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# Strength and density of metakaolin based geopolymer mortar for a use as a repair material

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**Abstract—Geopolymers are inorganic polymeric substances, which are synthesized from raw materials rich in silicates and aluminates. When these raw materials are activated by an alkaline solution, a complex aluminosilicate gel is formed due to the polymerization process. However, the proportion in which alkaline solutions are used largely controls the geopolymerization process. Thus, it is important to understand the influence of alkaline solutions on the behavior of geopolymers. This study elucidated the density and mechanical properties of geopolymer mortar obtained by alkaline activation of metakaolin. A systematic investigation was conducted to understand the effect of sodium hydroxide concentration on the feasibility of the synthesis of the Na-PSS geopolymer mortar was treated thermally of 65 ◦C for 24h using calcined kaolin at 850 °C. Six molarity variations i.e. 6, 8, 10, 12, 14 and 16 with $Na_2SiO_3$/NaOH and liquid-solid mass ratios are fixed at 2.25 and 0.68 respectively, were prepared for testing at 7, 14 and 28 days. The results show that GPM6 achieved the optimum mechanical strengths (compressive and tensile) and density of ($R_{c28}$=25.80 MPa, $R_{t28}$=12.25 MPa and $\rho_{28}$=2.350 g/cm3) respectively. The molarity of NaOH plays an important role in producing a continuous Si-O-Si network with increasing density, which is confirmed by the mechanical strength. Based on the overall results, [NaOH]= 6 M molarity is recommended on the basis of workability, compressive strength and density for concrete repair. However, the high compressive strength is not yet confirmed to promise good bond strength between the repair materials and the concrete substrate. Therefore, further analysis is needed.**

**Key-words : Geopolymer; Molarity; Alkaline Solution; Geopolymerization; Metakaolin.**

## I. INTRODUCTION

Geopolymers have attracted great interest in the academic world as alternative materials to conventional OPCconcrete, thanks to their excellent mechanical and physical characteristics, thermal stability [1] and chemical stability against the aggressive environment [2], or even the immobilization of toxic and radioactive waste [3]. In addition, geopolymers also have great potential for real recycling of industrial solid waste, reduction of $CO_2$ emissions from OPC concrete, and conservation of natural resources [4]. In the construction engineering industry, ordinary Portland cement (OPC) has been widely used as an effective binder in concrete and other construction materials. OPC production is widely recognized as one of the main sources of greenhouse gas emissions [5].

Which represents 6 to 7% of all $CO_2$ emissions, as shown by the International Energy Agency (IEA) [6]. However, global demand for portland cement will increase by almost 200% in 2050 [7]–[9]. To mitigate these emissions and the problems associated with them, a new type of alternative, green and environmentally friendly material, called geopolymer concrete, is being produced [8]. In general, geopolymer mortars (GPMs) are considered to be much more sustainable than CPOs in terms of low energy requirement for its production with significantly lower CO2 emissions [10]–[12]. Furthermore, despite some prevention techniques, the degradation of civil cement or concrete infrastructure and the shortening of the age of structures are inevitable sustainability problems worldwide[13]–[15]. Therefore, new materials are being sought to meet not only the strength requirements necessary for the basic

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performance of buildings but also higher sustainability requirements.

Geopolymer is a low carbon material first proposed by Davidovits in 1978 [16], [17]. Geopolymeric cement can be produced by a reaction between industrial by-products (usually fly ash, slag, metakaolin, etc.) and alkaline solutions. The particular structure of geopolymers makes them durable materials with many mechanical [18] and durable [19] advantages. Therefore, mineral geopolymers have been described as the most promising green cement material of the 21st century [20].

Hydroxide solutions are the most used activators in the production of geopolymers. The activator solution depends on the alkali metal used; then, the degree of hydration of the cation and the alkalinity are determining for the dissolution of the aluminosilicate source [21]. When using NaOH to activate metakaolin, the experimental design, influencing parameters and prediction of mechanical properties are uncommon [22]. Due to the small size of Na+ relative to $K^+$ in this system, which favored the formation of strong pairs with smaller silicate oligomers, the matrices showed greater compressive strength and higher specific surface area; in addition, a reduced degree of crystallinity and resistance to hydrochloric acid attack [23].

