EVALUATION OF THE POZZOLANIC ACTIVITY OF METAKAOLIN, GLASS POWDER AND SILICA POWDER FOR USE IN CEMENTITIOUS MORTARS

Issiaka SANOU^a, Saada ZOUNGRANA^a, Moussa OUEDRAOGO^a, Ali SANOU^{b,c,*}, Younoussa MILLOGO^a

ABSTRACT. The objective of this study is to evaluate the pozzolanic activity of metakaolin, glass powder and silica powder for use in cementitious mortars. Thus, metakaolin was produced by calcination at 700 °C of a clay soil from Burkina composed of kaolinite (62 wt.%), quartz (30 wt.%) and goethite (6 wt.%). Glass powder consists of amorphous silica and silica powder essentially contains guartz. The chemical characterization of materials showed that the metakaolin and the silica powder belong to the category of type F pozzolans while the silica powder would be type N. The lime saturation test reveals low kinetics of lime fixation by the silica powder. On the other hand, the rate of lime fixation by metakaolin and glass powder increases with treatment time. The pozzolanic index of metakaolin and glass powder at 28 and 90 days is higher than the minimum value of 75% required by the ASTM C618 standard. The presence of metakaolin and glass powder within the cement matrix improves the compressive strength of the resulting mortars due to their good pozzolanic reactivity inducing the formation of calcium silicates hydrated (CSH). Metakaolin and glass powder are therefore suitable for replacing cement in the production of mortars in the construction field.

Keywords: Glass powder, Metakaolin, Silica powder, Cementitious mortars, Compressive strength.

©2024 STUDIA UBB CHEMIA. Published by Babeş-Bolyai University.



This work is licensed under a Creative Commons Attribution-NonCommercial-NoDerivatives 4.0 International License.

^a Université Nazi BONI, Unité de Formation et de Recherche en Sciences Exactes et Appliquées (UFR/SEA), Laboratoire de Chimie et Energies Renouvelables (LaCER), Bobo-Dioulasso, B.P. 1091 Bobo 01, Burkina Faso.

^b Institut National Polytechnique Félix Houphouët Boigny, Unité Mixte de Recherche et d'Innovation en Sciences Agronomiques et Procédés de Transformation (SAPT), Laboratoire des Procédés Industriels de Synthèses de l'Environnement et des Energies Nouvelles (LAPISEN), BP 1313 Yamoussoukro, Côte d'Ivoire.

^c Ecole Normale Supérieure, Laboratoire des Sciences Physiques Fondamentales et Appliquées (LSFPA), 08 BP 10 Abidjan, Côte d'Ivoire.

^{*} Corresponding author: ali.sanou@inphb.ci / sanouali2007@yahoo.fr

INTRODUCTION

Cement is a necessary material for carrying out construction projects. Unfortunately, its production is energy-intensive and not ecological [1-3]. Also, cementitious materials are confronted with certain pathologies such as alkaline reactions, attacks by sulfate ions and the diffusion of chloride ions, which considerably reduce the lifespan of cementitious products [4, 5]. To remedy these problems linked to cement, several solutions are being considered, including the use of pozzolanic additions to cement production [6]. Among these pozzolanic additives, metakaolin and glass powder have been the subject of several scientific studies [7–10].

Indeed, metakaolin is a material obtained by dehydroxylation of kaolinite between 650 °C and 850 °C [11]. Its use as a cement additive makes it possible to improve the properties of composites in terms of durability. mechanical strength and resistance to chemical attack [12, 13]. This is how the studies carried out by Sinngu et al showed an increase of approximately 30% in the long-term compressive mechanical strength of mortars containing metakaolin compared to the reference mortar [14]. Also, the drying shrinkage of the mortars was reduced by approximately 50%, after the incorporation of 10 to 15 wt.% of metakaolin in the mixtures. As for the glass powder, it comes from the crushing of glass bottles. In most developing countries, glass bottles are most often thrown into the environment after use, which constitutes environmental waste. Its use as a pozzolan constitutes a solution to preserving the environment. The work carried out on glass powder has also produced convincing results [7, 15]. For Wang et al, the initial mechanical strength of mortars containing glass powder is greater than that of the reference mortar not containing glass powder [15]. Furthermore, to our knowledge, little work has been devoted to the pozzolanic reactivity of silica powder [16]. Although glass powder and metakaolin have been the subject of studies with a view to their valorization as cement additives, the substitution rates vary from one author to another author. Thus, according to Harbi et al, the addition of 25 wt.% metakaolin in mortars contributes to improving their mechanical resistance and durability thanks to its pozzolanic character which reduces porosity and also water absorption [7]. For Malla et al, the addition of 12 wt.% metakaolin and 10 wt.% glass powder leads to a considerable improvement in the split tensile strength of concrete and can therefore be recommended as a potential mixture [17].

