

EFFECT OF SPECIAL CEMENT CPA ON THE CHARACTERISTIC PROPERTIES OF CEMENTITIOUS MATERIALS BASED WASTE GLASS POWDER: POZZOLANIC ACTIVITY AND HEAT TREATMENT

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KEY WORDS: Glass powder (GP), Special cement CPA, Pozzolanic Activity Index (I), Heat treatment, Sustainability, Compressive strength

ABSTRACT:

The main purpose of this study is to evaluate the effect of curing method on the compressive strength of waste glass powder as a supplementary cementitious material. This work presents an experimental study on the physico-mechanical characterization of waste glass powder (GP) as partial replacement of special cement (Algerian cement without additions CPA) based high performance cementitious material, varying the percentage of GP by 10%, 20% and 30% (by weight of cement), the curing methods: water curing at 20 ± 2 °C and heat curing by under accelerated drying in an oven at 100 °C (stoving). Half of the mortar samples 40x40x160 mm were treated with stoving just after demolding then kept in the open air, the other half was kept in fresh water in order to evaluate their sustainability and index of Pozzolanic Activity (I) at different ages: 7, 28, 90 and 365 days. The compressive strength results showed that there is an increase in compressive strength with the increase in age of the two curing methods but the strength of all mixtures which have been stoved is inferior to those of the same mixtures preserved in fresh water at different ages. The best rate of replacement of the cement by GP is 20% following the results obtained for the compressive strength and Pozzolanic Activity Index I.

1. INTRODUCTION

The needs of the modern world require to produce a building material faster, more durable and economic, requiring a long enough time to acquire its optimal capacities, we sought in this work accelerated its setting and hardening so that it becomes compatible with the requirements of industrial production. These objectives can be obtained by the heat treatment (stoving) on the one hand and on the other hand by minimizing the cost of the cement and its harmful impact on the environment by exploiting a local waste such as glass.

Million tons of waste glass is being generated annually all over the world. Once the glass becomes a waste it is disposed as landfills, there is an impetus to use industrial by-products/waste material in construction industry to achieve sustainability in this sector. One ton cement manufacturing results in emission of 0.9 ton of carbon dioxide (CO₂) to the atmosphere (Imbabi et al., 2012) Cement production also involves emission of moderate quantities of NO, SO, and particulates which is unsustainable for environment as this is not bio-degradable. Glass is principally composed of silica, use of milled (ground) waste glass in concrete as partial replacement of cement could be an important step toward development of sustainable environmentally friendly, energy-efficient and economical infrastructure systems (Prusty et al., 2016).

When waste glass is milled down to micro size particles, it is expected to pozzolanic reactions with cement hydrates in pastes (Saeed and Ebead 2013) and concrete (Shi et al., 2005) forming

secondary Calcium Silicate Hydrate (C-S-H). The use of bottle glass powder by replacing sand (Kiang and DU 2013), granulate (Limbachiya et al., 2012) or cement in mortar (Walczaka et al., 2015) and concrete have a promising way to recover and recycle waste glass powder (GP) is generally considered to be pozzolanic according to the works of (Idir et al., 2011, 2010) since it contains quantities of silica. This valorization of GP in concrete has several advantages: economic, ecological and technological (Saribiyik et al., 2013). Indeed, the glass replaces a fraction of the cement that enters in the composition of the concrete; it is therefore used as an alternative cementitious addition like others fines like calcinated bentonite (Mesboua et al., 2021) and brick compared to GP effect studied by (Bouleghebar et al., 2023). The recycling of glass in concrete makes it possible to contribute to the sustainable development of this indispensable building material. In addition, the GP is used as it is, without any change in its chemical composition and without the addition of an additive according to (Bhandari and Tajne, 2013), thus for a better results exploitation the macro and micro structure of this cementitious material was carried out by (Matos and Sousa, 2016).