Over the years, geopolymers have been exploited as protective coating materials for marine concrete and transportation infrastructure [24], [25]. However, the adhesion strength between the substrate concrete and the repair material [26], [27] plays a decisive role in the selection of geopolymers as repair materials. The properties of GP [28], [29], including modulus of elasticity, Poisson's ratio and tensile strength, are similar to those of OPC concrete. This clearly demonstrates the compatibility between GP and OPC concrete. Additionally, like conventional concrete, GP can cure at room temperature [30]–[32]. The degree of degradation of GP when soaked in an acid solution is significantly lower than that of OPC concrete [33], [34]. Additionally, they possess low permeability and excellent anti-corrosion properties which are beneficial for effective bonding with cement paste and mortar [35]. Geopolymers can be implemented using the same equipment and practices used for OPC concrete to repair deteriorated infrastructure such as manholes, pipes and chambers [36]. The high temperature stability of geopolymers makes them an excellent alternative to epoxy resins [37]. More significantly, the production of geopolymeric mortars or concretes based on FA releases 80 to 90% less $CO_2$ than their OPC counterparts [38]–[41]. All these notable merits make geopolymer an excellent candidate for pavement repair. Despite numerous efforts in the synthesis and characterization of geopolymers, their durability as a repair material is far from understood.

## II.EXPERIMENTAL PROGRAM

### *A.MATERIALS*

Metakaolin has been used as aluminosilicate raw material. Metakaolin was obtained by calcining kaolin called KT from El Milia, supplied by the SAOLKA company (Algeria), at 850 °C for 5 h in a static oven. The limestone sand used is crushed limestone sand with a fraction of 0/5 mm coming from a quarry located north of the town of Laghouat (southern Algeria). The chemical composition of metakaolin and calcareous sand determined by the X-ray fluorescence spectrometer (XRF) is shown in Table 1. The main physical characteristics of calcareous sand are summarized in Table 2 (measured according to current standards [42]–[44]). Chemical analysis showed that the sand used is purely calcareous in nature and does not contain any harmful materials for the manufacture of mortars. Furthermore, XRD analysis of the sand used reveals the presence of the following minerals: calcite ($CaCO_3$) almost exclusively and quartz and dolomite in traces as shown in Fig. 1. It is well calibrated and its grain size curve is located within the grain size zone of normal sand, as shown in Fig. 2.

The sodium hydroxide powder (NaOH) was caustic soda micro-pearls and 99% purity from the Coprochim brand, manufactured in Algeria. The sodium silicate solution ($Na_2SiO_3$) was supplied by the company Galaxy-Chimie. M'sila, Algeria, with a chemical composition of 26% $SiO_2$, 13% $Na_2O$ and 61% $H_2O$ and a $SiO_2/Na_2O$ modulus of 2.1, a specific weight at 20°C = 1.46 and a viscosity at 20°C = 0.4 Pa-s.

