Effect of Metakaolin Addition on The Mechanical Performance and Durability of Granulated Blast Furnace Slag Based Geopolymer Mortar with Micro-Encapsulated Phase Change Materials

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Abstract
Incorporating microcapsule phase change materials (MPCM) into geopolymer is one of the most successful solutions for enhancing building thermal comfort and replacing Portland cement-based materials. Although MPCM improves the thermal capacity of the cementitious matrix, whether it's made of cement or geopolymer, it presents a number of disadvantages in terms of mechanical and physical performance. Several researchers have pointed out that this scientific subject remains unresolved. The purpose of this study is to investigate the influence of 10% and 20% metakaolin (MK) inclusions on the mechanical properties and durability of geopolymer-MPCM mortars based on granulated blast furnace slag (GBFS) and to compare them with Portland cement-MPCM based mortars. The results show that the addition of two proportions of metakaolin is able to compensate well for the loss of mechanical strength associated with the addition of MPCM. Thus, up to 20% MPCM, the addition of metakaolin increases compressive strength by approximately 10 MPa. Compared to Portland-MPCM cement mortars, all geopolymer-MPCM mortars show higher compressive strength, better workability and lower porosity. Finally, in terms of durability evaluation, the resistivity measurements reveal that the risk of corrosion of the cement-based mortar on the steel bars is negligible, while the risk of corrosion of the geopolymer-based mortar on the steel bars is low.

1. Introduction
The construction sector accounts for 40% of final energy consumption and 30% of greenhouse gas emissions [1]. Between 2012 and 2040, this energy consumption is expected to increase by about 48%, mainly due to the rapid expansion of some non-OECD (Organization for Economic Cooperation and Development) countries [2]. Drissi et al [2] pointed out that most of the energy consumption is reserved for improving the thermal comfort of the building during the hot and cold seasons.

In fact, certain types of materials can maintain a comfortable indoor temperature and reduce the energy consumption of buildings. Microcapsulated phase change materials (MPCM) are among these types of materials that rely on latent heat storage systems [3]. The reason is that MPCM stores a large amount of heat energy in the form of a constant temperature (an ambient temperature close to human comfort) during its phase change (solid-liquid) process, thereby preventing the heat flow from entering the peak period of construction [4]. This helps to improve thermal comfort and reduce energy consumption for heating and cooling [5].

In order to use MPCM properly, it must be incorporated into building materials such as concrete, plaster and plaster. Portland concrete is considered the most widely used construction material in the world [6]. It has a high volume and surface area exposed to the indoor environment, a high thermal energy storage capacity, and high mechanical strength. Therefore, it is a promising candidate for use with MPCM. Some researchers showed the beneficial effects of using MPCM with Portland concrete on reducing the use of heating and cooling systems in buildings [7, 8]. However, the manufacture of Portland cement to produce concrete is responsible for 7% of CO2 emissions [9] worldwide and causes other pollution in the air, water, etc.

Compared to Portland concrete, the use of geopolymers has attracted a lot of interest in the research field because of their low environmental impact and their superior technical advantages compared to Portland cement-based materials. Geopolymer is the result of activation of aluminosilicate materials by alkaline solutions, these aluminosilicate materials are industrial by-products or types of clay, such as granulated blast furnace slag (GBFS), metakaolin (MK), fly ash, and red mud, etc [10]. The use of these materials to replace Portland cement will reduce CO2 emissions and waste produced by the industries, which will contribute to reduce the environmental impacts of this sector. The study of Wang and [11] showed that CO2 emissions caused by geopolymer production are reduced by about 70 to 80% compared to Portland cement manufacturing. In addition, geopolymer has several advantages over Portland cement-based materials, such as higher initial mechanical strength, low drying shrinkage, high fire resistance, shorter curing time, superior acid resistance and better durability [12-15].

Several researchers have shown in the last few years that the incorporation of MPCM in geopolymers could be a promising solution to reduce CO2 emissions related to energy consumption in buildings and Portland cement production. Shadnia et al (2015) [16] constructed three small cells from fly ash-based geopolymer mortars, among these
three cells two containing MPCM. The measured internal temperature showed a reduction of 4.5 and 5.5 °C for both MPCM geopolymer cement compared to the reference cell. Second, Cao et al (2019) [17] numerically investigated the influence of different climatic conditions on the energy efficiency of a wall constructed of geopolymer-MPCM concrete. The reduction in the wall's interior temperature was about 3 °C and the reduction in energy consumption was 25%, while maintaining the interior temperature at 23 °C.

