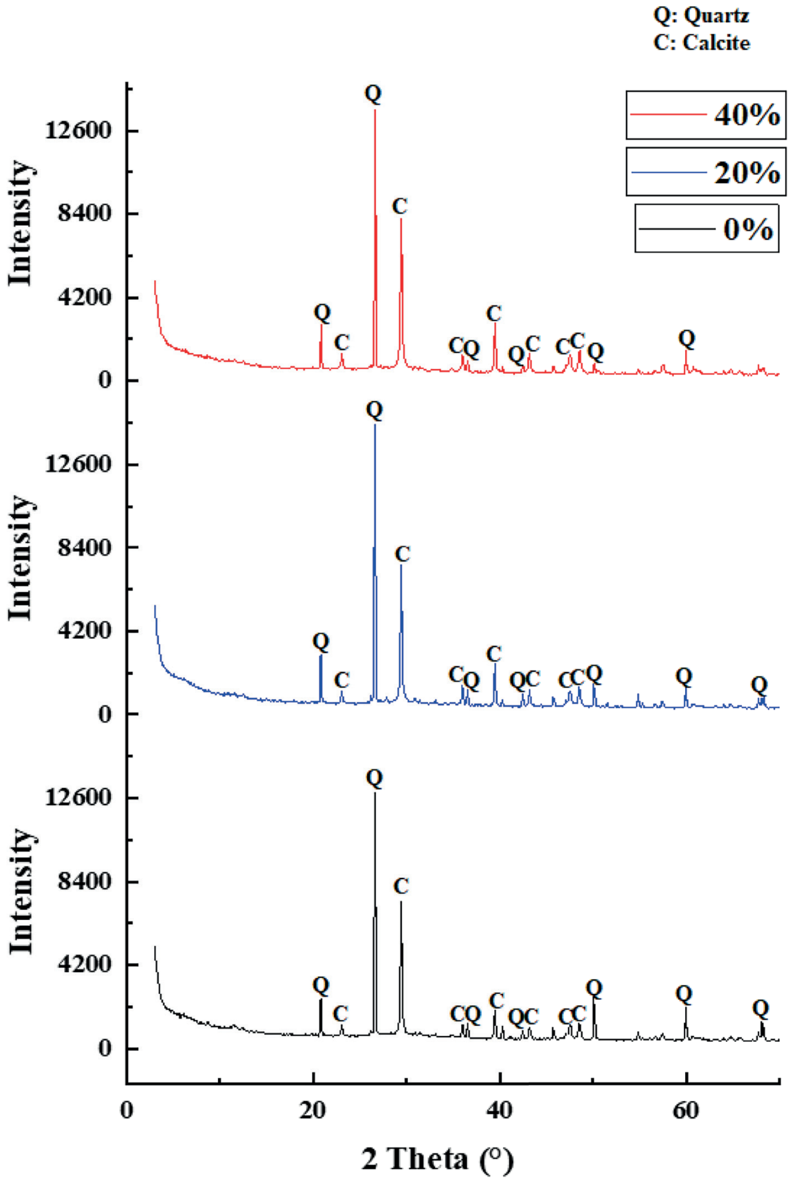


Figure 10. Mineralogical composition of geopolymer mortars (GB_0, GB_{20}, GB_{40})

respectively 5.66%, 6.48%, 8.58%, 10.65% porosity, (the control sample has a porosity of 5%). The evolution of the density was opposite to the porosity, i.e. it decreased as the slag was replaced by the limed sludge (GB_0: 2210 kg/m^3; GB_{10}: 1967 kg/m^3; GB_{20}: 1810 kg/m^3; GB_{30}: 1663 kg/m^3; GB_{40}: 1605 kg/m^3) thus the density decreases as the percentage of the added sludge increases. Substitution of 10%, 20%, 30%, 40%, gives an increase in absorption (2.88%, 3.58%, 5.15%, 6.637%) respectively.

