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## Compensation of the negative effects of micro-encapsulated phase change materials by incorporating metakaolin in geopolymers based on blast furnace slag

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1     **Compensation of the negative effects of micro-encapsulated phase change**  
2           **materials by incorporating metakaolin in geopolymers based on blast**  
3                           **furnace slag**

4  
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18    **HIGHLIGHTS**

- 19       • Geopolymer-MPCM mortars have shown better mechanical performance compared to Portland  
20       cement-MPCM mortars.
- 21       • The CASH and NASH gels formed after the inclusion of metakaolin also improved the  
22       mechanical and thermal performance of the geopolymer-MPCM mortars.

- 23 • Good linear correlations were obtained between compressive strengths on the one hand and  
24 Young's modulus and thermal conductivity on the other.

25

## 26 **Abstract**

27 This study aims to use NASH (sodium alumina silicate hydrate) gel and CASH (calcium alumina  
28 silicate hydrate) gel to overcome the negative effects of incorporating microencapsulated phase  
29 change materials (MPCM) on the mechanical strength and thermal conductivity of blast furnace  
30 slag-based geopolymer mortars. The coexistence of CASH and NASH gels was obtained by using  
31 an inclusion of a small amount of metakaolin in a geopolymer matrix based on blast furnace slag;  
32 this results in a geopolymer with high mechanical strength. Several tests were performed to  
33 characterize different mortars (with and without MK) such as workability, microstructural  
34 properties, water porosity, mechanical properties (compressive strength, dynamic Young modulus)  
35 and thermal properties (thermal conductivity, the specific heat capacity). The results obtained  
36 showed that the coexistence of NASH and CASH gel brought improvements in terms of mechanical  
37 properties and thermal conductivity compared to GBFS-based geopolymer-MPCM only. Indeed,  
38 the addition of 10 and 20% of metakaolin was sufficient to obtain this coexistence. With a  
39 concentration of MPCM up to 10% in the geopolymer mortars, the compressive strength was  
40 increased by about 10 MPA and the thermal conductivity was increased by about 31%, which led  
41 to an improvement in the specific heat capacity of up to 1280 J/Kg.K. These improvements were  
42 due to the high reactivity of MK under the activation conditions used. This favored the good  
43 dissolution of silica and aluminum in the MK, which participated well with the calcium in the GBFS  
44 to create the NASH and CASH gels. In fact, it was felt that these two gels filled the small pores  
45 caused by the MPCM and that this compensated well for their negative effects on the geopolymer  
46 matrix.

47 Geopolymer-MPCM based mortars after their optimizations with NASH and CASH gel coexistence  
48 showed good workability, compressive strength, and thermal performance. On the other hand, a  
49 reduced water porosity compared to Portland cement-MPCM based mortars.

50 **Keywords: Geopolymer; Microcapsule Phase Change Materials; Granulated Blast Furnace**  
51 **Slag; Metakaolin**

## 52 **1. Introduction**

53 The building sector is the world's largest energy consumer, consuming 36% of the world's total final  
54 energy and emitting more than 40% of total direct and indirect carbon dioxide (CO<sub>2</sub>) emissions [1].  
55 Most of the energy consumption in the building is reserved for thermal comfort improvement in the  
56 hot and cold seasons (building use phase) [2]. Furthermore, 80% of the greenhouse gas emissions  
57 are related to this phase [3].

58 Several researchers in recent decades have studied thermal storage through latent heat in order to  
59 reuse it when the building needs the heat (e.g., store heat during the day and use it at night) [4–6].  
60 Among the techniques to improve thermal comfort is the use of Microencapsulated Phase Change  
61 Materials (MPCM). These materials help to regulate heat exchange in building compartments and  
62 reduce the requirement for heating and cooling systems [4–8]. MPCM store at a constant  
63 temperature (ambient temperature around human comfort) a large amount of energy in the form of  
64 latent heat during their phase change (solid liquid), which prevents the heat flow from entering the  
65 building during peak hours. This amount of heat is stored during the day and released at night [9].  
66 This technology ensures the desired thermal comfort and reduces the need for air conditioning and  
67 heating, as well as reducing CO<sub>2</sub> emissions related to energy consumption in the building. A recent  
68 literature review showed that the use of these materials in buildings reduces annual energy  
69 consumption by about 50% for cooling and heating [2].

70 After mentioning the enormous energy consumption of the building sector, there is also another  
71 problem in this sector, namely is the massive use of Portland cement in concrete. The cement

72 industry is responsible for 7% of the world's CO<sub>2</sub> emissions [10] and causes other pollution in the  
73 air, water, etc.

74 The reflection of a strategy to reduce the environmental impacts related to CO<sub>2</sub> emissions and the  
75 improvement of the durability of concrete encouraged the researcher Davidovits [11] to invent a  
76 new ecological cement (with less environmental impacts) called the Geopolymer. This material is  
77 the result of the activation of aluminosilicate materials by alkaline solutions [11]. Aluminosilicate  
78 materials are industrial wastes, by-products, or types of clays such as Granulated Blast Furnace Slag  
79 (GBFS), metakaolin (MK), fly ash, and red mud, etc. The use of these materials to replace Portland  
80 cement will reduce CO<sub>2</sub> emissions and waste caused by the industries, thus helping to reduce the  
81 impact on the environment. Wang et al. [12] showed that the CO<sub>2</sub> emissions caused by the  
82 production of geopolymer are reduced by about 70 to 80% compared to the production of clinker.  
83 Furthermore, geopolymer has several advantages over traditional cement-based materials, such as  
84 higher initial mechanical strength, low drying shrinkage, high fire resistance, shorter curing time,  
85 higher resistance to acid attack [13–16]. Moreover geopolymer concrete is more resistant to chloride  
86 penetration because of its reduced overall permeability compared to Portland concrete [17].

87 However, the incorporation of MPCM in the geopolymer can be a solution to decrease the  
88 environmental impacts on two major phases of the building life cycle, which are the building  
89 operation phase (energy use to improve thermal comfort) and the cement production phase.

90 Shadnia et al. [18] constructed three small cells of activated fly ash geopolymer mortars. Among  
91 these three cells, two contain MPCM. Measurements of their internal temperature showed a  
92 reduction of 4.5 and 5.5°C for the two MPCM geopolymer cells compared to the reference cell. Cao  
93 et al. [5] numerically investigated the influence of different conditions on the energy efficiency of a  
94 wall constructed from geopolymer-MPCM concrete using the finite difference method. With the  
95 addition of 5.2% MPCM, there was a reduction in the interior wall temperature of approximately  
96 3°C and a reduction in energy consumption of 25% to maintain the interior temperature at 23°C.

97 In contrast, to date, and based on all current research, most studies show that MPCM exhibits a  
98 negative effect on the mechanical, physical and thermal conductivity performance of the cement  
99 and geopolymer matrix [7,18–20].

100 Cao et al. [7] studied the effect of MPCM addition on the compressive strengths of Portland cement-  
101 based concrete and geopolymer-based concrete. Their results show that the presence of MPCM  
102 increased the heat capacity of both types of concrete studied up to the value of 1500 (J/kg ° C), but  
103 the compressive strengths were reduced by about 42 and 51%. The same observations were reported  
104 by Shadnia et al. [18] that cited a decrease in compressive strength of up to 25%. The study by Cao  
105 et al. [21] showed that the decrease in mechanical performance is related to the increase in open  
106 porosity when the concentrations of MPCM increase in the fly ash geopolymer concrete matrix.

107 Disadvantages of incorporating MPCM into the cement matrix include the rate of decrease in the  
108 thermal conductivity of the total cement matrix [22]. Recall that in the case of a material not  
109 containing MPCM, its thermal insulation performance is related to its low conductivity, which will  
110 delay the penetration of heat into the building. Nevertheless, in the case of a material containing  
111 MPCM, the decrease in its thermal conductivity will disrupt the latent heat of MPCM to complete  
112 their charge-discharge cycle due to the low heat flux transmitted. This is a problem in this field and  
113 has been highlighted by several researchers [23–25]. The effect of reducing thermal conductivity  
114 after the addition of MPCM is similar to its effect on increasing porosity (porosity caused by the  
115 presence of MPCM in the cement matrix) [22,23]. Several researchers have attempted to improve  
116 the thermal conductivity of pure PCM (without capsules) after their incorporation into a cement  
117 matrix by using some carbon-based or metal-based materials that have high thermal conductivity  
118 [26–29]. Nevertheless, unfortunately, few studies have been conducted to improve the thermal  
119 conductivity of a cement or geopolymer matrix after the incorporation of MPCM.

120 The scientific question of this study is how to overcome the negative effects of incorporating MPCM  
121 into the geopolymer matrix to improve the mechanical and thermal performances of this new  
122 material.

123 Concerning the use of geopolymer, we note that the main gel produced in the geopolymerization  
124 process is in the form of a gel called sodium alumina silicate hydrate (NASH), resulting from the  
125 activation of alumina and silica found in metakaolin or fly ash [30]. This gel is the result of the  
126 chemical reaction between  $\text{Na}_2\text{O}-\text{Al}_2\text{O}_3-\text{SiO}_2-\text{H}_2\text{O}$ .

127 In recent years, several researchers have shown that it is possible to have in a single geopolymer  
128 matrix another hydration gel in addition to the geopolymer gel itself. This gel can be obtained in a  
129 matrix that is rich in calcium, silica and alumina to produce calcium alumina silicate hydrate  
130 (CASH), and it is obtained from the chemical reaction between  $\text{Na}_2\text{O}-\text{CaO}-\text{Al}_2\text{O}_3-\text{SiO}_2-\text{H}_2\text{O}$  [31–  
131 34]. The CASH gel results from the activation of blast furnace slag [35]. The use of calcium-rich  
132 blast furnace slag with the inclusion of a small amount of silica ( $\text{SiO}_2$ ) and alumina ( $\text{Al}_2\text{O}_3$ ) rich  
133 metakaolin (MK) contributes to a better workability and a structure mainly composed of the  
134 coexisting gels mentioned above. This coexistence of gels, once obtained, improves the mechanical  
135 performance and durability of the geopolymer [34].

136 However, most of the research that has been carried out on the geopolymer-MPCM is focused on a  
137 geopolymer based on a unique base material such as GBFS, fly ash (FA), MK, etc. In addition, there  
138 are few studies on combining two primary materials rich in calcium, alumina, and silica at the same  
139 time, and no studies have investigated the inclusion of MK into a GBFS-based matrix containing  
140 MPCM.