In contrast, the incorporation of MPCM into Portland cement or geopolymer-based materials affects its mechanical performance. This scientific issue is unresolved to date and has been highlighted by several researchers [4, 16–18]. Cao et al [18] studied the effect of MPCM addition on the compressive strengths of Portland cement-based concrete and geopolymer-based concrete. Their results show that although MPCM generated an increase in the heat capacity of both types of concrete studied up to the value of 1500 (J/kg °C), the compressive strengths were reduced by about 42 and 51%. The same observations were reported by Shadnia et al [16] who reported a decrease in compressive strength of up to 25%. Furthermore, it is important to note that the study of the durability of geopolymers with MPCM is very limited in the research field because this type of material was first introduced in the last decade.

However, the activation of aluminosilicate materials with low calcium content, as in the case of fly ash or metakaolin, produces a matrix composed of a gel which is called NASH (sodium alumina silicate hydrate). This gel is the result of the chemical reaction between Na2O-Al2O3-SiO2-H2O. On the other hand, the activation of a material rich in calcium, as in the case of blast furnace slag, produces the CASH (calcium alumina silicate hydrate) gel, which is the result of the chemical reaction between Na2O-CaO-Al2O3-SiO2-H2O [19]. Indeed, researchers have shown that the combination of two materials rich in SiO2, Al2O3 and CaO can present advantages compared to the activation of a single aluminosilicate material. Among these advantages is the improvement of the mechanical strength and durability of the total matrix of the geopolymer [19, 20]. PHOO-NGERNEKHAM et al [21] studied the effect of adding a certain concentration of SiO2 and Al2O3 nanoparticles in a geopolymer matrix. Their study concluded that the compressive strength, flexural strength, and modulus of elasticity of the geopolymer pastes were increased due to the formation of CASH and NASH gels, which led to the filling of the pores to form a dense and strong geopolymer matrix.

The same observations were confirmed by the studies of BERNAL et al [22] and HUSEIEN et al [23], who used a substitution of 10 and 20% metakaolin in the matrix of blast furnace slag-based geopolymers and found that the mechanical performance and durability improved due to the appearance of these gels.

Most of the research studies on geopolymer-MPCM focus on a geopolymer based on a single base material, such as granulated blast furnace slag, fly ash, or metakaolin. There are a limited number of studies that combine two materials (rich in calcium, silicon and aluminum) with MPCM, and no research on the inclusion of metakaolin in a matrix based on blast furnace slag.

The purpose of this study is to examine the effect of including 10 and 20% metakaolin in geopolymer-MCP mortars based on blast furnace slag and to compare them to Portland-MPCM cement-based reference mortars with respect to mechanical performance and durability. Several characterizations were performed in this study, such as workability, compressive strength, dynamic Young’s modulus and dynamic shear modulus, morphological analyses, electrical resistivity and total water porosity.

2. Materials and experiments design

2.1. Materials

The MPCM considered in this study is in the form of a white spherical micro-encapsulate marketed by the laboratory Microteck–United States. Its technical name is Nextek 28 D. Its melting temperature is equal to 28 °C while its density is equal to 0.84 g/cm3. Figure 1 was obtained by SEM. It shows the material morphology. Indeed, according to the images, its grains show spherical shapes and sizes between 2.19 and 38.22 μm.

Figure 1. Morphology of MPCM

The granulated blast furnace slag is provided free of charge by the company ECOCEM in France and the metakaolin was provided by the company KENZA1 (ecological materials) in France. The cement used is CEM II (32.5). The chemical compositions and physical properties of cement II, GGBS and MK are presented in Table 1.

Table 1. Chemical composition and physical properties of CEM II, GGBS and MK.