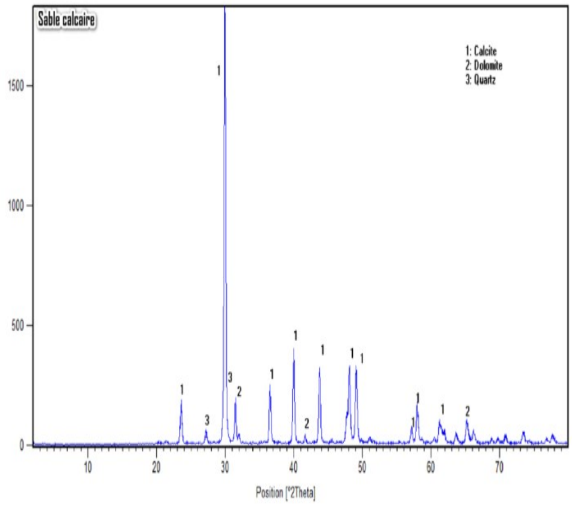

Fig. 1 Analysis of the X-ray diffractogram of the limestone sand

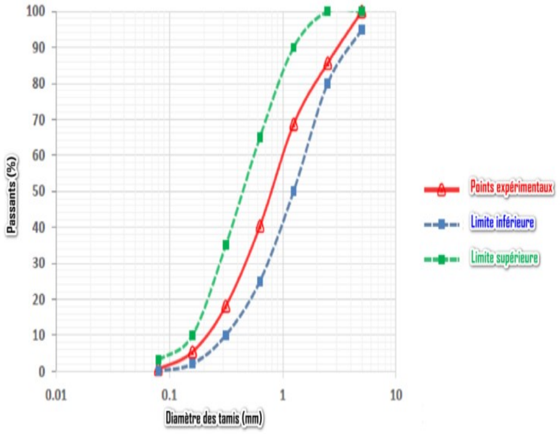

Fig. 2 Grain size curve of the limestone sand

TABLEAU I
CHEMICAL AND MINERALOGICAL COMPOSITION OF METAKAOLIN (MK850) AND LIMESTONE SAND

| XRF of metakaolin (MK850) and limestone sand | | |
|---|---|---|
| Oxides | MK850 | limestone sand |
| **CaO** | 3.27 | 54.82 |
| **$SiO_2$** | 68.44 | 0.51 |
| **$Al_2O_3$** | 19.09 | 0.33 |
| **$Fe_2O_3$** | - | 0.31 |
| **$K_2O$** | 3.86 | 0.001 |
| **$Na_2O$** | 0.18 | 0.16 |
| **Others** | 5.16 | 43.87 |

TABLEAU II
MAIN CHARACTERISTICS OF LIMESTONE SAND

| Characteristics | Standard | Values |
|---|---|---|
| Absolute density(g/cm$^3$) | NF EN 1097-6[45] | 2.59 |
| Apparent density (g/cm$^3$) | - | 1.49 |
| Sand equivalent : SE(%) | NF EN 933-8[46] | 66 |
| % of elements <0.08 mm | NF EN 933-1[47] | 7.50 |
| Fineness module: FM | NF EN 12620 [48] | 3.21 |
| Absorption coefficient: AC 24 (%) | NF EN 1097-6[45] | 4.70 |

### *B. Preparation of metakaolin-based geopolymer mortars*

Mixtures of metakaolin-based geopolymer mortars with different concentrations of sodium hydroxide "NaOH" (6, 8, 10, 12, 14 and 16) were prepared, as shown in Table 3.

TABLEAU III
FORMULATION OF GEOPOLYMER MORTARS

| Sample | $Na_2SiO_3$ / NaOH | L/S * | [NaOH] | Water/GPM |
|---|---|---|---|---|
| **$GPM_6$** | 2.25 | 0.68 | **6** | 0.4 |
| **$GPM_8$** | 2.25 | 0.68 | **8** | 0.4 |
| **$GPM_{10}$** | 2.25 | 0.68 | **10** | 0.4 |
| **$GPM_{12}$** | 2.25 | 0.68 | **12** | 0.4 |
| **$GPM_{14}$** | 2.25 | 0.68 | **14** | 0.4 |
| **$GPM_{16}$** | 2.25 | 0.68 | **16** | 0.4 |

*L/S : liquid-to-solid ratio

The NaOH solution was prepared in a volumetric flask using distilled water. The alkaline activator solution was formed by mixing solutions of $Na_2SiO_3$ and NaOH with a mass ratio of $Na_2SiO_3$/NaOH of 2.25 until clear and homogeneous solutions were obtained. The molarity of sodium hydroxide will be varied from 6M to 16M, the liquid/solid mass ratio and the Water/GPM mass ratio (mass of geopolymer) used in this study was 0.4 and 0.68 respectively. As the selected metakaolin precursors have a fixed $SiO_2$/$Al_2O_3$ ratio. The metakaolin (MK850) and the alkaline solution are slowly added to the mixer and mixed for 3 min then adding the limestone sand. The fresh cement is quickly poured into the 4 × 4 × 16 cm 3 steel prismatic molds. All samples are vibrated for 2 minutes.