141 The aim of this study is to investigate the feasibility of developing the coexistence of CASH and  
142 NASH gels in geopolymer-MPCM mortars to overcome the negative effects of MPCM  
143 incorporation on the mechanical and thermal performance of these mortars. This coexistence of gels  
144 will be achieved by adding three percentages of MK inclusion (0, 10 and 20%) in a GBFS-based  
145 mortar. The geopolymer-MPCM mortars were also compared to Portland-MPCM cement-based  
146 reference mortars.

147 Several characterizations of two types of mortar are carried out in this study, such as workability,  
148 microstructure properties (SEM, EDS and XRD), water porosity, mechanical properties

149 (compressive strength, dynamic Young's modulus resistance) and thermal properties (thermal  
150 conductivity, the specific heat capacity).

## 151 **2. Materials and experiments design**

### 152 **2.1. Materials**

153 The MPCM studied here is considered in the form of white spherical microcapsules marketed by the  
154 laboratory Microteck-United States. Its technical name is Nextek 28 D. The microcapsule consists  
155 of two parts, a polymer wall and the core material (paraffin). Its composition is  $\geq 97\%$  paraffin  
156 powder. The polymer is a urea polymer, cross-linked with a modified low molecular weight  
157 polyethylene. MPCM possesses a melting temperature between 26 and 28°C and a heat capacity  
158 between 180 and 190 (J/g).

159 The density was measured by the pycnometer method according to NF T 20-053 and is equal to 0.84  
160 g/cm<sup>3</sup>, almost equal to the density of MPCM in the literature [7,18]. Its thermal conductivity was  
161 measured with the Hot Disk 1500 device (the measurement procedure will be explained in the  
162 following sections). It is equal to 0.2 W/mK. This value is the same as that found in the literature  
163 [25]. MPCM grains range in size from 6.19 to 38.22  $\mu\text{m}$ .

164 The GBFS was delivered by the company ECOCEM in France while the MK was provided by the  
165 company KENZAI (ecological materials) in France. The cement used is CEM II (32.5). The  
166 chemical compositions and physical properties of cement II, GGBS and MK are presented in Table1.

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

<b>Chemical composition</b>	<b>CEM II</b>	<b>GBFS</b>	<b>MK</b>
(%)			
SiO <sub>2</sub>	7,47	37,3	55
Al <sub>2</sub> O <sub>3</sub>	2,18	10,7	41
Fe <sub>2</sub> O <sub>3</sub>	2,84	0,2	1,2
CaO	69,02	43,0	0,1

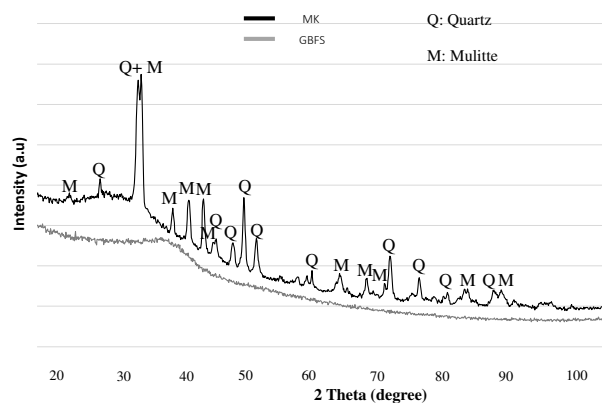


MgO	-	6,5	0,2
TiO <sub>2</sub>	-	0,7	0,4
(Na <sub>2</sub> O + K <sub>2</sub> O) <sub>eq</sub>	-	0,8	1,8
Specific gravity	3.03	2.90	2.40
Blaine specific surface area (m <sup>2</sup> /g)	0.37	0.445	17
average grain size (μm)	8.47	13.25	7.13

168

169 According to Table 1, GBFS is composed of a sum of 48% SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> and a high content of  
 170 42.3% CaO while metakaolin MK is rich in SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> with a sum of 96%.

171 The mineralogical structure is shown by X-ray diffraction (XRD) in Figure 1. MK shows a sharp  
 172 crystalline peak at 32°, which is attributed to the presence of amorphous quartz structure (SiO<sub>2</sub>) and  
 173 mullite crystalline phases (Al<sub>6</sub>Si<sub>2</sub>O<sub>13</sub>). GBFS is composed mainly of an amorphous phase.

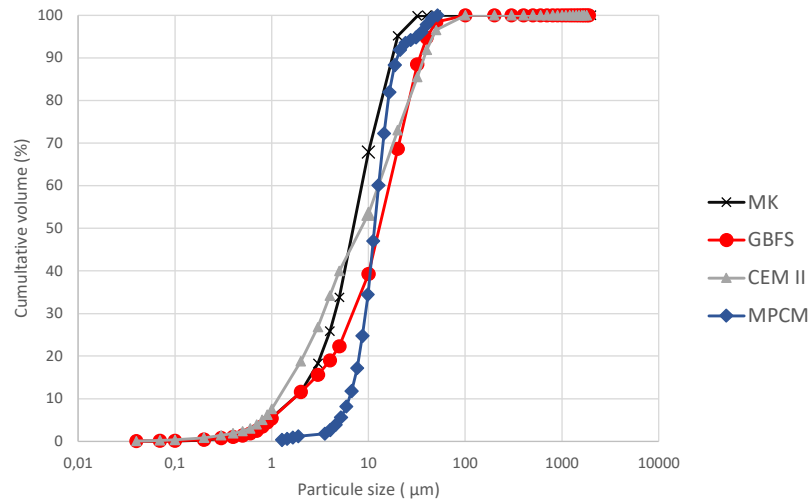


174

175 Figure 1. XRD patterns of MK and GBFS.

176 Figure 2 shows the particle size distribution of GBFS, MK, CEM II and MPCM, which was determined  
 177 using a laser granulometer type Cilas 1190. The measurements were performed using the wet method.  
 178 Indeed, we had doubts about the fact that water would not disperse MK particles, but the results of the  
 179 measurements are close to the results quoted in the study of Kamath et al. [35].

180 It is also noted that MK has finer particles than GBFS with an average particle size that is equal to 7.13  
181  $\mu\text{m}$  while the average particle size of GBFS and cement is equal to 13.25  $\mu\text{m}$  and 8.47  $\mu\text{m}$ . On the other  
182 hand, their specific surface areas are equal to 17; 0.44; and 0.37  $\text{m}^2/\text{g}$ .



183

184 Figure 2. Particle size distribution of MK, GBFS, CEM II and MPCM.

185

186 The activation solution is a mixture of sodium silicate and sodium hydroxide. According to the supplier,  
187 the composition by mass of the sodium silicate is 27.53%  $\text{SiO}_2$ , 11.47%  $\text{Na}_2\text{O}$  and 61%  $\text{H}_2\text{O}$ . Sodium  
188 hydroxide  $\text{NaOH}$  is a caustic soda of 98% purity. The two solutions were provided by the company  
189 E2EM in France.

190 A standardized sand (CEN NF 196-1). This type of sand is generally used for laboratory tests. The  
191 supplier is the same as for two alkaline solutions. The purpose of using standardized sand is to eliminate  
192 the secondary effects on the binder of unfavorable impurities that natural sand may contain.

## 193 2.2. Mixing method and curing condition

194 Twelve formulations were investigated in this study; three were based on standardized mortar, and nine  
195 were based on geopolymer mortar. The water/binder ratio of 0.5 and the sand/binder ratio of 3 have been  
196 fixed for the two types of mortars, knowing that the equivalent binder for geopolymer (equivalent to  
197 cement) is considered as the total of GBFS, MK, and alkaline solution (solid part). For this, the mass

198 ratio (GBFS+MK)/SA was set to 3, SA corresponding to the solid part of the alkaline solution. These  
199 choices are recommended by the study of Hasnaoui et al [14].

200 MK will replace part of the GBFS in the geopolymer matrix with three substitution rates (0, 10 and  
201 20%). Bernal et al. [34] cited that increased MK content resulted in significant reductions in compressive  
202 strength, due to the incomplete reaction of MK in the activation solution used. In fact, the optimal  
203 parameters were determined in this study to provide a high alkalinity of the activation solution to get  
204 good reactivity of the MK. Regarding this observation, a mass ratio of 2.5 between sodium silicate (SS)  
205 and sodium hydroxide (SS/NaOH) was set according to the study of Bernal et al. [34].

206 The sodium hydroxide solution was prepared with a concentration of 12M and mixed with the sodium  
207 silicate and allowed to cool for 24 hours before the mixing procedure.

208 A high concentration of NaOH (above 14M) can disrupt the dissolution of calcium in the matrix [36],  
209 resulting in an absence of CASH gel, while a low concentration of NaOH does not allow the dissolution  
210 of silica and aluminum in GBFS and MK, while the latter require sufficient  $\text{Na}^+$  to be dissolved. Huseien  
211 et al. [37] carried out a study on the effect of NaOH concentration in a geopolymer matrix. They  
212 indicated that the mechanical properties and microstructures of the geopolymer were improved with a  
213 concentration of 12M as chosen in this study.

214 Regarding MPCM, three concentrations were chosen of MPCM in the two types of mortar (cement-  
215 based and geopolymer) such as 0; 5 and 10. The method chosen for the incorporation of MPCM in the  
216 geopolymer is the method of replacement of sand by MPCM. This method is the most used in this field  
217 and, according to the study by Pilehvar et al. [38], its use allows the least reduction of the mechanical  
218 performance.

219 Table 2 illustrates the mixing proportions in  $\text{kg}/\text{m}^3$ . The first three formulations are based on  
220 standardized cement mortar while the last nine are based on geopolymer.