<table>
<thead>
<tr>
<th>Chemical composition (%)</th>
<th>CEM II</th>
<th>GGBS</th>
<th>MK</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO2</td>
<td>7.47</td>
<td>37.3</td>
<td>55</td>
</tr>
<tr>
<td>Al2O3</td>
<td>2.18</td>
<td>10.7</td>
<td>41</td>
</tr>
<tr>
<td>Fe2O3</td>
<td>2.84</td>
<td>0.2</td>
<td>1.2</td>
</tr>
<tr>
<td>CaO</td>
<td>69.02</td>
<td>43.0</td>
<td>0.1</td>
</tr>
<tr>
<td>MgO</td>
<td>-</td>
<td>6.5</td>
<td>0.2</td>
</tr>
<tr>
<td>TiO2</td>
<td>-</td>
<td>0.7</td>
<td>0.4</td>
</tr>
<tr>
<td>(Na2O + K2O)eq</td>
<td>-</td>
<td>0.8</td>
<td>1.8</td>
</tr>
<tr>
<td>Blaine specific surface area (m²/g)</td>
<td>0.37</td>
<td>0.445</td>
<td>17</td>
</tr>
<tr>
<td>Verage grain size (μm)</td>
<td>8.47</td>
<td>13.25</td>
<td>7.13</td>
</tr>
</tbody>
</table>

The activation solution is a mixture of sodium silicate and sodium hydroxide. According to the supplier, the composition by mass of the sodium silicate is 27.53% SiO2, 11.47% Na2O and 61% H2O. The sodium hydroxide NaOH is a caustic soda of 98% purity. Both solutions were supplied by the company E2EM in France.

A sand standardized CEN NF 196-1 with a density of 2.6 g/cm3. This type of sand is generally used for laboratory tests. The provider is the same as that of the two alkaline solutions. The purpose of using standardized sand is to eliminate the secondary effects on the binder of unfavorable impurities that natural sand might contain.

2.2. Mixing method and curing condition

Twelve formulations were investigated in this study, three based on standardized mortar and nine based on geopolymer mortar. The water/binder ratio of 0.5 and the sand/binder ratio of 3 have been fixed for both types of mortars. The binder (equivalent to cement) of geopolymer is considered as the total of GGBS, MK and the alkaline solution (the solid part). We fixed a mass ratio of 3 of (GGBS+MK)/SA, with SA corresponding to the solid part of the alkaline solution. These choices are recommended by the study of Hassanoui et al (2019) [24].

The inclusion percentages of MK are 0, 10 and 20%. The ratio of sodium silicate (SS) to sodium hydroxide (SS/NaOH) is 2.5 in all geopolymer formulations. This high sodium silicate content will maintain an optimal amount of silica after the addition of our higher percentage of metakaolin (20% MK) [22]. The MPCM will replace the same percentage by volume of sand with three concentrations, such as 0, 5 and 10% in the two types of mortar.

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Table 2 shows the mixing proportions in kg/m³ where the first three formulations are based on Portland cement mortar while the last nine are based on geopolymer mortar. The amount of water in the two alkaline solutions is considered to have a water/binder ratio equal to 0.5.

The standardized mortars based on Portland cement were manufactured according to the NF 196-1 standard [25]. They were chosen as a reference in order to compare them with the mortars based on geopolymer. However, for the geopolymer, the GBFS and MK were mixed with the alkaline solution and water for 90 seconds to have a homogeneous paste. The mixture was followed by the addition of sand and mixed for 5 minutes, and finally the MPCM was added and mixed for 2 minutes. After the mixing procedures, the standardized mortars and geopolymers were used for the measurement of workability. After this step, the molding was carried out in 40 × 40 × 160 mm³ specimens and vibrated using an impact table. They were stored in an air-conditioned room (temperature 20°C and relative humidity 50%) for 48 hours before demolding.

2.3. Characterization methods

2.3.1. Workability

Workability test was carried out directly after mixing according to standard NF P18-452 [26]. The test consists in measuring the flow time of mortars under the effect of the vibration caused by the vibrator.

2.3.2. Mechanical properties

Compressive strengths were realized following the NF EN 196-1 [27] standard, these tests were carried out on six samples in order to ensure a good repeatability.

Dynamic Young modulus and dynamic shear modulus were measured by a non-destructive impulse excitation technique using the Grindosonic® device (figure 2). These measurements were performed in accordance with ASTM E 1876-01 [28].