The samples were covered with a thin plastic film to prevent water evaporation. The samples were dried at room temperature for 24 hours and in an oven at 65 °C for an additional 24 hours. The prepared specimens were stored under normal laboratory conditions (25 ± 2 °C and 50 ± 10% of relative humidity). The mechanical resistance of the samples was tested after 7, 14 and 28 days.

### *C. Objectifs*

The main objective of this paper is to study the effect of NaOH concentration on the physical and mechanical properties of metakaolin-based geopolymer Na-PSS.

### D.*Methods*

To study the tensile strength of mortars, tensile tests "three-point bending test" were carried out according to standard EN 196 – 1 [49], on three prismatic samples with a size of 4 × 4 × 16 cm 3 by composition using a “CONTROLS” type press with a maximum load of 100 kN with load increases of 50 N/s coupled to a microcomputer equipped with software for calculating loads (e.g. Fig. 3) test consists of subjecting prismatic samples resting on two points of support to an increasing load concentrated at the center until rupture. The tensile strength by bending is calculated by the following formula:

$$\sigma = \frac{3.F.l}{2b^3} \qquad (1)$$

The six half-samples resulting from this test were then subjected to a compression test on a section of 4 × 4 cm 2 according to European standard EN 196-1 [49]. These two tests were carried out at ages 7,14 and 28 days.

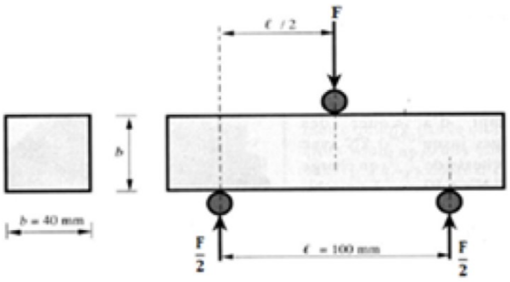

Fig. 3 Experimental tensile test for the mortar studied

## III. RESULTSAND DISCUSSIONS

### *A. Apparent density*

The bulk density of metakaolin-based geopolymer mortars with different NaOH concentrations (6M-16M) is shown in Fig. 4. The measured density values were between 2.340 and 2.225 g/cm3 for GPM with a NaOH molarity between 6M and 16M. It can be clearly seen that GPM6 showed the highest bulk density (2.340 g/cm3) while the lowest bulk density is observed for GPM12 of 2.225 g/cm$^3$.

After the molarity value of NaOH 6 M, the apparent density is varied inversely proportionally with the increase in the molarity of NaOH can be explained by the excess of hydroxide ions necessary for the activation of MK850 in the case of use of high NaOH concentrations. Indeed, this excess caused the precipitation of the aluminosilicate gel at a very early stage. Therefore, geopolymerization was blocked, leading to porous geopolymers; as a result, it limited the flow of the geopolymer and therefore reduced the setting time. The reduction in workability would probably contribute to poor compaction of the geopolymer paste and therefore to a lower density [50]. This was confirmed by Azzahran et al. [51] where the density dropped by increasing the NaOH concentration up to 12M.

Beyond the molarity value of 12 M NaOH, the apparent density is increased with the molarity of NaOH. In general, the solubility and dissolution of aluminosilicates were strongly affected by the molarity of NaOH. Molarity increases the dissociation of active species from the raw material and thus produces a greater amount of geopolymer network [52], [53]. Indeed, increasing the concentration of NaOH increases the apparent density of geopolymer mortars [54].

The geopolymeric structure mainly depends on the dissolution of metakaolinite particles, which is directly affected by the concentration of the NaOH solution. A NaOH solution with a higher concentration provides better dissolution capacity of the metakaolinite particles (providing sufficient Na+ and OH$^-$ ions for a complete geopolymerization reaction) and produces a more reactive bond for the monomer, which increases the intermolecular bond strength of the geopolymer [51], [55]. Apparent density data clearly confirm this trend.