221 The quantity of water in the two alkaline solutions is considered to have a water/binder ratio equal to  
222 0.5.

223 Table 2. Formulations of cement II mortars and geopolymer mortars ( $\text{kg}/\text{m}^3$ ).

224

225

226

Sample	Mixtures proportions (kg/m <sup>3</sup> )								
	Cement	GBFS	MK	Sand	MPCM	Na <sub>2</sub> SiO <sub>3</sub>	NaOH	MGP water	MCII water
MCII <sub>0/0/0</sub>	585,9	-	-	1757,8	-	-	-	-	293,0
MCII <sub>0/0/5</sub>	585,9	-	-	1673,8	28,4	-	-	-	293,0
MCII <sub>0/0/10</sub>	585,9	-	-	1582,0	56,8	-	-	-	293,0
MGP <sub>100/0/0</sub>	-	439,5	-	1757,8	-	267,9	127,7	42,6	-
MGP <sub>100/0/5</sub>	-	439,5	-	1673,8	28,4	267,9	127,7	42,6	-
MGP <sub>100/0/10</sub>	-	439,5	-	1582,0	56,8	267,9	127,7	42,6	-
MGP <sub>90/10/0</sub>	-	395,5	43,9	1757,8	-	267,9	127,7	42,6	-
MGP <sub>90/10/5</sub>	-	395,5	43,9	1673,8	28,4	267,9	127,7	42,6	-
MGP <sub>90/10/10</sub>	-	395,5	43,9	1582,0	56,8	267,9	127,7	42,6	-
MGP <sub>80/20/0</sub>	-	351,6	87,9	1757,8	-	267,9	127,7	42,6	-
MGP <sub>80/20/5</sub>	-	351,6	87,9	1673,8	28,4	267,9	127,7	42,6	-
MGP <sub>80/20/10</sub>	-	351,6	87,9	1582,0	56,8	267,9	127,7	42,6	-

227

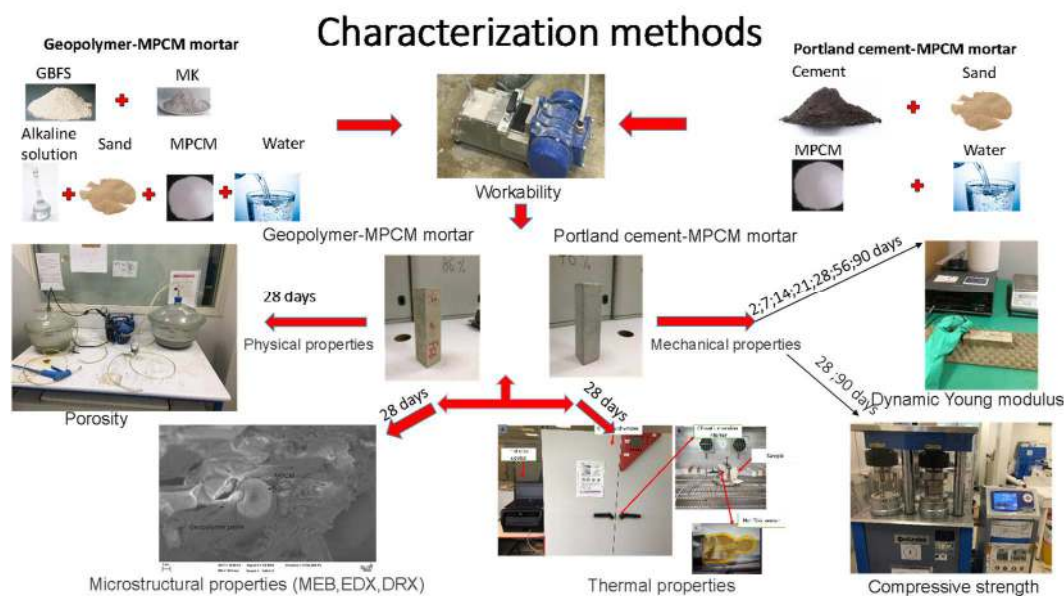
228 The standard NF 196-1 [39] was followed for the fabrication of standardized cement mortars, which  
 229 were chosen as a reference for comparison with geopolymer-based mortars. However, regarding the  
 230 mixing of geopolymers, GBFS and MK were mixed with alkaline solution and water for 90 seconds to  
 231 produce a homogeneous paste [19,20]. The mixing was followed by the addition of sand and mixed for  
 232 5 minutes and finally MPCM was added and mixed for 2 minutes.

233 The idea of increasing the mixing time is recommended by Ahmari et al. [40] and is intended to allow  
 234 the efficient dissolution of silica, alumina, and calcium (Ca) in the alkaline solution. After the mixing

235 procedures, standardized cement mortars and geopolymers were used for the measurement of  
 236 workability. After this step, the casting is done in specimens of  $40 \times 40 \times 160 \text{ mm}^3$  and vibrated using  
 237 an impact table and are stored in an air-conditioned room (temperature  $20 \pm 1^\circ \text{C}$  and relative humidity  
 238  $50 \pm 5\%$ ) for 48 hours before demolding. All samples except the standardized cement mortars (water  
 239 conservation) were covered with plastic films.

### 240 2.3. Experimental methods

241 In this research, all the experiments tests were carried out at room temperature, i.e., between  $19^\circ \text{C}$  and  
 242  $21^\circ \text{C}$ , excepted the thermal properties (see dedicated item). All the tests, including age of mortars, are  
 243 listed in Figure 3. The samples used have the same dimensions ( $40 \times 40 \times 160 \text{ mm}^3$ ) for mechanical  
 244 tests, water porosity and thermal properties determination.



245

246

Figure 3. Characterization methods

247 The workability test was carried out directly after mixing according to standard NF P18-452 [41] using  
 248 a workability meter. The test consists in measuring the flow time of mortars under the effect of the  
 249 vibration caused by the vibrator. The water porosity was determined according to the NF P 18-459  
 250 standard [42].

251 Morphological analyses were performed using a ZEISS CVO®4 scanning electron microscope (SEM)  
 252 combined with a BRUKER marker energy dispersive X-ray spectroscopy (EDS) analyzer. The analyses

253 were performed on fractured samples after 28 days of curing. Indeed, the geopolymer samples were  
254 placed in the oven at 60°C to limit hydration before the SEM analyses. XRD analysis was performed on  
255 samples ground to powder using a mortar pestle, then sieved using an 80 µm sieve after 28 days of  
256 curing. Hydration was then stopped using an acetone quench. Measurements were performed using a  
257 SEIFERT MZ VI E X-ray diffractometer with Co K $\alpha$  radiation and a constant scanning speed in the  
258 range  $2\theta = 15^\circ$ - $115^\circ$ .

259 Compressive strengths were performed according to the NF EN 196-1 [40] standard. These tests were  
260 carried out on six samples to ensure good repeatability. Dynamic Young's modulus measurements were  
261 performed following the ASTM E 1876-01 standard [43] and using the Grindosonic apparatus. These  
262 measurements were performed on three samples of each formulation at 2, 7, 14, 21, 28, 56 and 90 days.  
263 The technique is based on pulse excitation of the vibrations. This modulus is calculated with the use of  
264 the equation 1 and by using the flexural frequency ( $f_f$ ) obtained during the measurement with the  
265 dimensions and weight of the samples:

$$E = 0.9465 \times \left( \frac{m \times f_f^2}{b} \right) \times \left( \frac{L^3}{t^3} \right) \times T_1 \quad (1)$$

266

267 With E,  $f_f$ , m, L, b, t represent the Young's modulus (Pa), flexural frequency (Hz), mass in (g), length  
268 in (mm), width in (mm) and thickness in (mm) of the sample.  $T_1$  represents correction factor that  
269 accounts for the finite thickness of the bar, Poisson's ratio, and so forth.

270 One of the main objectives of this work is to evaluate the thermal properties of the materials studied  
271 here to determine their thermal performance so that they can be used in the future in a real application.

272 In the research field, there are two techniques for measuring the thermal properties of a material; namely,  
273 the steady-state measurement technique and the non-steady-state (transient) measurement technique  
274 [44]. Transient measurements have attracted a great deal of research because of their speed and accuracy.

275 Among these is the hot disk technique which uses the transient planar source technique (Hot Disk TPS).

276 The advantage of this method is that it can measure the thermal properties of a wide range of materials  
277 and measure the thermal conductivity in the range of 0.005 to 500 W/m.K.

278 The Hot Disk method can measure three thermal properties at the same time, such as thermal  
279 conductivity, thermal diffusivity, and specific heat capacity, using the TPS sensor, which has two roles  
280 (heat transmitter and temperature measurement receiver) [45].

281 Nevertheless, thermal property measurements with the TPS Hot Disk are often performed in an  
282 environment where the temperature is ambient (between 20 and 22°C). This is not possible in our case  
283 due to the melting temperature of the MPCM used in this study (between 26 and 28°C). Given this  
284 constraint, the measurements were carried out in a climate controlled chamber with a temperature range  
285 between 15 and 40°C and a constant humidity of 50%. The climatic chamber used is the KBF 720 type.  
286 Its temperature ranges are between 0 and 70 ° C while the relative humidity ranges are between 10 and  
287 80 %.

288 Figure 4 shows a picture of the climatic chamber and the hot disk device with the measurement samples.



289

290

291 Figure 4. Thermal measuring device, A: climatic chamber and hot disk; B: interior view of the  
292 climatic chamber; C: the measuring sensor

293 The thermal conductivity is influenced by the moisture content of the measurement samples. Due to this  
294 influence, the measurements were carried out on samples dried at 60 °C until they had a constant mass.  
295 This drying method was recommended by Zhang et al. [46]. Thermal measurements are performed by  
296 placing the sensor on the upper surface of the sample, which is connected to a data acquisition unit as  
297 shown in Figure 4.

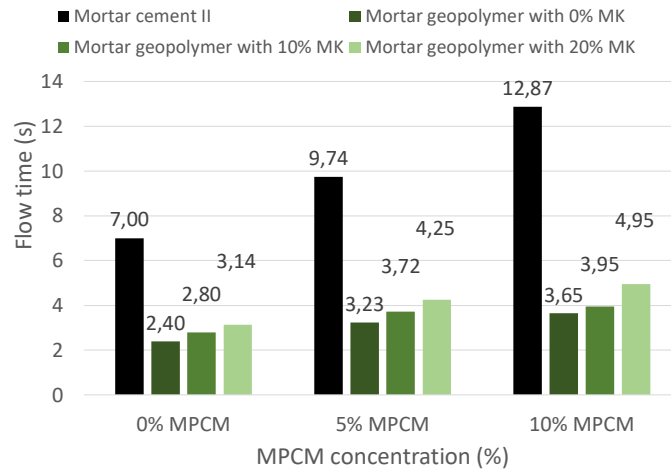
### 298 3. Results and discussion

#### 299 3.1. Workability

300 Figure 5 shows the flow time of the different formulations studied. It should be noted that the main ratios  
301 [Water/Binder, (GBFS+ MK)/SA, SS/NaOH and NaOH concentration] that affect the workability of the  
302 geopolymer were fixed. The increase in the ratio (GBFS+ MK)/SA increases progressively the flow  
303 time, which means that the workability of geopolymer mortars decreases with the incorporation of raw  
304 materials (GBFS+ MK). This decrease is due to the high water demand of raw materials, especially  
305 metakaolin, which has a high specific surface [14]. On the other hand, increasing the SS/NaOH ratio  
306 and the concentration of NaOH increases the viscosity of the activation solution and thus reduces the  
307 flow of the mortar. This is due to the very high viscosity of sodium silicate and sodium hydroxide [47].  
308 Then the only variables in this study are the MPCM in both types of mortar (cement-based and  
309 geopolymer) and the MK in the geopolymer mortar. Cement mortar without MPCM showed a flow time  
310 of about 7 seconds that is confirmed from the literature [14].