These measurements were performed on three samples of each formulation at 2, 7, 14, 21, 28, 56 and 90 days.

Dynamic Young’s modulus and dynamic (Edyn) shear modulus (Gdyn) are calculated using the equations 2 and 3 and by using the flexure (ff) and torsion (ft) frequencies obtained during measurements with the dimensions and weight of the samples:

\[ E_{\text{dyn}} = 0.9465 \frac{m_l^2}{b l^2} f_{\text{f}} \]  
\[ G_{\text{dyn}} = 4 L m_l^2 R \frac{f_{\text{t}}}{b l} \]  

Where m, L, b, t represent the mass (g), length (mm), width (mm) and the thickness (mm) of the sample. T and R represent the coefficients of correction, which can be defined by following certain steps in ASTM E 1876-01 [28].

2.3.3. Morphological analyses

Morphological analyses were performed using a ZEISS EVÒ®4 Scanning Electron Microscopy (SEM). The analyses were performed on fractured samples after 28 days of curing.

2.3.4. Durability

2.3.4.1. Electrical resistivity

The structure and connectivity of the concrete pores of a material are an important indicator for determining the transport properties and, consequently, the durability characteristics. Indeed, the electrical resistivity of concrete is a measurement method that provides a good indication of its permeability by providing an indication of the pore connectivity and tortuosity of the pore network of a material, which are important factors influencing the penetration of aggressive ions into concrete. It is defined as a volumetric property that indicates the ability to carry an electrical charge through the material [29].

Concrete containing fewer interconnected pores with a more tortuous structure will exhibit higher electrical resistivity since it will be more difficult for ions to pass through these types of pore networks. In addition, several researchers have shown that measuring the electrical resistivity can also predict the corrosion rate of reinforcement [30–32]. Porder et al. [33] (2000) cited that low concrete resistivity correlates with rapid chloride penetration and high corrosion rate, while Bonnet et al. [34] (2018) showed a linear correlation between electrical resistivity and gas permeability of concrete.
Indeed, the electrical resistivity was measured with a non-descritive method and using the Wenner resistivity device [35]. The apparatus consists of 4 electrodes which are arranged linearly and spaced at a constant distance (a) which is equal to 5 cm (figure 3).

The principle of this method is to inject an electric current, I, between the two external electrodes. Then, the electric potential is measured between these two internal electrodes. The electrical resistivity is calculated by the control acquisition unit, based on equation (4), where \( \rho \) is the resistivity \((\Omega \times m)\), a is the distance interval between the electrodes \((m)\), V is the voltage \((\text{volts})\), and I is the intensity of the current injected into the sample.

\[
\rho = 2\pi \times a \times \frac{V}{I} \tag{4}
\]

![Figure 3. Non-destructive measurements: Wenner resistivity method](image)

### 2.3.4.2. Water porosity

Water porosity was determined according to the NF P 18-659 standard [36]. The relation to calculate the porosity is as follows:

\[
P = \frac{M_{\text{MPPM}} - M_{\text{dry}}}{M_{\text{w}}} \times 100 \tag{1}
\]

Mair is the mass of the sample saturated at the saturated surface-dried state, Mdry is the mass of the sample at oven dried state while Mw is the mass of mortar specimen saturated in water.

### 3. Results and discussion

#### 3.1. Workability

The flow time of the various formulations investigated is represented in Figure 4. The MPCM concentrations in the two types of mortar (cement-based and geopolymer) and the addition of MK in the geopolymer mortar were the only variables that differ. Cement mortar without MPCM exhibited a flow time of about 7 seconds, that was confirmed by Hasnaoui et al (2019) [24].

According to Muhammad et al (2019) [37] the difference in the flow time of cement-based mortars, the geopolymer-MPCM mortars showed good workability when utilizing up to 10% MPCM and 20% MK, with a maximum flow time of 4.95 seconds, which is much less than the flow time of Portland cement-based mortar.

#### 3.2. Mechanical properties

##### 3.2.1. Compressive strength

Figure 5 shows the compressive strength results after 28 and 90 days of curing. It appears that there is no significant difference between the compressive strengths between 28 and 90 days for the geopolymer mortar, contrary to the cement-based mortar which shows an increase in its compressive strength between 28 and 90 days. Indeed, the geopolymer can achieve most of its mechanical strength in the first days of its curing due to its strong chemical bond [22].