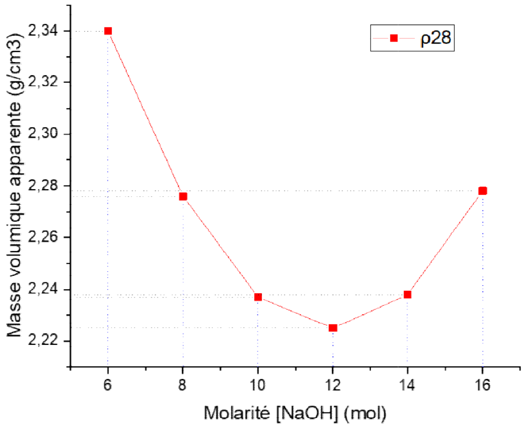

Fig. 4 Evolution of apparent density at 28 days according to theNaOH concentration

### B. Tensile strength

The tensile strength result of metakaolin-based geopolymer mortars with different NaOH concentrations (6M-16M) is shown in Fig. 5. In general, the tensile strength values of the geopolymer samples (Rt7, Rt14 and Rt28) increased to reach the values 9.17, 8.84 and 13.38 MPa respectively.

A linear increase in Rt7 and Rt14 which are varied by a positive correlation with the increase in the molarity of NaOH, while after 14 M we see a decrease in flexural strength for Rt14 of GPM14 and GPM16 compared to Rt7 of the same samples, this decrease probably occurred due to a PH disturbance [56].

At the molarity range of 6 M to 10 M a reduction of Rt28. The high viscosity of the high molarity NaOH solution would disrupt the leaching of Si and Al ions from the aluminosilicate sources and thus lead to premature precipitation of the geopolymer matrix and deterioration of the mechanical properties of the final product due to remnants of precursor materials [57]. At high molarity of NaOH (14M), the geopolymerization process could be disrupted due to the excessive amount of OH- ions present in the geopolymer matrix. This statement was confirmed by Alonso & Palomo [58] who observed that when the molarity of NaOH is too high and exceeds the optimal level, the formation of the geopolymer matrix is delayed due to the stability of the ionic species and the mobility ions, which leads to a drop in the bending resistance, after 10 M the resistance Rt28 increased with the increase in the concentration of NaOH. This result can be clarified by the activation of the chemical reaction of the internal components of Si and Al, caused by the increased breakage of the glassy chain of fly ash, influenced by the high alkalinity resulting from the increased molarity of NaOH [59]. The highest concentration of NaOH positively influences the dissolution of the starting material and thus the formation of the amorphous N-A-S-H gel, which is responsible for the mechanical properties of the obtained geopolymers [60]. Most obviously, the obtained result is attributed to the increase of sodium ions in the matrix, which was significant for the geopolymerization because the sodium ions were used to equalize the charges and created the rigid networks of aluminosilicate as agent binding in the mixture [61].

This flexural strength result is consistent with the bulk density of fly ash geopolymer tiles obtained in Fig. 4 where a more compact density indicates a higher flexural strength.

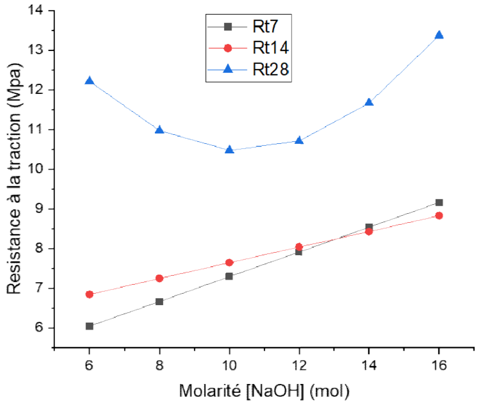

Fig. 5 Evolution of the bending resistance (Rt7, Rt14 and Rt28) according to the NaOH concentration

### C. Compressive strength

The compressive strength results of metakaolin-based geopolymer mortars with different concentrations of NaOH (6M-16M) are shown in Fig. 6. In general, the compressive strength values of the geopolymer samples (Rc7, Rc14 and Rc28) increased to reach the values (19.80, 19.96 and 25.80) MPa respectively.