Thousands hours of research have resulted in the development of a technology that allows the use of crushed or glass powder to make stronger concrete (Sadiqul et al., 2017, Bekir et al., 2004), with low permeability and more Sustainable (Krati and Kansal, 2016, Boucheneb et al., 2023), this shows that investing in research, development, and participating in a greener economy. Sadiqul (Sadiqul et al., 2017) showed that in general, considering the similar performance with replaced material,

glass addition can reduce cost of cement production up to 14%. In addition, production of every six ton glass powder concrete results in the reduction of each ton CO₂ emission from cement production and save the environment significantly by reducing green-house gas and particulate production. Generally, the high surface area of milled waste glass changes the kinetics of chemical reaction toward beneficial pozzolanic reaction utilizing the available alkalis.

The Effects of glass powder on the characteristics of concrete subjected to high temperatures has been studied by Belouadah (Belouadah et al., 2018), the concrete is exposure to various temperatures (20, 200, 400, and 600°C) by working with 5% and 10% of glass powder and it conclusion is high curing temperature is beneficial to the compressive strength development for cement pastes incorporating GP because it can promote both the hydration of cement and the pozzolanic reaction of GP simultaneously.

CEM I is the Portland cement composed of 95% or 100% clinker, they are grayish enough, these cements are used in structures of civil engineering (bridges, large slender structures) or prefabrication; they allow a resistance to the young age which allows a quick stripping.

The choice of special cement without additions CEM I will allow us on one side to avoid the interaction between the additives used in the manufacture of other industrial cements and our main substitute the glass powder, on the other side to study the effect of this glass powder on the compression performance of high performance mortars.

2. EXPERIMENTAL STUDY

2.1 Materials

2.1.1 Cement: The special cement CPA used for the manufacture of the test pieces is a cement CEM I 52.5 without any addition (95% clinker + 5% gypsum), produced by the Sour El Ghouzlane cement industrial group (GICA) located in the wilaya of Bouira (Algeria) and conforms to the norm NF EN 197-1(NF EN 197-1 2000) and ASTM C150 (ASTM 150 2009) whose clinker is produced and crushed together with gypsum. Its initial setting time is 2h08 min and final setting time is estimated to 5Hh13 min. For the mechanical characteristics, a compressive strength is 56 MPa at 28 days. The chemical composition by using the X ray fluorescence technique (XRF), physical characteristics and the constituents according to the Bogue formula of this cement are given in Tables 1, 2 and 3.

Characteristics	CEM I 52.5
Normal consistency (%)	25.8
Initial setting time	2h50 min
Final setting time	5h13 min
Specific surface	2530 cm ² /g
Specific gravity	3470 kg/m ³

Table 1. Physical characteristics of cement

Oxides(% by mass)	CEMI 52.5	Glass Powder
SiO ₂	21.29	70.50
Al ₂ O ₃	4.72	1.42
Fe ₂ O ₃	3.49	0.10
CaO	64.68	8.43
MgO	0.44	4.01
Na ₂ O	0.17	15.28
SO ₃	2.06	0.25
Loss on ignition	1.930	0.22

Table 2. Chemical characteristics of cement and glass powder

Constituents	C ₃ S	C ₂ S	C ₃ A	C ₄ AF	Gypsum
Percentage (%)	60.61	15.36	6.6	10.61	5

Table 3. Mineralogical composition of CEM I 52.5

2.1.2. Glass powder: This powder comes from the recovery of glass green bottles in roadside dumps and scattered in nature. The glass powder is obtained by selecting only the green and white bottles. These are cleaned to remove the paper labels, dried, crushed and finely ground (see Figure 1), with a specific surface area of 3600 cm²/g. The valorization of bottles glass in concrete and mortar could constitute an interesting alternative (Bekir et al., 2004), from the environmental point view, avoiding its landfill.

However, from a technical point view, bottle glasses contain significant quantities of alkalis which may cause detrimental effects on the concrete in relation to the alkali-silica or alkali-aggregate reactions (Khmiri et al., 2012). Many authors agree that glass results in beneficial behavior associated with the pozzolanic reaction (Idir et al., 2011, Matos and Sousa 2016) if used in the fine powder form. The chemical composition and particle size curve analysis of the glass powder are shown in Table 2 and Figure 2 respectively.