Increasing the MPCM rate (5 and 10%) reduces the mechanical strengths of the cement and geopolymer mortars to 25 and 40 MPA after 28 days, respectively. For the 90-day period, a comparable effect was observed. The reduction in compressive strength is due to the effect of replacing MPCM with sand, as MPCM has low stiffness and mechanical strength compared to sand and can easily fracture under compressive force [16, 18]. On the other hand, the increase in matrix porosity after MPCM incorporation is one of the causes of the reduction in mechanical strength as observed in the results detailed in Section 3.4.2.

The mechanical strength of the geopolymer mortar with 0% MK was fairly high. The high activator content, according to NAZARI et al (2015) [38], is the reason for this increase. This is due to the high PH of the solution causing excellent dissolution of silica ions and alumina from GBFS, resulting in a very mechanically efficient gel. Bernal et al (2012) [22] reported that the calcium produced during the interaction between Na ions and Si and Al ions resulted in the formation of CASH gel, which is rich in Al, unlike the CSH gel observed in conventional concrete.
The inclusion of MPCM, on the other hand, decreased the modulus for both mortars (cement and geopolymer). These results are similar to the compressive strength results in Section 3.2.1. These two moduli depend on the strength of the material [42], so the inclusion of MPCM decreases the stiffness of the matrix due to their low stiffness compared to sand. The second reason may be due to the porosity caused by these materials (section 3.4.2), which decreases the resonant frequencies of the flexure (ff) and torsion (ft) while the latter are the main elements that control these two moduli.

Comparing the geopolymer mortars with and without the addition of MK, Figures 6.C, Figures 6.D, Figures 7.C, and Figures 7.D show that the addition rates of MK improved the Young's modulus and shear modulus for all geopolymer samples. With a concentration of 10 MPCM, the Young's modulus increased from 19.4 to 26.6 GPA. The optimum rate of MK on Young's modulus is 10% for geopolymer mortars with MPCM, while for geopolymer mortars without MPCM, the optimum rate is 20% MK. These results are in good agreement with the compressive strengths and can be explained by the pore filling effect caused by the activation of silica and alumina in the MK.

Figure 8 represents a comparison of the dynamic Young's modulus results of the geopolymer mortars investigated in this study (MGP) with other researchers who have used dynamic methods on geopolymer mortars [24, 43].

A good correlation between Young's modulus and compressive strength expressed as a linear variation with a correlation coefficient of R² = 0.97 for MGP and linear correlations in the two references mentioned can be observed in Figure 8. The MGP results are higher than those of the mentioned references. This is probably a consequence of the use in our case of 80/20 GBFS and MK, unlike Hasnasoui et al (2019) [24] who used 50/50 GBFS and MK and the use of Mobili et al (2016) [43] up to 100% fly ash. The main gel observed in the GBFS activation process is CASH gel. This gel is denser compared to NASH gel (gel obtained by metakaolin or fly ash activation) and has a higher pore filling capacity, which may explain the better mechanical performance obtained in our case compared to the two references [24, 43].

3.3. SEM results

SEM images obtained from the MGP100/0/10 sample after 28 days of curing are presented in figure 9. They show that most of the MPCM have retained their spherical shape and structure, while some have been damaged. This damage may be produced during mixing and is related to the low mechanical strength and stiffness of the MPCM [16]. This might be one of the reasons for the decrease in compressive strength when more MPCM is incorporated.

Indeed, the images also show that the MPCM particles are well bonded to the geopolymer binder. This might explain the high compressive strength with up to 10% MPCM.

The results of Young's modulus and dynamic shear at different times (2, 7, 14, 21, 28, 56 and 90 days) are presented in figures 6 and 7. It can be clearly noticed that these modulus increase with the increase in the curing time for cement-based mortars, while for geopolymeric mortars, the geopolymerization time is stabilized at 14 days. This observation is an indication that cement hydration remains continued during this time, contrary to geopolymerization. This was observed in the compressive strength results.