311 Furthermore, as the MPCM concentration increases, the flow time increases for both types of mortars,  
312 meaning that workability is reduced. This effect is attributed to the agglomeration of MPCM during  
313 mixing due to its small size. This causes a larger water adsorption surface compared to the sand surface.  
314 Indeed, MPCM traps water and prevents it from penetrating the matrix, which results in a decrease in  
315 workability. In fact, MPCM strongly affected cement mortars while it did not show significant effects  
316 on geopolymer mortars. This difference is explained by the very high workability of geopolymers  
317 compared to cement-based mortars. This is related to the difference in activation of aluminosilicate  
318 materials compared to cement-based materials, as confirmed by Muhammad et al. [48] and Deb et al.  
319 [49].





320

321

Figure 5. Workability of different formulations

322

323 It is further observed that there is a slight increase in the flow time in all the mortars of geopolymers  
 324 with 10 and 20% MK. This increase is related to the water demand of MK due to its large specific  
 325 surface area.

326 Based on the comparison with the flow time of cement-based mortars, geopolymer-MPCM mortars  
 327 showed good workability using up to 10% MPCM and 20% MK where the maximum flow time is equal  
 328 to 4.9 seconds that is lower than the flow time of Portland cement-based mortar (7 seconds).

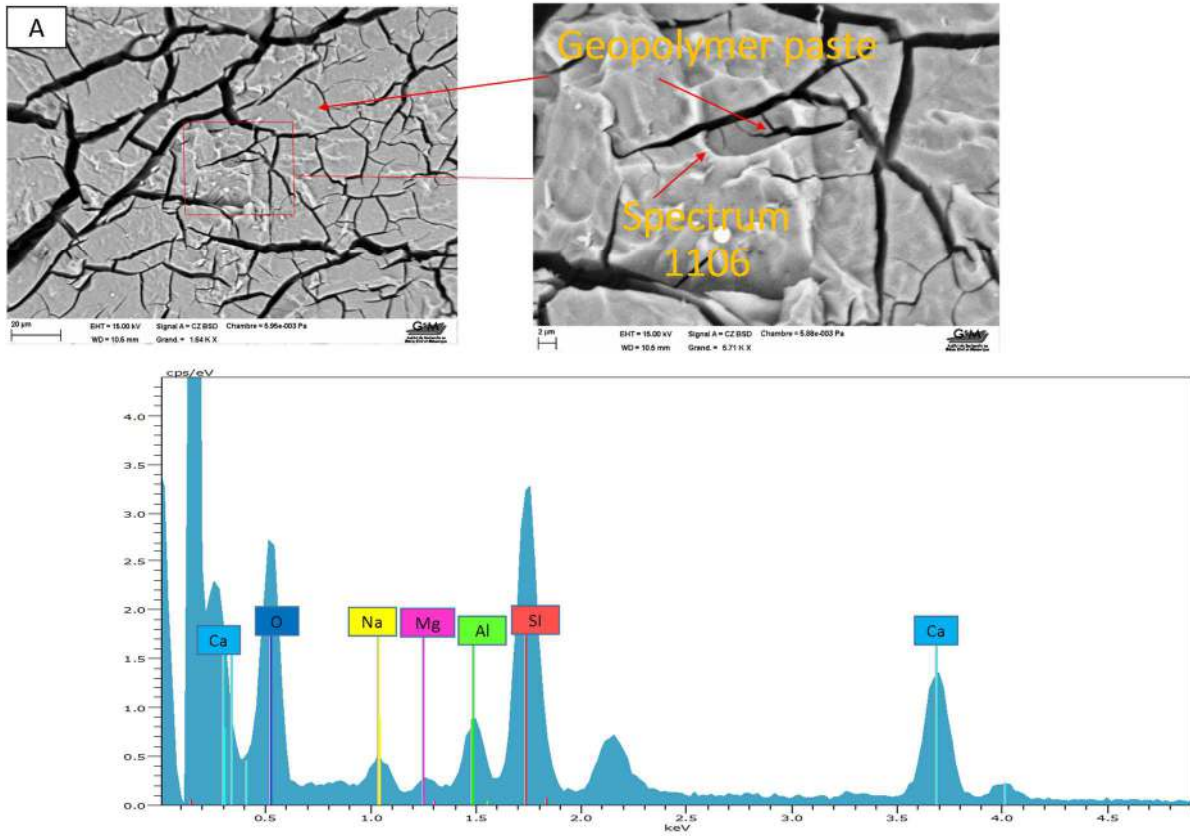
### 329 3.2. Microstructural analysis

330 Morphological analyses were performed with SEM for the observation of the microstructure of the  
 331 geopolymer samples and EDS for the analysis of the elemental composition. Figure 6 compares the  
 332 fractured geopolymer samples MGP<sub>100/0/0</sub> (Figure 6.A) and MGP<sub>80/20/0</sub> (Figure 6.B).

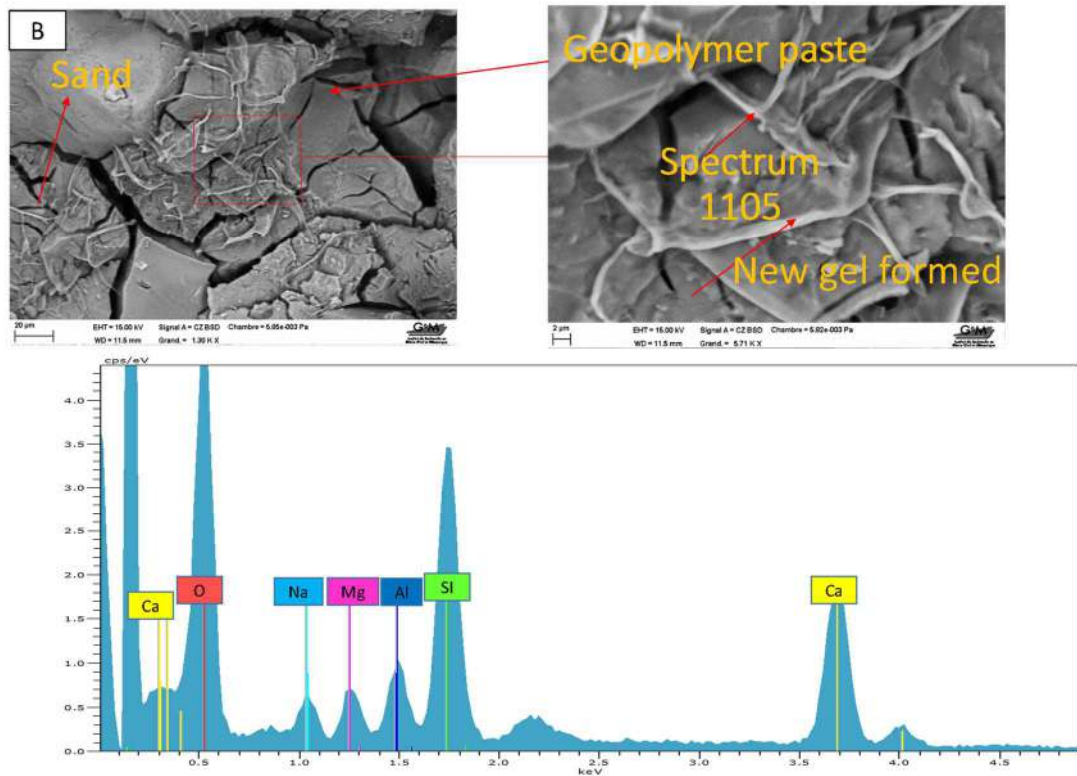
333 The SEM observations clearly show the appearance of additional gels in the matrix with 20% MK  
 334 (Figure 6.B), while this appearance of gels was not observed in the matrix with 0% MK. The EDS  
 335 analyses showed the presence of the main elements Si, Al, Ca, Na, which shows the formation of CASH  
 336 gel, the main component of the GBFS activation [35,50]. Indeed, the analyses also show a higher content  
 337 of the main elements mentioned previously for the MGP<sub>80/20/0</sub> formulation compared to the MGP<sub>100/0/0</sub>  
 338 formulation. The increase in Si, Al, Ca, Na confirms the presence of NASH in addition to CASH gel in

339 the MGP<sub>80/20/0</sub> formulation. According to Mijarsh et al. [30], the coexistence of these two gels results a  
340 single stable gel called (C, N)ASH.

341



342



343

344

Figure 6. Microstructures of fractured samples at 28 days, A: MGP<sub>100/0/0</sub>; B: MGP<sub>80/20/0</sub>.

345

Table 3 compares the elemental compositions obtained by EDS of the geopolymer mortars MGP<sub>100/0/0</sub>,

346

MGP<sub>100/0/10</sub>, MGP<sub>80/20/0</sub>, and MGP<sub>80/20/10</sub>.

347

Table 3: Elemental compositions of MGP<sub>100/0/0</sub>, MGP<sub>100/0/10</sub>, MGP<sub>80/20/0</sub>, MGP<sub>80/20/10</sub>.