Thus, the objective of this present work is to evaluate the pozzolanic activity of metakaolin, glass powder and silica powder for use in cementitious mortars. This involves developing ecological materials that can optimally replace cement in the formulation of mortars. To do this, the materials will first

be characterized and then their pozzolanic activity will be evaluated through Frattini tests, lime saturation and determination of the pozzolanic index. Finally, their influence on the compressive mechanical strength of mortars will be studied.

RESULTS AND DISCUSSION

Materials characterization

The heat treatment of clayey materials results in the dehydration of some of its components. Thus, the hydroxyl groups of goethite and kaolinite become detached from the initial structures. In order to better understand the dehydroxylation process, infrared spectra of raw clay and metakaolin obtained by calcination at 700 °C with a heating rate of 10 °C/min for 2 hours were carried out. Figure 1 highlights the comparison of the infrared spectra of the raw clayed material and that thermally activated at 700 °C.



Figure 1. Infrared spectra of raw clay and metakaolin

The analysis of Figure 1 shows that all the characteristic bands of kaolinite have disappeared. Thus, at high frequencies, the disappearance of the bands at 3689 cm⁻¹ and 3620 cm⁻¹ of the hydroxyl groups of kaolinite is due to the effect of heat treatment [9,18]. Furthermore, the characteristic bands of the Si-O bond of kaolinite at 1114 cm⁻¹ and 1028 cm⁻¹, initially present in the raw clay, were transformed into a single wide band around 1055 cm⁻¹ attributed to the asymmetry of Si–O–Al and Si–O–Si vibrations of amorphous

silica [19]. The deformation vibration band of adsorbed water contained in the raw material at 1642 cm⁻¹ also disappeared [20]. The band at 911 cm⁻¹ of the AI-OH bond of kaolinite is also absent. The disappearance of the Si-O-AI bands (788 cm⁻¹ and 750 cm⁻¹) of kaolinite against the band at 752 cm⁻¹ for metakaolin seems to indicate a distortion of the tetrahedral layers of the SiO₄ group and the octahedral layers. The infrared spectrum of the glass powder (Figure 2) shows only two bands around 1000 cm⁻¹ and 764 cm⁻¹ which could be attributed to amorphous silica. The bands at 1140 cm⁻¹ and 775 cm⁻¹ of the silica powder are due to the Si – O – Si bond of the quartz [9].



Figure 2. Infrared spectra of glass powder and silica powder

It appears from the analysis of the infrared spectra that the heat treatment at 700 °C results in the transformation of the kaolinite from the clay raw material into an amorphous phase which is metakaolinite.

The diffractogram of the glass powder in Figure 3 shows a halo around 22° (20) showing the disorder reigning in the material. This disorder could characterize the presence of amorphous phases within the glass powder [21]. Silica, the main element of glass powder, is then in amorphous form and will give it good pozzolanic reactivity [22]. Analysis of the diffractogram (Figure 4) of the silica powder shows that it is essentially made up of quartz. The chemical composition of the materials is recorded in Table 1. Analysis of the table shows that the samples are mainly composed of silica. Metakaolin is particularly rich in alumina and iron oxide due to the mineralogical composition of the clayey raw material. Glass powder contains a high content of SiO₂,

EVALUATION OF THE POZZOLANIC ACTIVITY OF METAKAOLIN, GLASS POWDER AND SILICA POWDER FOR USE IN CEMENTITIOUS MORTARS

CaO and Na₂O. Furthermore, the sum of the oxides SiO₂, Al₂O₃ and Fe₂O₃ is greater than 70 wt.%, according to the ASTM C 618 standard, metakaolin, and silica powder belong to the class of type F pozzolans [23]. However, the CaO content of the glass powder is greater than 10 wt.%, so it belongs to the category of N-type pozzolans [24].