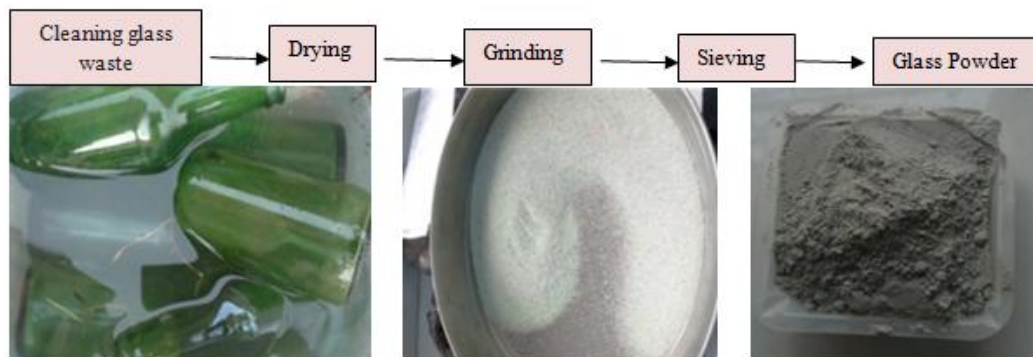


Figure 1. The glass powder obtained after grinding the waste glass

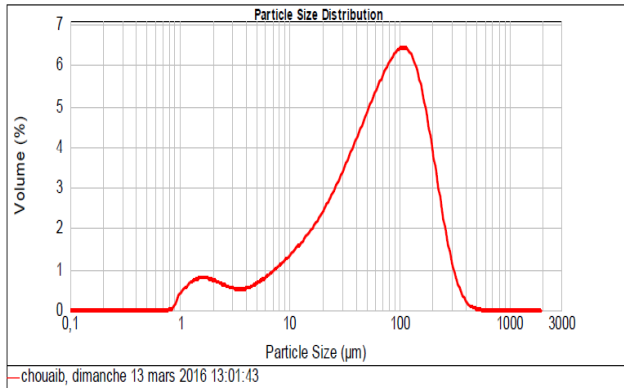


Figure 2. Particle size curve of Glass Powder

10%, 20% and 30%).

After demoulding, the half prismatic samples (4x4x16 cm) were cured in water at 20 ± 2 °C until the age of test (7, 28, 90 and 365 days), and dried in the open area inside the laboratory 24 hours before the test.

In order to accelerate hardened of mortar the second half cubic specimens are treated by stoving to accelerate the curing process, 15 minutes after their molding, they have undergone heat treatment as shown in the Figure 3, after cooling they are broken by compression at 2 days (after a hardening day), 28, 90 and 365 days.

2.2 Mix design and conservation of studied mortars

In order to follow the mechanical behavior of the mixtures, we considered the different compositions illustrated in Table 4, the factors to be varied are the percentage of glass powder (0%,

Constituents (g)	W/B [*] =0.35 (B: Binder = cement +glass powder)			
% of Glass Powder	0%	10%	20%	30%
Mixtures	M0GP	M10GP	M20GP	M30GP
Cement	450	405	360	315
Glass Powder	0	45	90	135
Sand	1350	1350	1350	1350
Water	157.5	157.5	157.5	157.5
Superplasticizer	9	9	9	9

Table 4. Composition detail of mixtures

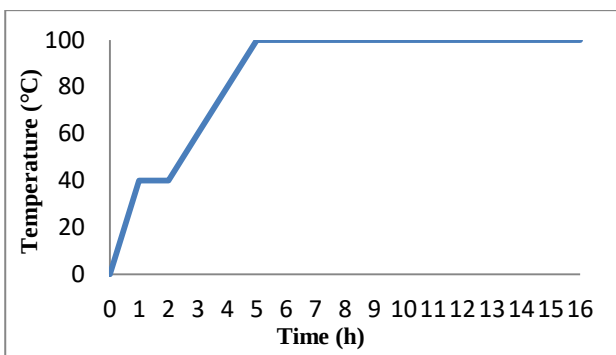


Figure 3. The used furnace temperature function in present study

= 0 minute the contact water-binder, the tests on the grouts are carried out after the mixing, the mini slump with the mini cone at $t = 5$ min.