348

### Specimen

Elemental compositions (%)	MGP <sub>100/0/0</sub>	MGP <sub>80/20/0</sub>	MGP <sub>100/0/10</sub>	MGP <sub>80/20/10</sub>
<b>O</b>	60,69	54,71	65,91	58,17
<b>Si</b>	14,68	18,16	14,32	20,97
<b>Al</b>	3,64	3,94	2,79	3,74
<b>Ca</b>	15,05	19,35	12,29	14,06
<b>Na</b>	3,31	3,20	3,66	2,25

Si/Al

4,03

4,60

5,13

5,60

349

350 Table 3 shows that the concentrations of silica, calcium and aluminum increased after the addition of  
351 20% MK. The mass content of silica is equal to 14.68% in the case of MGP<sub>100/0/0</sub> while it increased to  
352 18.16% with 20% MK (MCP<sub>80/20/0</sub>). This content also increased from 14.32 to 20.97% in the geopolymer  
353 mortar which contains 20% MPCM after the inclusion of 20% MK. Indeed, the main precursors of MK  
354 activation are Si and Al, which may indicate that the high silica and alumina content of MK was well  
355 dissolved by alkaline activation to form Si-O-Si and Si-O-Al bonds. On the other hand, the increase in  
356 silica content increased the Si /Al ratio, confirming that the Si-O-Si bond was dominant over the Si-O-  
357 Al bond. The Si/Al ratio increased from 4.03 in the MGP<sub>100/0/0</sub> matrix to 4.60 in the MGP<sub>80/20/0</sub> matrix,  
358 and from 5.13 in MGP<sub>100/0/10</sub> to 5.6 in MGP<sub>80/20/10</sub>. The increase of the Si/Al ratio improves the  
359 microstructure of the geopolymer, which results in an increase in its mechanical performance [30,51,52].  
360 Moreover, it is also noted that the calcium content increased after the addition of MK, showing that the  
361 calcium reacted well to the addition of MK and that its dissolution is not disturbed by the presence of  
362 the 12M NaOH concentration. The increase in the calcium content increases the mechanical  
363 performance of the geopolymer, as it will be shown in section 3.5 [34,53,54].

364 In addition, the presence of CASH and NASH gel can be confirmed by the mass ratio values in  
365 percentages of Na/Si, Al/Si, Ca/Si. The Na/Si ratios are between 0.10 and 0.22 and Al/Si ratios are  
366 between 0.17 and 0.24. They are in the range of the study by Ibrahim et al. [51] from which they  
367 confirmed the appearance of these gels. On the other hand, the Ca/Si ratios are between 1.02 and 1.06,  
368 which are higher than their ratios. This difference is due to the use of up to 80% GBFS in this study with  
369 high calcium content.

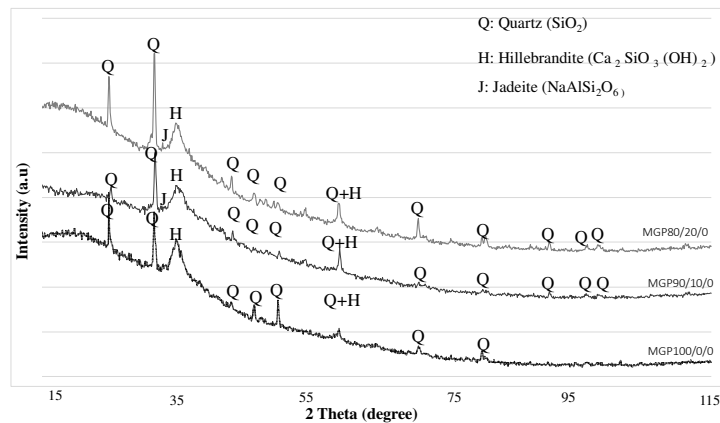
### 370 3.3. X-ray diffraction analysis

371 Figure 7 shows the XRD analyses performed on the powders of samples MGP<sub>100/0/0</sub>, MGP<sub>90/10/0</sub> and  
372 MGP<sub>80/20/0</sub> at 28 days. From a global point of view, it appears that all the samples show crystalline phases.  
373 Around the 35° peak, the presence of hillebrandite of chemical formula Ca<sub>2</sub> SiO<sub>3</sub> (OH)<sub>2</sub> is observed in

374 all samples. Indeed, hillebrandite belongs to the CSH family of gels; Mijarsh et al. [30] explained that  
375 with the presence of aluminum, the main composition of CSH turns into CASH gel. These results were  
376 observed in EDS in the previous section (3.2) and are due to the use of up to 80% GBFS which leads to  
377 the creation of CASH gel in general.

378 Several researchers have confirmed the presence of hillebrandite around this peak at 35° [30,55,56]. On  
379 the other hand, Mijarsh et al. [30] have noted that hillebrandite serves the improvement of the  
380 mechanical performance of the geopolymer. These results are in good agreement with the mechanical  
381 performance results in Section 3.5.1.

382



383

384 Figure 7. X-ray diffraction of samples MGP<sub>100/0/0</sub>; MGP<sub>90/10/0</sub>; MGP<sub>80/20/0</sub>) at 28 days.

385 After MK inclusion, the peak of jadeite (NaAlSi<sub>2</sub>O<sub>6</sub>) increased around 33°. Jadeite is a member of the  
386 NASH family (the main gel for MK activation) [30]. It has a crystal structure with tetrahedral Si  
387 coordinated in single chains and Al and Na in octahedral coordination [30,57]. The presence of  
388 hillebrandite and jadeite in a single matrix confirms the coexistence of NASH and CASH gel and results  
389 also in a high mechanical strength [51,55]. According to Ibrahim et al. [51], the CASH and NASH gel  
390 renders the microstructure very compact and dense and this serves to improve the mechanical strength.  
391 Indeed, this can be explained by the dissolution of silica, alumina, and calcium, which serve to fill the  
392 small pores in the geopolymer matrix [50]. EDS analysis also showed that the silica, alumina, and  
393 calcium contents were increased after the addition of MK.

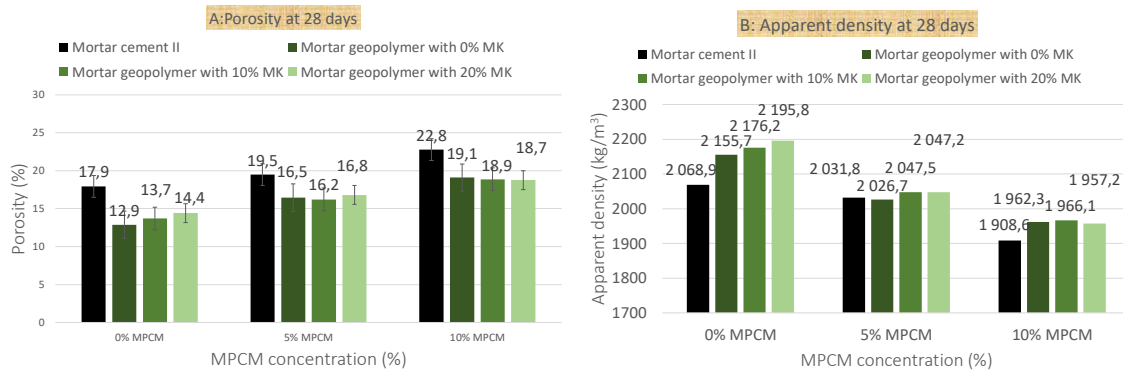
394 On the other hand, around the 60° peak, the intensity of the hillebrandite plus quartz (SiO<sub>2</sub>) peak was  
395 increased. This clustering may be related to the participation of calcium after the inclusion of MK in the  
396 formation of the CASH gel with the presence of the NASH gel. The EDS confirmed the same  
397 observations where the calcium content was increased after the inclusion of MK. Regarding this  
398 observation, it is not possible to confirm these results because the samples used are in the form of mortars  
399 (presence of sand). Quartz is therefore the main element of the sand.

#### 400 **3.4. Water porosity and apparent density**

401 Figure 8 shows the water porosity results as well as the density of the studied samples. It indicates that  
402 the cement-based mortar without MPCM presents a porosity of 17.90%. This value is approximately  
403 similar to that reported in several studies that have investigated porosity for standardized cement mortars  
404 [14,58].

405 It is noted that the porosity values of geopolymer mortars without MPCM are lower than those of  
406 cement-based mortars. This difference may be related to the difference in workability between the two  
407 types of mortars in section 3.1. Yang et al. [59] mentioned similar observations and cited that the  
408 improvement of the workability of geopolymer reduces its porosity.

409 In addition, the increase in MPCM concentration caused an increase in porosity and a decrease in density  
410 of all the samples studied. The decrease in density may be caused by the difference in density between  
411 the sand (2.6 g/cm<sup>3</sup>) and the MPCM (0.84 g/cm<sup>3</sup>). Furthermore, the increase in porosity may be due to  
412 the MPCM not filling the matrix cavities due to their agglomeration surface area being larger than that  
413 of the sand. This agglomeration surface serves to adsorb a small amount of the binder paste, while this  
414 can produce voids during mixing and increase porosity.



415

416

Figure 8. Water porosity and apparent density at 28 days.

417 However, the addition of MK shows no effect on porosity, showing that MPCM exerts a major effect  
 418 on controlling the porosity of the geopolymer samples studied here. In contrast to the porosity results,  
 419 the density was improved after the addition of MK in the samples without MPCM. This improvement  
 420 was due to the high reactivity of MK compared to GBFS, which promoted the good dissolution of silica  
 421 and aluminum to form new NASH and CASH gels in the matrix, as shown in Sections 3.2 and 3.3.

### 422 3.5. Mechanical properties

#### 423 3.5.1. Compressive strength

424 Figure 9 shows the results of the compressive strength at 28 and 90 days. Globally, it should be noted  
 425 that there is no significant difference between the compressive strength between 28 and 90 days for  
 426 geopolymer mortars, contrary to cement-based mortars, which show an increase in their compressive  
 427 strength between 28 and 90 days. Geopolymer is considered as a material that can gain most of its  
 428 mechanical strength in the first few days of curing due to its strong chemical bonding, unlike cement-  
 429 based materials.

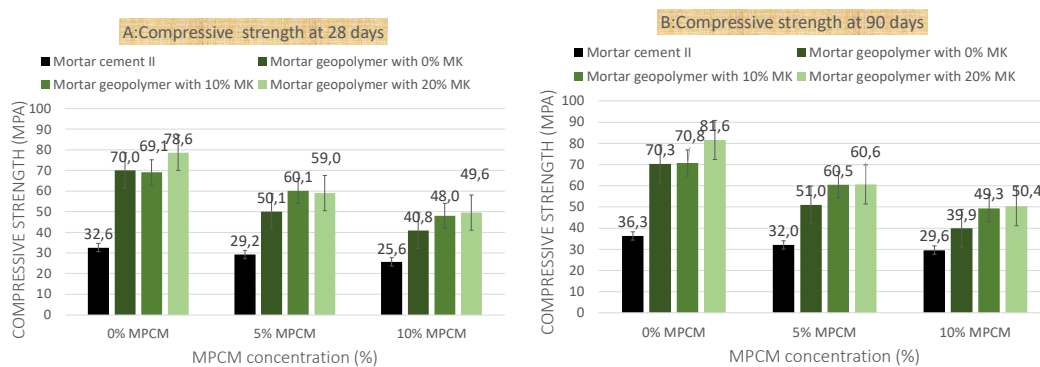
430 Bernal et al. [34] found that there is no difference in the compressive strength between 28 and 90 days  
 431 for geopolymer without the addition of metakaolin (100% GBFS), while Bai et al. [60] pointed out that  
 432 the increase in the GBFS rate serves to accelerate the development of the mechanical strength of the  
 433 geopolymer. This may explain why there is no great difference between the 28- and 90-day time frames  
 434 in this study.