Figure 4. The diffractogram of silica powder

Oxide (wt.%)	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	Na₂O	K₂O	TiO ₂	LOIª
MK	59.38	24.64	5.36	0.08	0.04	0.08	0.10	0.47	9.68
GP	76.77	1.17	0.48	1.65	11.15	10.28	0.40	-	0.57
SP	86.67	9.82	0.10	0.05	0.13	0.16	0.07	0.3	2.7

 Table 1. Chemical composition of materials

MK : metakaolin ; GP : glass powder ; SP : silica powder ; ^a Loss on ignition at 1000 °C

Pozzolanic activity of materials

Results of Frattini and lime saturation tests

The results of the Frattini test are shown in Figure 5. Analysis of the figure shows that the silica powder does not exhibit any pozzolanic activity because it is above the solubility curve of portlandite. On the other hand, glass powder and metakaolin are found below the solubility curve of portlandite, which highlights their pozzolanicity [1, 24]. The results of the lime saturation test are presented in Figure 6. Analysis of the figure shows that the lime fixation rate of the materials is a function of the treatment time. From the first 24 hours, metakaolin presents strong lime consumption kinetics with a fixation rate greater than 50%. This strong reactivity of metakaolin is due to its chemical composition. In fact, metakaolin is rich in silica, alumina and iron oxide which constitute the reactive phases of a pozzolan. According to Seynou et al, pozzolanic reactivity at a young age is governed by the chemical composition, more precisely the silica, alumina and iron oxide content of a pozzolan [9]. Glass powder and silica powder have a rate fixation of 35.26 and 36.92% respectively, these values are slightly higher than the value of 35% which constitutes the lime fixation rate at this age of commercial metakaolins [16]. From 3 days, the pozzolanic reactivity of the glass powder increases significantly with a lime fixation rate of more than 99% at 28 days. The good pozzolanic reactivity of glass powder would be due to its amorphous character [22]. Also, metakaolin has good reactivity between 7 and 28 days but less than glass powder. The lime fixation rate of silica powder is low from the 3rd day and becomes almost constant between 7 and 28 days. This low reactivity would be due to the high guartz content in the silica powder which would constitute an impurity and therefore hamper the pozzolanic reactivity.

EVALUATION OF THE POZZOLANIC ACTIVITY OF METAKAOLIN, GLASS POWDER AND SILICA POWDER FOR USE IN CEMENTITIOUS MORTARS



Figure 6. Results of lime saturation test of materials

Pozzolanic activity index

The pozzolanic activity index was evaluated from the mechanical compressive strength at 7, 28 and 90 days of age of the mortars containing a partial replacement of 25 wt.% of Portland cement by the materials and those containing only cement. The various results obtained are recorded in

Table 2. Analysis of the table shows that the evolution of the pozzolanic index of the materials is almost similar to their lime fixation kinetics highlighted by the lime saturation test. Thus at 7 days, metakaolin displays the best pozzolanic index. From 28 days to 90 days, glass powder presents the highest value of the pozzolanic index followed by metakaolin. Silica powder has a pozzolanic index that is almost low and constant over time. The pozzolanic index values of the materials are then due to their pozzolanic reactivity. Indeed, pozzolanic reactivity favors the consumption of portlandite in favor of calcium silicates hydrated (CSH), hydrates responsible for the mechanical strength of the mortar. Mortars containing metakaolin and glass powder at 28 and 90 days of age display a pozzolanic index higher than the minimum value of 75% required by the ASTM C 618 standard for a pozzolanic material [23].