Thermal conductivity

The increase of the limed slurry content led to a decrease of the thermal conductivity, and the substitution of the slurry by 10%, 20% 30%, and 40% of the slurry induces a decrease of the thermal conductivity successively 0.530; 0.518; 0.43; 0.413 and 0.391 W/mK. The λ value of the geopolymers followed the trend of bulk density figure and compressive strength (Figure 9). The denser the geopolymer, the higher the compressive strength as well as the λ value.

X-ray diffraction (XRD)

Figure 10 shows the mineralogical composition of geopolymer mortars containing 0%, 20% and 40% limed mud (GB_0, GB_{20}, GB_{40}). The X-ray diffractograms of the three geopolymer mortars show the dominance of two crystalline components in the geopolymer structure. The first crystalline phase is marked by quartz, coming mainly from the sand added in the mortar formulation, and the second is calcite in the blast furnace slag. However, according to Figure 10, it is difficult to determine the amorphous geopolymer phase formed during the geopolymerization reaction because of the dominance of sand in the matrix, which has a crystalline structure, and the crystallized calcite in the slag over the amorphous phase.

DISCUSSION

The objective of this study is to investigate the effect of sludge dehydrated and stabilized with hydrated lime ($Ca(OH)_2$) on the properties of geopolymer based on blast furnace slag. The substitution of slag by (10%, 20%, 30%, and 40%) dewatered sludge stabilized with lime acts significantly and according to the amount of sludge added to the synthesized geopolymers. The results of this study were discussed in light of the Previous studies in order to:

1) To study the effect of sludge only dehydrated without incineration and stabilized by slaked lime on the physicochemical properties of slag-based geopolymers synthesized at room temperature,
2) To identify the possible application of the synthesized geopolymers according to their physicomechanical properties.

Effect of sludge dewatered and stabilized by hydrated lime on the physicochemical properties of slag-based geopolymers

To improve the hardness and mechanical performance of geopolymers, it is necessary to optimize the curing conditions, by heat curing and/or inclusion of a high calcium addition, and to incorporate an aluminosilicate binder with a calcium source [Saif et al., 2022]. The absence of calcium in the geopolymer mixture may result in a longer setting time of the geopolymer [Saif et al., 2022]. In addition, the presence of slaked lime in the slurry leads to an increase in PH, which promotes the production of calcium, which reacts with silicon and aluminum to form the different phases of amorphous calcium silicate hydrate (CSH), and calcium alumino-silicate hydrate (CASH), these components give the synthesized geopolymers denser bonds. Ayoub et al. [2020] showed that the involvement of Ca^{2+} could also produce a CSH gel, thus increasing the porosity. These authors also found that calcium ions contribute to faster precipitation of the products and consequently, a denser microstructure. In our case, the partial replacement of the slag by the limed slurry ensured an abundance of dissolved calcium in the medium and consequently the hardening process is accelerated. The substitution of slag for sludge crucially affected the number, volume, and size distribution of pores produced. The macroscopic aspect showed that the porosity increases with the increase of the percentage of sludge (10 to 40%), this increase was confirmed on the microscopic scale where the porosity increases from 5.66% (addition of 10% sludge) to 10.65% (40% sludge added) (Figure 6). Aboulayt [2017] studied the properties of metakaolin-based geopolymer incorporating calcium carbonate where a heterogeneous microstructure characterized by the presence of micropores, microcracks, and a few spherical macropores is observed has demonstrated similar results. These heterogeneities are uniformly distributed in the matrix and generated during the elaboration process probably due to the retention of air and water bubbles evaporating during the drying stage. Ma et al. [2020] reported that, when slag is fully used as raw material, the increase in alkali content would lead to an increase in the characteristic pore size and a decrease in the total porosity. La concentration d'activateurs alcalins a également une grande influence sur les structures des pores en raison du changement de l'étendue de la géopolymérisation et de la teneur en eau [Chen et al., 2022]. This increase in porosity was accompanied by a decrease in density from 2210 kg/m^3 at 0% slurry and decreases to 1.605 at 40% slurry, the addition of slurry would result in an increase in porosity and a decrease in density. The densities of the synthesized geopolymers thus obtained (1605 –2210 kg/m^3) are comparable to those of Rożek [Rożek et al., 2018] (1300–1600 kg/m^3). According to Tippayasam et al. [2016], the concentration of potassium alkali and heat treatment affected the metakaolin-based geopolymer for metakaolin geopolymer (calcination are between 600 °C and 850 °C) using potassium hydroxide (1720 kg/m^3). This value is close to the value of the density 1680 kg/m^3 obtained after the addition of 30% of the slurry. It has been reported that the increase in porosity has a clear negative effect on the compressive strength of geopolymers. It is thus observed that the increase in porosity induces a decrease in compressive strength. Several researchers [Chengying et al., 2016, Hassan et al., 2020] have reported the relationship between compressive strength and porosity (or material density).