435 The compressive strength of geopolymer mortars (with and without MPCM) is higher than the  
 436 compressive strength of cement-based mortars. This may be related to the optimal formulation ratios  
 437 that have been set according to the literature [14,34,37] and that have contributed to the improvement  
 438 of geopolymerization.

439 The compressive strength of cement-based mortars decreases as the rate of MPCM incorporation  
 440 increases. After 28 days, the compressive strength decreased from 32.6 to 29.2 MPA with 5% MPCM  
 441 and then to 25.6 MPA with 10% MPCM. The effect is remarkably similar after 90 days. In the case of  
 442 geopolymer-based mortars containing 0% MK, the same results are obtained. The compressive strength  
 443 value in this case decreased from 69.9 to 50 MPA with 5% MPCM and reached 40.8 MPA with 10%  
 444 MPCM after 28 days. Therefore, the rate of reduction of mechanical performance of geopolymer mortars  
 445 with 10 and 20% MK is less compared to geopolymer mortars without MK.

446 The compressive strength value for the 10% MK geopolymer mortar is reduced from 69.08 to 60.12  
 447 with 5% MPCM, and then it is reduced to 47.9 MPA with 10% MPCM, while for the 20% MK  
 448 geopolymer mortar, the value is reduced from 78.5 to 58.9 until it reaches a value of 49.5 MPA.

449 This reduction in compressive strength is caused by the effect of replacing MPCM with sand. Recall  
 450 that MPCM has replaced a certain volume percentage of sand in the matrix. Therefore, MPCM has low  
 451 stiffness and mechanical strength compared to sand and can easily break under compressive force  
 452 [7,18,23]. Furthermore, the increase in matrix porosity after MPCM incorporation is one of the causes  
 453 of the reduction in mechanical strength as observed in the results detailed in the section. **3.4.**



454

455

Figure 9. Compressive strength; A: 28 days; B: 90 days.



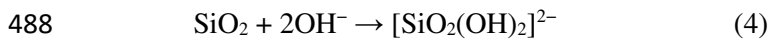
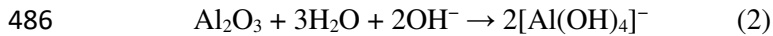
456 In addition, it can be observed that the geopolymer mortars with 0% MK showed a high mechanical  
457 strength. This increase is caused by the high activator content and is due to the high Si/Al ratio [61]. The  
458 presence of this high ratio in this study can be explained by the good dissolution of silica ions and  
459 alumina of the GBFS due to the high PH of the solution, which results in a gel with high mechanical  
460 performance [34].

461 Moreover, the addition of 10% MK in geopolymer mortars with MPCM concentrations (5 and 10%) led  
462 to an increase in compressive strength at 28 days. The value increased from 50.06 to 60.12 MPA  
463 (geopolymer mortar with 5% MPCM) and from 40.8 to 47.9 MPA (geopolymer mortar with 10%  
464 MPCM) compared to the mortar without MK. The same effect of the addition of MK on the increase of  
465 compressive strengths is observed also at 90 days. On the contrary, the addition of 10% MK did not  
466 show any improvement in the compressive strength for geopolymer mortars without MPCM. About this  
467 observation, the CASH and NASH gels created after the addition of MK showed a significant effect on  
468 the mortars that have both concentrations of MPCM. Indeed, these mortars showed a high porosity,  
469 while CASH and NASH gels are generally used to fill small pores [51]. This observation may be the  
470 reason for the improved compressive strength.

471 In the case of the addition of 20% MK, the compressive strength is almost the same in the case of 10%  
472 MK compared to the geopolymers with 5 and 10% MPCM, but for the geopolymers without MPCM,  
473 the strength was increased from 69.9 to 78.5 MPA at 28 days and it reaches 81.6 MPA at 90 days. This  
474 effect of increased compressive strength can be explained by the small particle size and large specific  
475 surface area of MK compared to GBFS, which allow for accelerated reactivity [31,62] on the one hand,  
476 and the large amount of  $Al_2O_3$  and  $SiO_2$  in MK. These two effects led to an improvement in  
477 geopolymerization by producing sodium aluminosilicate hydrate (NASH) gel and calcium silicate  
478 hydrate (CASH) gel in addition to the CASH produced by the activation of GBFS, which are rich in  
479 calcium [34].

480 In a matrix that contains both NASH and CASH, the geopolymerization process proceeds through the  
481 following steps:

482 1. The NASH gel is formed in a system containing Na<sub>2</sub>O-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub>-H<sub>2</sub>O and starts with the  
483 dissolution and hydrolysis of the silica and alumina of the aluminosilicate material to form Si-  
484 OH and Al-OH bonds and continues with a condensation. The hydrolysis of Si and Al is  
485 presented in the following equations (2), (3), (4) [30,63]:



489 However, the activator content (Na and Si concentrations) has a very important role in the  
490 condensation process, which strongly influences the properties of the geopolymer [30]. The use of  
491 a high activator content as in this study favored the condensation to form oligomeric silicates, which  
492 generate rigid polymeric 3D structures [poly (sialate-siloxo) and poly (sialate-disiloxo)] with Si/Al  
493 ratios greater than 2. Otherwise, the use of a low concentration of activator results in poly (sialate)  
494 polymer structures with Si/Al ratio equal to 1 and accompanied by low mechanical strength [30].

495 The EDS analyses in section 3.2 confirm that the Si/Al ratio is higher than 2, which explains the  
496 better mechanical strengths found in this study.

497 2. The CASH gel is formed in a system containing CaO-Na<sub>2</sub>O-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> -H<sub>2</sub>O. Calcium  
498 influences in a very important way the acceleration of the hardening and the improvement of  
499 the mechanical performances of the geopolymer because of its additional nucleation sites  
500 [30,34]. This may be one of the reasons for the improvement in mechanical performance, and  
501 the EDS analysis shows that the calcium content was increased after the addition of MK. The  
502 increase in this content confirms that it was indeed involved in the formation of both gels. These  
503 are two mechanisms cited by other researchers [30,40,64] to explain how calcium is involved  
504 in the geopolymer system. The first states that Ca<sup>2+</sup> acts as a charge balancer and this allows it  
505 to be integrated into the geopolymer network. The second mechanism indicates that Ca is a  
506 contributing element to the formation of a CASH gel which has the possibility to coexist with  
507 the NASH geopolymer gel as explained above.

508 The reason for the increase in mechanical performance may be that the CASH and NASH gels  
509 caused by MK activation filled the pores caused by MPCM, which was not observed in the  
510 geopolymer with 0% MK.

511 The other reason for the improved mechanical performance may be related to the strong chemical  
512 structure of the geopolymer after the addition of MK. This effect is confirmed by EDS, which shows  
513 that the silica content increased after the addition of MK, which increased the Si/Al ratio. With the  
514 increase in Si content, more silicate species are available for condensation and reaction between  
515 them, resulting in more oligomeric silicates and they become dominant [62]. Researchers have  
516 shown that oligomeric silicates of Si-O-Si bond are stronger than those of Si-O-Al and Al-O-Al.  
517 This can explain the increase in compressive strength of geopolymer mortars after the addition of  
518 MK, on the other hand [14,65].

### 519 **3.5.2. Dynamic Young's modulus**

520 The results of the Young's modulus at different times (2, 7, 14, 21, 28, 56 and 90 days) are shown  
521 in figure 10. First, it can be noticed that this modulus develops with the curing time for both types  
522 of mortar. This observation is an indication that the hydration of the cement and the  
523 geopolymerization remain continuous during this time. Comparing the hydration time for cement  
524 mortar and geopolymer, it is observed that the values of this modulus continue to increase for cement  
525 mortars up to 90 days, while for geopolymer their values have been stabilized from the 14th day.  
526 This is due to the very rapid acceleration of the mechanical strength of these materials compared to  
527 cement-based materials [60].

528 Contrary to the compressive strength values, the Young's modulus values of the cement-based  
529 mortar are higher than those of the geopolymer mortar. Although this effect has been granted  
530 according to the literature [14,58,66], which cites that geopolymer possesses low rigidity, in contrast  
531 the values of this modulus in this study are very close to those of standardized mortars with a  
532 maximum value equal to 35.1 GPA at 90 days (geopolymer mortar with 20% MK without MPCM).

533 Figure 10 shows that the inclusion of MPCM decreased the Young's modulus for the two mortars  
534 (cement and geopolymer). These results are like the compressive strength results in the previous  
535 section (3.5.1).

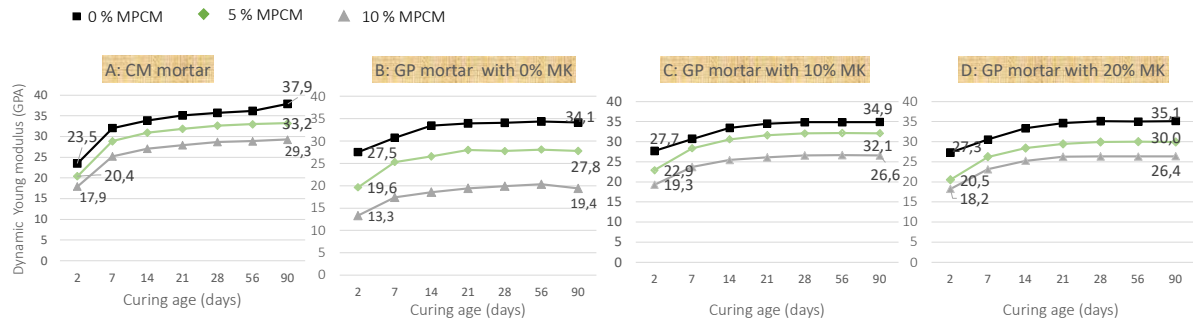
536 The values of Young's modulus of cement-based mortar decreased from 35.7 GPA to 32.6 and 28.6  
537 GPA with 5 and 10% MPCM at 28 days. This decrease after the incorporation of MPCM was  
538 accorded with the study of Šavija et al. [67]. Moreover, the same effects are observed in geopolymer  
539 mortars with 0% MK from which the values decreased from 34.0 to 27.7 and 19.8 GPA with 5 and  
540 10% MPCM.