Motoriolo	Pozzolanic index					
Materials -	7 days	ays 28 days	90 days			
Glass powder	71.43	92.52	106.19			
Metakaolin	78.12	82.64	93.99			
Silica powder	67.59	73.35	74.86			

Table 2.	Pozzolanic	index	of materials
----------	------------	-------	--------------

Mineralogical characterization

The pozzolanic reactivity of the materials was monitored through the analysis of infrared spectra (Figure 7) carried out on the specimens produced after 28 days of curing. Table 3 summarizes the different bands obtained as well as their attribution. The portlandite (CH) band at 3600 cm⁻¹ is intense at the level of the specimens containing silica powder, thus showing its strong release in this mixture. On the other hand, this band is present in metakaolinlime and glass-lime powder mixtures but less intense. Also, we note an absence of the band around 3500 cm⁻¹ of calcium silicates hydrated for the mixture containing silica powder and lime. This reflects the low or nonpozzolanic reactivity of this material as highlighted by the Frattini tests, lime saturation and the pozzolanic index. Indeed, in the presence of water, amorphous silica reacts with lime to form CSH, thus reflecting the pozzolanic reactivity of the material. The calcium silicates hydrated are obtained in two ways. First, when mixing cement with water, the dicalcium silicates (C_2S) and tricalcium silicates (C_3S) of cement react with the water molecules, thereby producing hydrated calcium silicates (CSH) and portlandite (CH) following reactions (1) and (2) [18, 25]:

$$2 C_3 S + 6 H \rightarrow C_3 S_2 H_3 (CSH) + 3 CH$$
 (1)

$$2 C_2 S + 4 H \rightarrow C_3 S_2 H_3 (CSH) + CH$$
 (2)

EVALUATION OF THE POZZOLANIC ACTIVITY OF METAKAOLIN, GLASS POWDER AND SILICA POWDER FOR USE IN CEMENTITIOUS MORTARS

CSH are also formed by pozzolanic reactions involving amorphous silica and portlandite previously released by the hydration of cement. This pozzolanic reaction takes place in three phases described by reactions (3), (4) and (5) [26]:

Hydration of lime to produce portlandite: $CaO + H_2O \rightarrow Ca(OH)_2$ (3) Dissolution of portlandite: $Ca(OH)_2 \leftrightarrow Ca^{2+} + OH^-$ (4)



Figure 7. Infrared spectra of specimens

Formation of CSH after solubilization of silica: $Ca^{2+} + 2OH^{-} + SiO_2 \rightarrow CSH$ (5)

The presence of calcite is highlighted through the bands around 1400 and 874 cm⁻¹. This calcite (CaCO₃) comes from the carbonation of portlandite $(Ca(OH)_2)$ following the chemical reaction (6) [18]:

$$Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O \tag{6}$$

Table 3.	Bands	attribution
----------	-------	-------------

Wave number (cm ⁻¹)	attribution	References
3640	Portlandite (CH)	[9]
3500-3400	CSH hydrate	[9,26]
1640	Hydration water	[27]
1400-1440	Calcite (CaCO ₃)	[28]
874	Calcite (CaCO ₃)	[26]

Influence of materials on the mechanical strength of cement mortars

After the study of pozzolanic reactivity, cementitious mortars were manufactured by partial substitution of cement with different materials. Then, their mechanical compressive behavior was studied. Figure 8 shows the evolution of the mechanical compressive strength after 90 days of curing depending on the content of the materials in the cementitious mortars. Analysis of the figure shows that the evolution of the mechanical strength is of the same order as the pozzolanic reactivity of the materials. Indeed, according to the results of the lime saturation test in Figure 6, glass powder had the best lime fixation rate followed by metakaolin. Similarly, the pozzolanic indices from 28 days of glass powder (Table 2) were higher than those of metakaolin and silica powder.

Thus, mortars containing 20 wt% of glass powder present the best compressive strength values (54.8 MPa) followed by mortars amended with 15 wt% of metakaolin (49.55 MPa). The mechanical compressive strength of mortars containing silica powder drops beyond 10 wt.% substitution. The improvement in the mechanical resistance of mortars would be due to the formation of CSH resulting from the hydration reaction of the anhydrous compounds of cement (C_3S and C_2S) and by pozzolanic reaction following reactions (1), (2), (3), (4) and (5) described previously [18, 25].