3.2.2. Dynamic Young's modulus and dynamic shear modulus

Contrary to the compressive strength values, the Young's modulus values of cement-based mortar are higher than those of geopolymer mortar. Geopolymer in general presents a low stiffness [24, 41]. However, it should be noted that the values are very close to those of standardized mortars with a maximum value equal to 35.14 GPA at 90 (MGP80/20/0). This may be related to the good parameters of the formulations set according to the literature.
Figure 6. Dynamic Young’s modulus as a function of curing time, A: (cement-based mortar), B: (GP mortar with 0% MK), C: (GP mortar with 10% MK), D: (GP mortar with 20% MK).

Figure 7. Dynamic shear modulus as a function of curing time, A: (cement-based mortar), B: (GP mortar with 0% MK), C: (GP mortar with 10% MK), D: (GP mortar with 20% MK).

Figure 8. Correlation between dynamic Young’s modulus and compressive strengths at 28 days.

Figure 10. Electrical resistivity as a function of curing time, A: (cement-based mortar), B: (GP mortar with 0% MK), C: (GP mortar with 10% MK), D: (GP mortar with 20% MK).
However, it has been observed that geopolymer mortars have low electrical resistivity compared to the electrical resistivity of cement-based mortars. This phenomenon is attributed to the low solubility of Ca(OH)2 compared to Na+ ions present in geopolymer mortars [46, 47]. Indeed, the alkaline activator contains many conductors of Na+ ions that are present in the interstitial solution, and this could introduce more connections to the electrical network and thus considerably reduce the electrical resistivity of geopolymers.

### 3.4.2. Water porosity

The porosity and density of the examined samples are shown in Figure 11. The porosity of a cement-based mortar without MPCM equals 17.9%. This value is close to the porosity of standardized mortars as measured in various studies [24, 48].

It can be observed that the porosity values for geopolymer mortars without MPCM are lower compared to cement-based mortars. The reason for this difference might be related to the good workability obtained by the geopolymer mortar compared to the cement mortar, this effect is observed in the study of Yang et al. (2020) [49] while they mentioned that the improvement of the workability of the geopolymer reduces its porosity.

The increase in MPCM content, on the other hand, resulted in an increase in porosity and a decrease in density in all of the samples tested (geopolymer mortar and cement). The decrease in density is due to the difference in density between the sand (2.6 g/cm³) and the MPCM (0.84 g/cm³), whereas the increase in porosity might be caused by the fact that the MPCM could not fill the cavities in the matrix due to their agglomeration during mixing and this is due to their agglomeration surface which is larger than the surface of the sand. This agglomeration surface serves to adsorb a quantity of the binder paste whereas this can produce voids during mixing and increases the porosity.

4. Conclusion

The objective of this study was to investigate the effect of the inclusion of 10 and 20% of metakaolin in a matrix of geopolymer-MPCM mortar based on granulated blast furnace slag and to compare it with a mortar based on Portland cement-MPCM.

The conclusions of the different tests are the following:

- The geopolymer-MPCM mortars showed good workability with up to 10% MPCM and 20% MK. The maximum flow time is equal to 4.95 seconds which is less than the standard mortar flow time (7 seconds).
- The inclusion of 10 and 20% metakaolin increased the compressive strength, Young’s modulus and dynamic shear modulus of all geopolymer-MPCM mortar samples. The minimum compressive strength of the geopolymer-MPCM mortar after the inclusion of MK is equal to 49.3 MPA.
- All geopolymer mortars showed low risk of reinforcement corrosion according to the recommendations cited by the ACI 222 committee.
- Despite the good results obtained for the total water porosity of geopolymer mortars (without MK) compared to cement-based mortars, we did not obtain accurate results on the porosity of geopolymers after the addition of MK, this can be explained by the fact that the porosity was controlled only by the incorporation of MPCM.

Finally, it can be concluded that the geopolymer-MPCM mortars developed in this study are apparently capable of meeting a wide range of applications in the construction field. Due to the good mechanical, physical (reduced porosity) and workability performances compared to Portland-MPCM cement-based mortars.

### Nomenclature

- **MPCM**: Microencapsulated phase change materials
- **MGP**: Geopolymer mortar
- **GGBS**: Blast furnace slag
- **MK**: Metakaolin
- **SS**: Sodium silicate

### Declaration of Conflict of Interests

The authors declare that there is no conflict of interest. They have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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