A linear increase in Rc7 and Rc14 which are varied by a positive correlation with the increase in NaOH molarity. After the value 14 M, a drop in resistance Rc14 compared to Rc7 up to the value of [NaOH]=16 M, probably because of a PH disturbance, The stability zone of the surface species > T-O-Na depends of pH and is located at extremely high pH values, which relates to aluminol groups [56].

Overall, the compressive strength Rc28 of the geopolymer samples reduced from 6 to 12M (16.27 MPa), but above [NaOH]=12 M, the strength Rc28 increased with increasing NaOH concentration. The Rc28 resistance of geopolymer mortar depends on the NaOH concentration, because it affects the dissolution of Si4$^{+}$ and Al3$^{+}$ ions in MK850 particles [62]. The increased resistance at 6 M is due to the OH$^{-}$ concentration which is high enough to accelerate the dissolution and hydrolysis process [63]. Optimal compressive strength is obtained when NaOH is sufficient to ensure charge balance for the substitution of tetrahedral Si by Al.

On the other hand, it is observed that a higher concentration of NaOH (>10M) in the geopolymer mixture gives lower strength. Excessive concentration of hydroxide ions caused precipitation of aluminosilicate gel at a very early stage [64]. Additionally, Lee and al. [65] noticed that the setting time decreased with increasing NaOH concentration. Reducing the setting time is believed to decrease the strength of the geopolymer due to lack of time for the geopolymerization process. In this study, the optimal concentration of NaOH to prepare metakaolin geopolymers was 6M. The concentration of the NaOH solution is lower than that of Patankar et al [66] who obtained 12M as the optimum concentration. This could be due to the different chemical composition of the aluminosilicate materials used. It is well known that the Si/Al and Na/Al ratio are key factors that can affect the mechanical, physical and microstructural properties of geopolymers. The resistance obtained is linked to the structure and density of the geopolymers. The compressive strength results correspond to the bulk density values in Figs. 4 and 6. Higher density contributes to greater compressive strength [67].

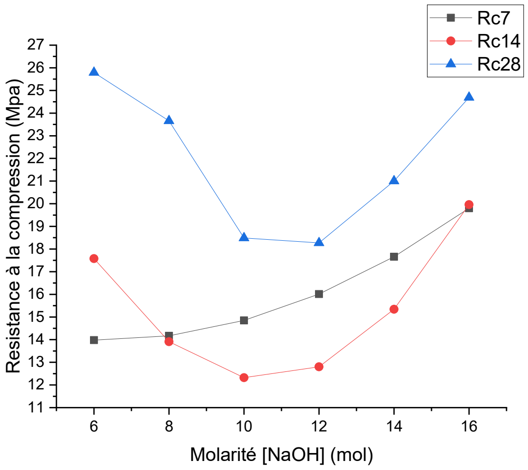

Fig. 6 Evolution of compressive strength (Rc7, Rc14 and Rc28) as a function of NaOH concentration

## IV.CONCLUSIONS

This article presents the effect of various concentrations of NaOH solution (6 - 16M) on geopolymer Na-PSS mortars based on Metakaolin.

The bulk density of the metakaolin-based geopolymer mortar showed a range of bulk density from 2.225 g/cm3 to 2.340 g/cm3.

For the present study, the optimum concentration of NaOH was concluded to be 6M for the formation of geopolymer Na-PSS. It was assumed that there were sufficient Na+ and OH- ions for a complete geopolymerization reaction at a concentration of 6M NaOH to produce a denser geopolymer. The strength results were consistent with the density values. The NaOH content can affect the dissolution of Si4$^{+}$ and Al3$^{+}$ ions of metakaolin (MK850) and the dissolution and hydrolysis process. When the NaOH content is sufficient to ensure charge balance for possible substitution of tetrahedral Si by Al, the optimal compressive strength is achieved.

The mechanical properties of geopolymers indicated by flexural strength show that the molarity of NaOH plays an important role in improving the properties of metakaolin geopolymer Na-PSS and makes the geopolymer an excellent material for it to be used consider it as a repair mortar.

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