For Suzan et al, the improvement in the compressive strength of cement mortars amended with metakaolin is mainly attributed to the pozzolanic effect of metakaolin present in the cement matrix which could be detailed as follows: when water is added to the cement-metakaolin mixture, free calcium hydroxide (CH) is released and its concentration increases due to the initial hydration of the cement [29]. The silica and alumina of metakaolin react with calcium hydroxide to form secondary hydration products mainly in the form of hydrated calcium silicate (CSH) with the formation of calcium aluminate hydrates (C_2ASH_8 , C_4AH_{13} , C_3AH_6) which were precipitated upon saturation according to the reaction equation (7):

$$\begin{array}{c} \mathsf{Ca}(\mathsf{OH})_2 + \mathsf{Mk}(\mathsf{Al}_2\mathsf{O}_3.\ 2\mathsf{SiO}_2) + \mathsf{H}_2\mathsf{O} \to \mathsf{CSH}\ (2^{ry}\ hydration\ product) + \\ \mathsf{C}_2\mathsf{ASH}_8,\mathsf{C}_4\mathsf{AH}_{13},\ \mathsf{C}_3\mathsf{AH}_6 \end{array} \tag{7}$$

With :
$$C = CaO$$
; $S = SiO_2$, $A = AI_2O_3$ and $H = H_2O$

These secondary hydration products serve as micro-fillings which leads to a reduction in the total porosity of the pastes and therefore an increase in the total content of the bonding centers in the mortars consequently causing an increase in their compressive strength values. According to Wang et al, the improvement in the mechanical compressive strength of cement mortars amended with glass powder would be attributed not only to their pozzolanic reaction but also to the filler effect of filling the pores of the mortars with the powder [15]. The low mechanical resistance values of mortars containing silica powder are due to their low pozzolanic reactivity which would be attributable to their crystalline structure. According to studies carried out by Seynou et al, quartz has no pozzolanic reactivity, only its filler character can contribute to improving the mechanical resistance of mortars [9].

The reduction in the mechanical compressive strength of mortars amended with metakaolin and glass powder would be due to the excessive presence of these materials in the mortars. Indeed, the presence of impurities in metakaolin could negatively influence the formation of hydrates responsible for the good mechanical strength of mortars. The drop in mechanical strength beyond 20 wt.% of the glass powder would be due to the low presence of portlandite to ensure pozzolanic reactivity.



Figure 8. Mechanical compressive strength at 90 days of different mortars

CONCLUSIONS

The objective of this study was to evaluate the pozzolanic activity of metakaolin, glass powder and silica powder for use in cementitious mortars. From the various results obtained, the following remarkable conclusions emerge:

(1) Glass powder and metakaolin consist of amorphous silica and silica powder contains crystalline quartz. Metakaolin and silica powder belong to the class of type F pozzolans and glass powder belongs to the category of type N pozzolans.

(2) the Frattini test showed that metakaolin and glass powder have pozzolanic activity while silica powder does not. The results of the lime saturation test showed rapid lime fixation kinetics of metakaolin at 1 day and from 3 days the glass powder, due to the amorphous structure, fixes lime more than other materials. Silica powder has a low and almost constant lime fixation rate over time.

(3) the values of the pozzolanic index of the materials are of the same order of evolution as their pozzolanic reactivity with an index higher than the minimum value of 75% fixed by the ASTM C618 standard from 28 days for glass powder and metakaolin.

(4) the partial replacement of cement with glass powder and metakaolin improves their compressive strength after 90 days of curing. This improvement is due to the pozzolanic reactivity of these materials and their effect of filling the pores within the cement matrix.

In short, the metakaolin and glass powder obtained could be used at respective contents of 15 wt.% and 20 wt.% as cement substitutes in the elaboration of mortars.

EXPERIMENTAL SECTION

Raw materials

Metakaolin was produced from Burkina clayey soil taken from the rural commune of Saaba (12° 22' North and 1° 26' West). This raw material has been the subject of previous scientific work [18,30]. Analysis of the diffractogram (Figure 9) of this raw material shows that it contains kaolinite, quartz and goethite as crystalline phases. The content of each of these phases is recorded in Table 4. The presence of kaolinite in high content in the clay raw material shows that it can be used for the formulation envisaged. Indeed, kaolinite is a precursor for obtaining metakaolinite, which is the reactive phase of metakaolins. Quartz improves the hydration of cement and contributes to the pozzolanic reaction [1]. Thus, metakaolin was produced by calcination at a temperature of 700 °C with a heating rate of 10 °C/min for 2 hours. The choice of this temperature is based on the analysis of the Differential thermal analysis (DTA) curve of the clay raw material so as to be located between the phenomena of dehydroxylation of kaolinite at 538 °C

and recrystallization of metakaolinite at 973 °C (Figure 10). Also, this value is between 650 and 850 °C which constitutes the temperature interval provided by the literature [11].