The Fluidity test by mini-cone test useful for determining the compatibility of cement and superplasticizer called the saturation point which is evaluated as a percentage of dry extract in relation to the total mass of cement, which was determined in the laboratory its value was 2% of the mass of cement, but for confirmation purposes we checked this result thanks to the mini slump test showed in Figure 4 (b) and (c) while maintaining the value of $W / C = 0.35$ for cementitious grouts without glass powder, this saturation point value will be used later for the preparation of the various mortars.

2.3 Test methods

2.3.1 Fresh properties: The characterization of the fresh-state behavior of empirically tested cement pastes with and without glass powder allows the fluidity of the pasta to be examined by measuring the mini-slump and the initial and final setting time tests, measured according to standard NF EN 196-1 (NF EN 196-1 1995) for different percentage of glass powder (0%, 10%, 20%, 30%) and studied with the VICAT apparatus (Figure 4 (a)). In order to ensure uniformity of results, the temperature of all mixtures is maintained at 20 ± 2 °C after mixing; considering t

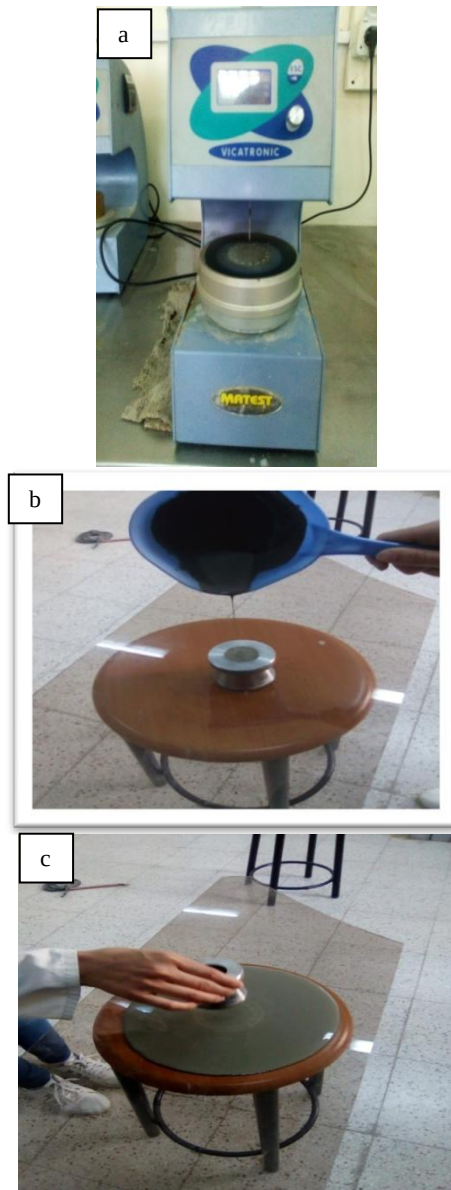


Figure 4. Fluidity test by mini-cone

The test was carried out by mixing 250g of CEM I 52.5 cement with water ($W / C = 0.35$) with an amount of adjuvant knowing that the latter changes starting with a percentage of 1.2% until the point saturation is reached. After having poured the mixture cement + water + adjuvant in the mini cone, a circle of paste is formed on the plate in plexiglass, after one measures the two diameters on circle to take their average such as:

$$D = (D1 + D2) / 2$$

Saturation point in SP of our CEM I 52.5 cement is the maximum uniform spreading diameter which is 2% compatible with the value obtained in the laboratory; the result is shown in Figure 5.

Beyond a spread of 203 mm for a dosage of 2%, the mixture is unstable, the end of the spreading wafers is not uniform, presence of bleed and sedimentation of fine particles. SP supersaturation occurs, which induces a highly unstructured mixture and therefore not viscous enough to keep the fine particles in suspension.