In general, a porous material reflects the presence of reduced mechanical properties due to the lower volume-to-mass ratio [Galiano et al., 2018], prepared geopolymer foam concrete with fly ash and foaming agents. The densities were about 1000 kg/m^3 and the compressive strength was between 7 and 10 MPa. As can be seen, the results obtained with the use of fly ash (density-compressive strength) are very low for the geopolymers described in these studies.

The thermal conductivity values of the synthesized geopolymers followed the trend of density (Figure 8) and compressive strength (Figure 7). A decrease in thermal conductivity was also observed with the decrease in the density of the geopolymers (0.530–0.391 W/mK).

Possible applications of synthesized geopolymers

The research conducted in this study showed that the addition of sludge (0 to 40%), produced geopolymers (GB_0 to GB_{40}). According to their physical-mechanical characteristics, they can be classified into two categories of geopolymers:

The synthesized geopolymers are dense and resistant with excellent mechanical properties, the compressive strength Rc is (GB_0: 56 MPa, GB_{10}: 33 MPa, GB_{20}: 20 MPa), these geopolymers can therefore be used in construction as a load-bearing building material.

The synthesized porous and light geopolymers with a low strength Rc (GB_{20}: 20 MPa, GB_{30}: 17 MPa, $GB_{40:}$ 14 MPa) and a conductivity GB_{20}: 0.439 W/m·K, GB_{30}: 0.413 W/m·K, GB_{40}: 0.391 W/m·K. These conductivity values are close to that of insulating materials such as vulcanized rubber: 0.36 to 0.40 W/m·K and Pozzolan Concrete: 0.44 W/m·K and can therefore substitute them. Depending on the percentage of slag substitution by sludge, geopolymers are obtained which can be used either in construction as load-bearing materials or in thermal insulation as separating materials. The physical-mechanical characteristics of the geopolymer GB_{20} (20% sludge) make it appropriate for use in construction as a load-bearing material and as a thermal insulator.

The incorporation of dewatered sludge in the production of slag-based geopolymers can allow an economic optimization of the management of sludge produced by wastewater treatment plants (installation of an incinerator, management of sludge storage, or sent to landfill), and an environmental optimization since lime stabilizes the sludge only temporarily (release of odors from the stored sludge).

CONCLUSIONS

The objective of this work is to analyze how the introduction of WWTP sludge in a geopolymer based on blast furnace slag can produce geopolymers. For each limed sludge content (from 0 to 40%), geopolymers (GB_0 to GB_{40}) are obtained, and Mechanical performance and microstructure analysis were tested (porosity, compressive strength, and thermal conductivity) taking into account the percentage of sludge added. The geopolymers showed a loss of compressive strength after the addition of the dewatered slurry, two classes of geopolymers were identified: Geopolymers (GB_0 to GB_{20}) dense and resistant can be used in construction as load-bearing materials, geopolymers (GB_{20} to GB_{40}) light and porous, can be used as thermal insulation materials. The GB_{20} geopolymer (20% mud) has physical-mechanical properties that give it the possibility to be used in both fields of construction. The combined use of sludge and slag in the production of geopolymers presents advantages: Economic (reduction of the investment cost of the sludge treatment process: absence of an incinerator and operating cost). Technical (elimination of a large quantity of sludge produced, absence of ash originating from incineration, and optimization of the operation of the WWTP), and environmental (risks of the release of odors from sludge stabilized by lime and stored).

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