Indeed, thermal activation leads to the dehydroxylation of the kaolinite of the raw material into metakaolinite according to the reaction equation (8) [18, 31]:

 $Si_2O_5Al_2(OH)_4$ (kaolinite) $\rightarrow 2 SiO_2-Al_2O_3$ (metakaolinite) + 2H₂O (8)



Figure 9. X-ray diffractogram of clayey raw material



Figure 10. DTA curve of the clayey raw material

Mineral	Kaolinite	Quartz	Goethite	Total	Balance
Composition (wt. %)	62	30	6	98	2

Table 4. Mineral phases composition of clayey raw material

The glass powder was obtained by grinding to a particle size of 45 μm used glass which was collected from landfills in the town of Bobo Dioulasso in Burkina Faso.

As for the silica powder, it was taken from a quarry in the town of Bobo Dioulasso in Burkina Faso and it has also been the subject of previous work [32]. The cement used for the mortars manufacturing is a CEM II with strength class 42.5 MPa from the manufacturing company CIMAF in Burkina Faso. The chemical, mineralogical composition and some physical properties of this cement are recorded in Table 5.

|--|

Chemical composition	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO₃	K₂O	N ₂ O
(wt. %)	21.44	5.38	2.93	55.45	4.87	1.8	0.65	0.23
Mineralogy	C₃S	5	C	2S	C3	A	C4	AF
(wt. %)	17.3	8	48	.36	9.	3	8.	92
Physical	Apparent	Specif	ic area	Wa	ter	Setti	ng star	t time
Properties	density	(cm	1²/g)	conter	nt (%)		(min)	
Value	1.18	39	969	2	9		≤ 120	

Chemical and mineralogical characterization

The chemical composition of the materials was determined by inductively coupled plasma atomic emission spectrometry (ICP-AES) using an ICPE-9800 type spectrometer. The solutions were carried out by dissolving the samples with lithium tetraborate (Li2B4O7). The mineral phases within the samples were identified by coupling X-ray diffraction (XRD) and Fourier transform infrared spectrometry (FT-IR). The powder diffractogram was recorded with a Bruker AXS diffractometer using CuK α radiation as the radiation source and graphite monochromator. The infrared spectrometer used for the identification of the functional groups of mineral species is of the Perkin Elmer FT-IR type.

Chemical methods of pozzolanic activity

The effectiveness of a pozzolan in a cementitious matrix depends on its ability to react with the portlandite released during the hydration of the cement in order to form cementitious hydrates. This reactivity defines its pozzolanic activity. Chemically, this reactivity can be carried out by Frattini and lime saturation tests [16].

The Frattini test is one of the commonly used chemical methods to determine the pozzolanicity of a material. The Frattini test procedure involves preparing a 20 g mixture, consisting of 80 wt.% Portland cement with 20 wt.% material added to 100 mL of distilled water. The mixture is stored at a temperature of 40 °C for eight (08) days in tightly closed plastic bottles. After these eight days, the mixture is filtered. The OH⁻ ions contained in the filtrate are then measured with a dilute solution of hydrochloric acid at 10⁻¹ mol.L⁻¹ using methyl orange as a colored indicator. Then, the calcium ions (Ca²⁺⁾ will in turn be measured by X-ray fluorescence (XRF). The results obtained are illustrated by a graph giving the concentration of Ca²⁺ ions expressed in mmol.L⁻¹, equivalent to the quantity of CaO as a function of the concentration of OH⁻ ions also expressed in mmol.L⁻¹. Additionally, from the lime solubility isotherm, the pozzolanicity of the material is determined. Indeed, any material whose coordinates ([OH-], [CaO]) are located below the solubility isotherm of portlandite has pozzolanic activity and any material whose coordinates ([OH-], [CaO]) are located above the solubility isotherm of portlandite has no pozzolanic activity.