541 The reason for the decrease in Young's modulus is explained in section 3.5.1 of compressive  
542 strength. The Young's modulus depends on the strength of the material [68], so the inclusion of  
543 MPCM decreases the rigidity of the matrix due to its low rigidity compared to sand. The second  
544 reason may be due to the porosity caused by these materials, which decreases the flexural resonance  
545 frequency ( $f_f$ ) while the latter is the main element that controls this modulus.

546 Figures 10.C and 10.D show that the MK inclusion rates improved the Young's modulus for all  
547 geopolymer samples. The addition of 10% MK increased the Young's modulus value of geopolymer  
548 mortars from 34.1 to 34.9 GPA (0% MPCM) and from 27.7 to 32.1 GPA (5% MPCM) and from  
549 19.9 to 26.6 GPA (10% MPCM).

550 Similar remarks are reported in Figure 10.D (20% MK), which shows that the addition of 20% MK  
551 increased the Young's modulus values to 35.1, 29.9 and 26.4 GPA.

552 The optimum MK content is 10% for geopolymer-MPCM mortars, but for geopolymer mortars  
553 without MPCM, the optimum content is 20% MK. These results are in good agreement with the  
554 compressive strengths and can be explained by the pore filling effect caused by the activation of  
555 silica and alumina in the MK.

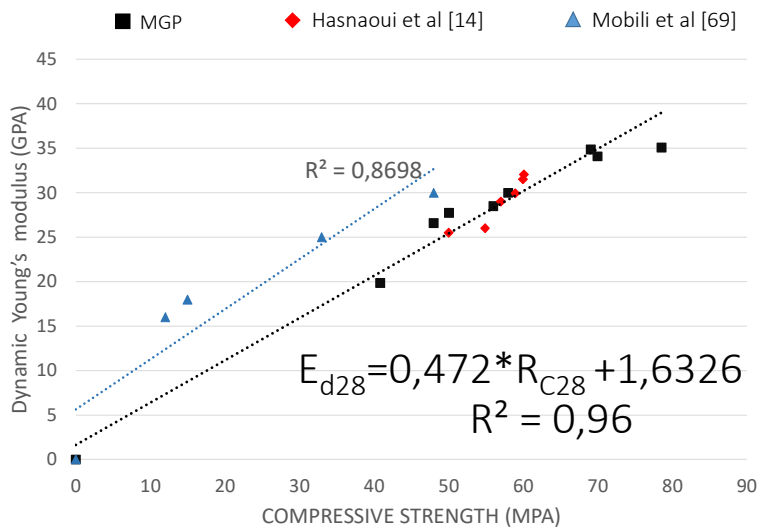


556

557 Figure 10. Young's modulus as a function of curing time, A: (cement-based mortar), B:  
 558 (Geopolymer mortar with 0% MK); C: (Geopolymer mortar with 10% MK); D: (Geopolymer mortar  
 559 with 20% MK).

560 Figure 11 represents a comparison of dynamic Young's modulus results on geopolymer mortars with  
 561 other researchers who have used dynamic methods to evaluate the values of this modulus [14,69].

562



563

564 Figure 11. Correlation between dynamic Young's modulus and compressive strength at 28 days.

565 According to figure 11, there is a good correlation between this modulus and the compressive  
 566 strength expressed by a linear variation with a correlation coefficient  $R^2 = 0.96$  for our geopolymer  
 567 mortars and those of Hasnaoui et al. [14] and a linear correlation for the mortars of Mobili et al.

568 [69]. Indeed, Duxson et al. [65] indicated that the Young's modulus of the geopolymer is correlated  
569 with its compressive strength.

570 The results presented in this study are higher than the reference results mentioned. This is probably  
571 the consequence of the use of 80/20 GBFS and MK, in contrast to Hasnasoui et al. [14] who used  
572 50/50 GBFS and MK, and the use of Mobili et al. [69] up to 100% fly ash. The CASH gel formed  
573 by GBFS activation is denser compared to the NASH gel and has a higher pore filling capacity  
574 [70,71], which may explain the better mechanical performance obtained in this study compared to  
575 the two references above.

### 576 **3.6. Thermal properties**

577 Figure 12 shows the thermal properties of two types of mortars at different measuring temperatures.  
578 These measurements were performed in a temperature range that is between 15 °C (MPCM in solid  
579 state) and 40°C (MPCM in liquid state). It can be clearly shown that the rate of increase of MPCM  
580 concentration caused a decrease in the thermal conductivity of two mortars (cement-based mortar  
581 and geopolymer-based mortar) independently of the measurement temperature. The reason for this  
582 decrease in the two cases can be explained by the two effects below:

- 583 • The first effect is attributed to the low thermal conductivity of MPCM (about 0.2 W/m.K),  
584 which replaced sand while the latter has a higher thermal conductivity. This effect was  
585 confirmed by the measurements that were carried out in the materials section (2.1).
- 586 • The second effect is the appearance of voids in the matrix after the incorporation of MPCM  
587 (see figure 8). These voids will transfer the heat flow from the state of conduction to the  
588 state of convection, resulting in a decrease in thermal conductivity.

589 However, the thermal conductivity in the solid state (measured at 15°C) of the two mortars with the  
590 two MPCM concentrations is higher compared to the thermal conductivity in the liquid state. This  
591 is confirmed by the study of Cui et al. [25] and Cao et al. [7] and due to the thermal conductivity of  
592 the solid state paraffin, which is generally higher than its thermal conductivity in the liquid state.  
593 Additionally, Sasaguchi et al. [72] have shown that the thermal conductivity of paraffin-type phase

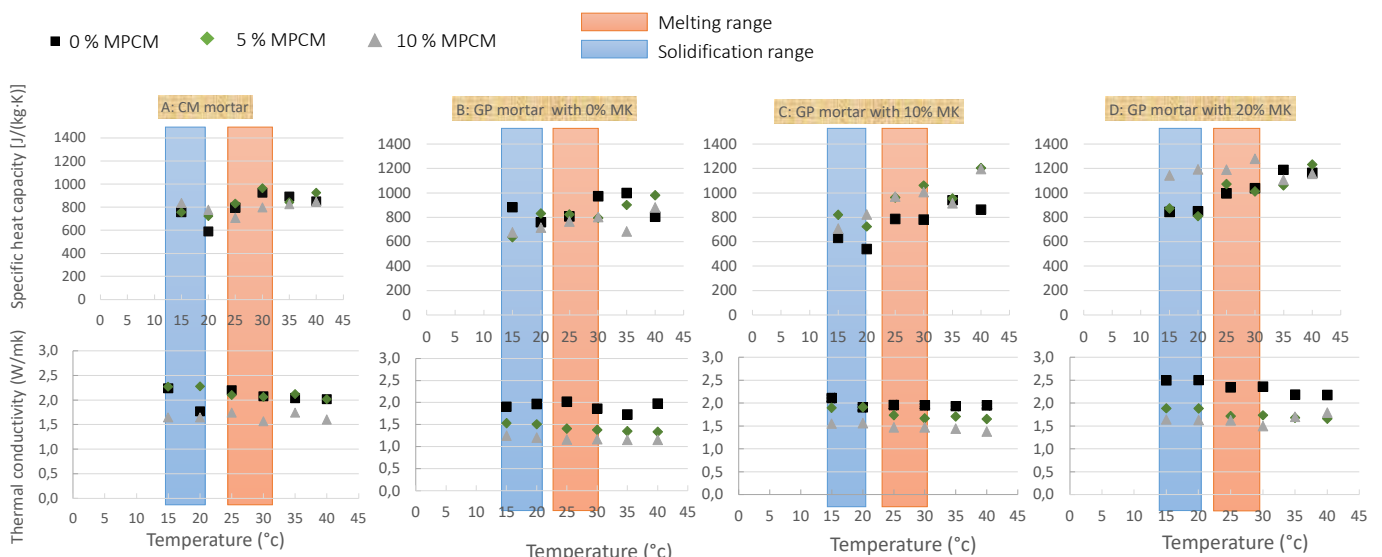
594 change material (n-octadecane) in the solid state is equal to 0.358 W/mK while in the liquid state is  
595 equal to 0.148 W/mK.

596 Nevertheless, the MPCM did not show an improvement in the specific heat capacity of the cement  
597 mortars and that of the geopolymer without MK (figures 12 A and B).

598 This consequence can be related to the low thermal conductivity of the samples presented in figure  
599 12 and due to the presence of more voids which appeared (figure 8 A). Indeed, these voids did not  
600 allow the ambient heat to transmit to the microcapsules to be stored on the one hand and can be  
601 related to the hot disk sensor which did not capture the latent heat stored by the MPCM on the other  
602 hand.

603 These results are not in agreement with the following references [9,18,73] which found an  
604 improvement in heat capacity with the use of the DSC method. When using the DSC method, the  
605 sample tested is in the form of a homogeneous powder with a mass of about 1-10 mg. Therefore, this  
606 method does not consider thermal conductivity, unlike the hot disk method used in this study.

607



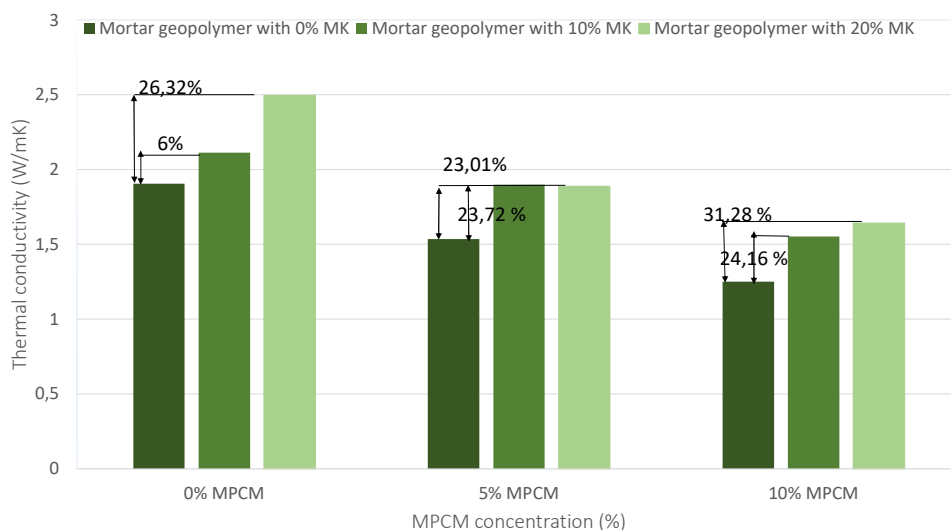
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610 Figure 12. Thermal properties at 28 days; A: (cement-based mortar), B: (Geopolymer mortar with  
 611 0% MK); C: (Geopolymer mortar with 10% MK); D: (Geopolymer mortar with 20% MK).