A simpler approach to studying pozzolanic reactivity is to use the lime saturation test. Its procedure consists of preparing a mixture consisting of 1 g of material and 75 mL of saturated lime solution initially prepared by dissolving 2 g of hydrated lime in 1 liter of distilled water. The hermetically sealed mixture is kept for the test period set at 1, 3, 7 and 28 days. The concentration of Ca²⁺ ions in the different mixtures is determined by X-ray fluorescence. Thus, from the quantity of calcium ions available in the initial mixture, the CaO content fixed by the material can be determined as a function of the period of the essay.

Pozzolanic activity index

The pozzolanic index constitutes the ratio of the mechanical compressive strength of the specimens containing pozzolan and those not containing it, called reference specimens. The value of the pozzolanic index is obtained from equation 9

$$I_P = \frac{R_P}{R_T} \times 100 \tag{9}$$

With, I_P : pozzolanic index ; R_P : mechanical compressive strength in MPa of mortars containing pozzolan; R_T : mechanical compressive strength in MPa of the reference mortar.

To do this, the mortar specimens were manufactured according to standard NF-P-15-403 [33]. First the cement, the pozzolan ground to 80 μ m and distilled water are introduced into a mixer to mix for one minute at a speed of 140 rpm. Afterwards, standardized sand is added and the mixture is mixed again for 3 minutes at a speed of 280 rpm. Then a manual scraping of the walls of the mixer tank was carried out. Finally, the cycle ends with a three-minute mixing session at 280 rpm. The kneaded mixture is introduced into 40x40x160 mm³ prismatic molds and compacted mechanically. Once leveled, the molds containing the samples are covered with plastic film and stored in the cold room at a temperature of 20±1 °C. Unmolding is carried out after 24 hours and the specimens are kept in the laboratory at 20±1 °C in a vase containing distilled water until the day of the test set at 7, 28 days and 90 days. Then, the mechanical compressive strength is determined using a CONTROLAB S type press according to standard NF P 15-471 [34]

Mineralogical reactivity

During pozzolanic reactivity, hydrates similar to those of cement are formed. Thus, monitoring the formation of these hydrates makes it possible to ensure the pozzolanic reactivity of the different materials. To do this, specimens consisting of lime, pozzolan and water were developed according to mass ratios: pozzolan/lime = 1 and water/(pozzolan + lime) = 0.5. After maturing for 28 days, the different composites were subject to mineralogical characterization by infrared spectrometry.

ACKNOWLEDGMENTS

The authors thank Pr. Jean Emmanuel Aubert (Laboratoire Matériaux et Durabilité des Constructions (LMDC), Université de Toulouse, INSA/UPS Génie Civil, 135 Avenue de Rangueil, 31077 Toulouse cedex 04 France de Toulouse) for providing the XRD analysis of the samples.

REFERENCES

- 1. F. Ganon; A. Yameogo; B. Sorgho; L. Zerbo; M. Seynou; Y. Millogo; R. Ouedraogo; *ChIBA.* **2015**, *16*, 371–383.
- 2. T. Halmagyi, E. Mosonyi, J. Fazakas, Studia UBB Chemia, LXIII, 1, 2018 (p. 73-86).

- K.L. Scrivener; V.M. John; E.M. Gartner; Cement and Concrete Research. 2018, 114, 2–26.
- M.Z. Al-mulali; H. Awang; H.P.S. Abdul Khalil; Z.S. Aljoumaily: Cement and Concrete Composites. 2015, 55, 129–138.
- 5. A.P. Fantilli; D. Jóźwiak-Niedźwiedzka; *Materials*. 2021, 14, 2291.
- 6. O. Chaib; M. Mouli; M. Hanifi; M. Hamadache; *J. Mater. Environ. Sci.* **2016**,7, 422–428.
- 7. R. Harbi; R. Derabla; Z. Nafa; Construction and Building Materials. 2017, 152, 632–641.
- 8. M. Kamali; A. Ghahremaninezhad; *Construction and Building Materials*. **2015**, *98*, 407–416.
- M. Seynou; Y. Millogo; L. Zerbo; I. Sanou; F. Ganon; R. Ouedraogo; K. Kaboré; JMMCE. 2016, 04, 195–209.
- 10.Y. Wang; Z. Shui; Y. Huang; T. Sun; R. Yu; G. Wang; *Construction and Building Materials*. **2018**, *172*, 19–28.
- 11. K.A. Melo; A.M.P. Carneiro; Construction and Building Materials. 2010, 24, 1529–1535.
- 12. F. Cassagnabère; G. Escadeillas; M. Mouret; *Construction and Building Materials*. **2009**, *23*, 775–784.
- 13. K. Weise; N. Ukrainczyk; E. Koenders; *Materials & Design.* 2023, 231, 112062.
- 14. F. Sinngu; S.O. Ekolu; A. Naghizadeh; H.A. Quainoo; *Developments in the Built Environment*. **2023**, *14*, 100154.
- 15. Y. Wang; Y. Li; Y. Su; X. He; B. Strnadel; *Advanced Powder Technology*. **2022**, 33, 103690.
- 16.S. Donatello; M. Tyrer; C.R. Cheeseman; *Cement and Concrete Composites.* **2010**, 32, 121–127.
- 17.M. C. Sekhar, M. H. Kumar, S. L. Raju, Materials Today: Proceedings. 2023, 03, 713
- I. Sanou; M. Ouedraogo; H. Bamogo; N. Meité; M. Seynou; J.-E. Aubert; Y. Millogo; *Emergent Mater.* 2024, 7, 1203–1217.
- 19. S.O. Sore; A. Messan; E. Prud'homme; G. Escadeillas; F. Tsobnang; *Construction and Building Materials*. **2018**, *165*, 333–345.
- 20. G. Jozanikohan; M.N. Abarghooei; *J Petrol Explor Prod Technol.* **2022**, *12*, 2093–2106.
- Niculăescu, L. Olar, R. Stefan, M. Todica, C-V. Pop, Studia UBB Chemia, LXIII, 2, 2018 (p. 63-70)
- 22. I. Sanou; M. Sawadogo; M. Seynou; L. Zerbo; R. Ouedraogo; *JMMCE*. **2019**, *07*, 373–384.
- ASTM; ASTM-C618-08: Standard Specification for coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete, West Conshohocken, PA : Annual Book of ASTM standards, 2008
- M. Ouedraogo; M. Sawadogo; I. Sanou; M. Barro; S. Nassio; M. Seynou; L. Zerbo; *Results in Materials.* 2022, 14, 100275.
- 25.K. Dao; M. Ouedraogo; Y. Millogo; J.-E. Aubert; M. Gomina; *Construction and Building Materials.* **2018**, *158*, 84–96.

- Y. Millogo; J.-C. Morel; K. Traoré; R. Ouedraogo; Construction and Building Materials. 2012, 26, 663–669.
- 27. G. Dal Poggetto; P. Kittisayarm; S. Pintasiri; P. Chiyasak; C. Leonelli; D. Chaysuwan; *Polymers.* **2022**, 14, 5091.
- 28. Y. Millogo; M. Hajjaji; R. Ouedraogo; *Construction and Building Materials*. **2008**, 22, 2386–2392.
- 29.S.S. Ibrahim; A.A. Hagrass; T.R. Boulos; S.I. Youssef; F.I. El-Hossiny; M.R. Moharam; *JMMCE*. **2018**, *06*, 86–104.
- 30. I. Sanou; M. Seynou; L. Zerbo; R. Ouedraogo; SJC. 2019, 7, 1.
- N. Méité; L.K. Konan; M.T. Tognonvi; B.I.H.G. Doubi; M. Gomina; S. Oyetola; Carbohydrate Polymers. 2021, 254, 117322.
- M. Sawadogo; I. Sanou; Y. Dah; B. Traoré; Y. Sawadogo; D. Samaké; C. Dembelé; L. Zerbo; M. Seynou; *Journal de la Société Ouest-Africaine de Chimie*. 2021, 050, 50–56.
- 33. AFNOR; NF P15-403 : Sable normal et mortier normal, 1996.
- 34. AFNOR; NF P15-471 Méthodes d'essais des ciments: Détermination des résistances mécaniques, **1990**.