612 In contrast to geopolymer mortars without MK, all samples with 10 and 20% MK showed the highest  
 613 specific heat capacity at the melting point of MPCM (between 25 and 30°C) and at the solidification  
 614 point (between 15 and 20°C). The highest value of specific heat capacity reached 1280.76 J/Kg.K at  
 615 the melting range for MGP<sub>80/20/10</sub> (20% MK and 10% MPCM). This value is close to the one that is in  
 616 the study of Cao et al. [7] who found a value of 1500 J/kg.K at the melting point of MPCM while they  
 617 used up to 72 kg/m<sup>3</sup> of MPCM while in this study the concentration of MPCM is equal to 56.8 kg/m<sup>3</sup>.  
 618 The same improvements were accorded by Shadnia et al. [18], who found an improvement in specific  
 619 heat capacity up to 1200, 33 J/kg.K. With this value of specific heat capacity, the conclusion of the  
 620 study by Shadnia et al. [18] indicates that the internal temperature measurements of two cells  
 621 constructed with geopolymer-MPCM mortar showed a reduction of 4.5 and 5.5 ° C compared to a  
 622 reference cell that does not contain MPCM. In this case, the improvement of the specific heat capacity  
 623 is related to the increase of the thermal conductivity.

624 Figure 13 is extracted from Figure 12. It shows the effect of the addition of MK on the thermal  
 625 conductivity of geopolymer mortars in the solid state where the measurements are taken at 15°C. This  
 626 point was chosen because the effect of adding MK on the thermal conductivity is roughly similar  
 627 compared to the other measurement points.

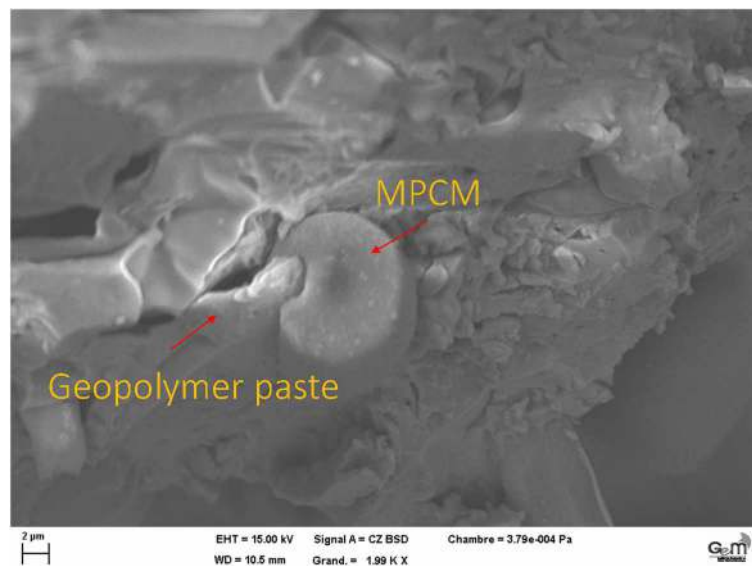




629 Figure 13. Thermal conductivity of geopolymer mortars at 15°C as a function of MPCM  
630 concentration before and after MK addition.

631 In Figure 13, it is clearly shown that the inclusion of two MK rates (10 and 20%) increased the  
632 thermal conductivity by 6 and 26.32% for the geopolymer mortar with 0% MPCM. This increase  
633 translates into an improvement in thermal conductivity of about 0.137 W/mK and 0.52 W/mK. On  
634 the other hand, an improvement in thermal conductivity of 23.01; 23.72; 24.16 and 24.16% is  
635 observed for geopolymer mortars with 5 and 10% MPCM. The increase of thermal conductivity  
636 after the inclusion of MK can be explained by the decrease of voids in the matrix due to the creation  
637 of gels in the geopolymer matrix, as several researchers have mentioned the advantage of the good  
638 reactivity of MK [14,34,53]. In addition, Ibrahim et al. [51] pointed out that the high Si content  
639 observed in the EDS leads to the filling of the pores of the studied matrix.

640 Figure 14 was obtained from the SEM for MGP<sub>80/20/20</sub> (20% MK and 20% MPCM). It shows a good  
641 cohesion of the geopolymer paste with the MPCM, which can explain the improvement of the  
642 thermal conductivity of the total matrix after the addition of MK and might be due to the increase  
643 of the thermal flux that was transmitted during the measurement.

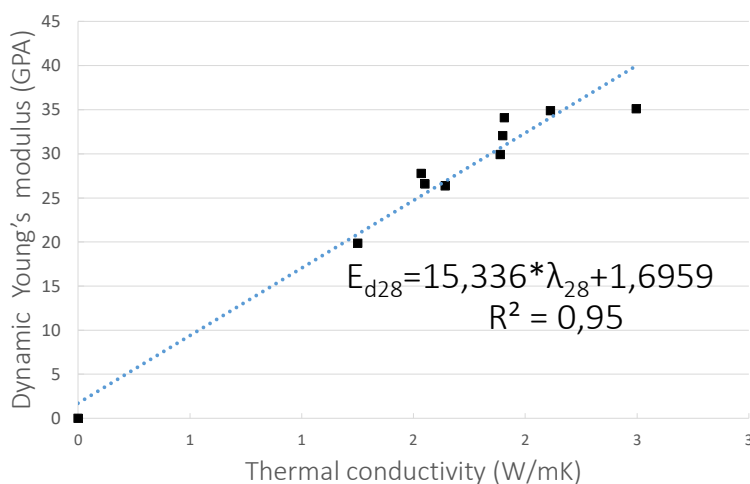


644  
645 Figure 14. Cohesion of the geopolymer paste with MPCM.

646

647 XU et al. [74] added carbon nanotubes to a cement matrix to increase its thermal conductivity after  
648 incorporation of PCM (without capsule). They found an increase of about 45% in thermal conductivity.  
649 Their study reveals that the increase in thermal conductivity improved the heat storage performance of  
650 PCM. On the other hand, they monitored the time taken for the temperature to increase from 27 to 31°C.  
651 Their results showed that the heat storage and release time decreased compared to the reference sample  
652 by about 181 seconds for the heat storage process and 266 seconds for the heat release process.  
653 Increasing the thermal conductivity of geopolymer mortars will help the MPCM to charge and discharge  
654 its latent heat, thus improving its thermal performance in actual construction.

655 Figure 15 shows a correlation between the Young's modulus and thermal conductivity of geopolymer  
656 mortars at 28 days. A linear correlation expressed by a correlation coefficient  $R^2 = 0.95$  is noted. These  
657 two properties are dependent on the density of the material, which may explain this linear correlation.  
658 A similar correlation between dynamic Young's modulus and thermal conductivity is presented in the  
659 study of Barnaure et al. [75] for other material constructions.



660  
661 Figure 15. Correlation between dynamic Young's modulus and thermal conductivity at 28 days.

#### 662 4. Conclusion

663 The objective of this study was to use the coexistence of two NASH and CASH gels by the inclusion  
664 of metakaolin to overcome the negative effects of MPCM incorporation in blast furnace slag-based

665 geopolymer mortars. The conclusions of the different tests after the inclusion of metakaolin are as  
666 follows:

- 667 • The geopolymer-MPCM mortars showed good workability with up to 10% MPCM and 20%  
668 MK. The maximum flow time was equal to 4.9 seconds, which is lower than the standard mortar  
669 flow time of 7 seconds.
- 670 • SEM, EDS and XRD analysis confirmed that incorporation of metakaolin into the blast furnace  
671 slag matrix produced CASH and NASH gel as stable reaction products.
- 672 • EDS analysis revealed a higher Si/Al ratio, which means it has the potential to produce a dense  
673 and compact bonding geopolymer.
- 674 • The compressive strength of geopolymer-MPCM mortars is between 49 and 61 MPa and is  
675 sufficient for structural applications.
- 676 • The CASH and NASH gels formed after the addition of MK increased the thermal conductivity,  
677 and the latter improved the specific thermal capacity of geopolymer-MPCM mortars. The  
678 maximum value obtained is equal to 1280 J/Kg.K This value is higher than the thermal capacity  
679 values of Portland cement mortars.

680 Finally, the increase in MPCM rate showed a reduction in mechanical strength and an increase in  
681 porosity, which limit the amount of MPCM that can be added to the geopolymer mortar. Future research  
682 could focus on developing microcapsules with low agglomeration, excellent compatibility with the  
683 concrete matrix and high mechanical strength (robust microcapsule) to overcome these constraints.  
684 These could be a viable option to reduce the loss of compressive strength and the increase of porosity.  
685 On the other hand, this solution will increase the amount of MPCM in the geopolymer, thus increasing  
686 the total thermal energy storage capacity of the geopolymer.

#### 687 **Declaration of Conflict of Interests**

688 The authors declare that they have no known competing financial interests or personal relationships that  
689 could have appeared to influence the work reported in this paper.

#### 690 **Credit authorship contribution statement**

691 **Bouha EL MOUSTAPHA:** Conceptualization, Methodology, Investigation, Writing - original draft.  
692 **Stéphanie BONNET:** Validation, Resources, Visualization, Supervision, review & editing. **Abdelhafid**  
693 **KHELIDJ:** Validation, Resources , Visualization, Supervision, review & editing. **Nordine LEKLOU :**  
694 Validation, review & editing. **Daniel FROELICH:** Validation, review & editing , Supervision.  
695 **Isselmou AHMEDOU BABAH:** review & editing, Supervision. **Carole CHARBUILLET:** review &  
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697

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703

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