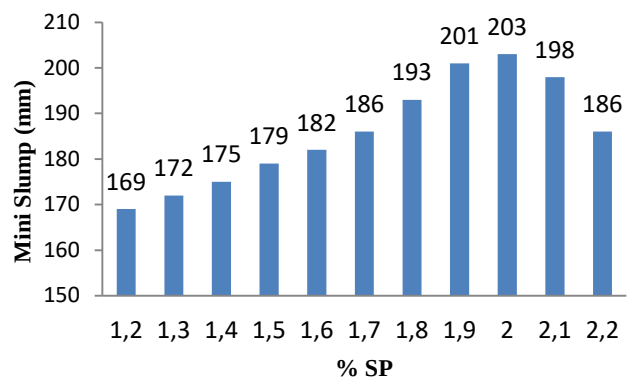
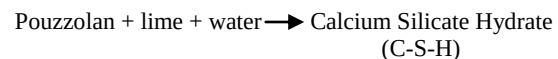


Figure 5. Slump of studied blends to determinate the point saturation on superplasticiser

2.3.2. Hardened properties: The mechanical (uniaxial compression) properties of studied mixtures were determined. Three-point bending and uniaxial compression tests are carried out at 7, 14, 28 and 365 days by using a hydraulic press with a capacity of 3,000 KN according to ASTM C349 (ASTM C349 2002)

2.3.3. Index of Poozolanic Activity: Most poozolanic materials contain a form of reactive vitreous silica which, in the presence of water can combine with the temperature liberated by the hydration of C_2S and C_3S , with lime to form a hydrated calcium silicate (C-S-H) similar to the one formed during the hydration of portland cement, which can increase the compressive strength of mortars and concretes, we can write a pozzolanic reaction in a simple way:



According to ASTM C618 (ASTM C618 2008), a material is considered poozolanic if:

- Its verified chemical composition: $Al_2O_3 + SiO_2 + Fe_2O_3 > 70\%$.
- Its activity index I is: $0.67 < I < 1$.

The activity index is a parameter that quantitatively describes the degree of reaction over time. Or the rate of reaction between a poozolanic material and $Ca(OH)_2$ in the presence of water. The index activity is determined through mechanical compression tests on mortar specimens of $40 \times 40 \times 160 \text{ mm}^3$. It is given by the ratio between the compressive strength of a mortar with % addition of clinker and the compressive strength of a control mortar with 100% of cement. The activity Index I is a way to evaluate the poozolanic reactivity of glass in mortars at the different ages.

$$I = (R_x / R_c) 100$$

where: R_x and R_c are the compressive strengths of mortars containing GP and the control mortar, respectively.

3. RESULTS

3.1 Fluidity and setting time

The fluidity of the fresh pasta were determined by the mini cone test with a constant amount of water for all preparations (W / B

= 0.35) or B represents in this case the sum of cement and glass powder, the values of the results are given in Figure 6. Fluidity of the pasta increases with the increase of the substitution rate of the cement CEM I 52.5 by the glass powder. This increase in the mini slump relative to the pasta with glass powder is reflected by the variation of the specific surface of the pasta thanks to the great fineness of the glass powder compared to the cements and therefore a decrease in demand for water.

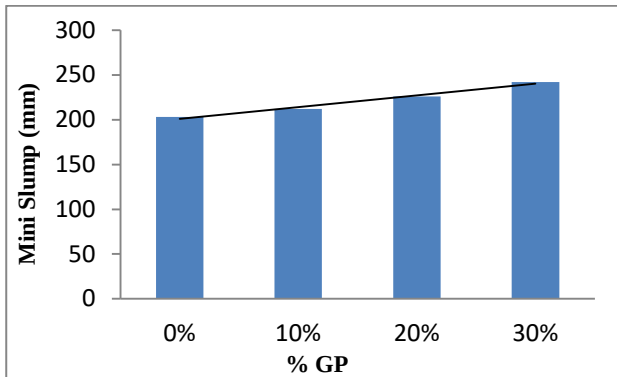


Figure 6. Slump according to the % of glass powder

It is clearly seen in Figure 7 that cement pastes with different levels of glass powder need more water to obtain a normal consistency, moreover, more the percentage of glass powder is large and the greater the water demand. This increase in the normal consistency of the pastes is probably due to the progressive increase in the specific surface area of the binder, which naturally leads to a strong appeal to the water molecules in order to wet the whole paste. It is therefore deduced that the incorporation of the glass powder in the mixtures modifies the need for water, influences the process of hydration of the cement and increases the initial and final setting times in proportion to the percentages of the glass powder.

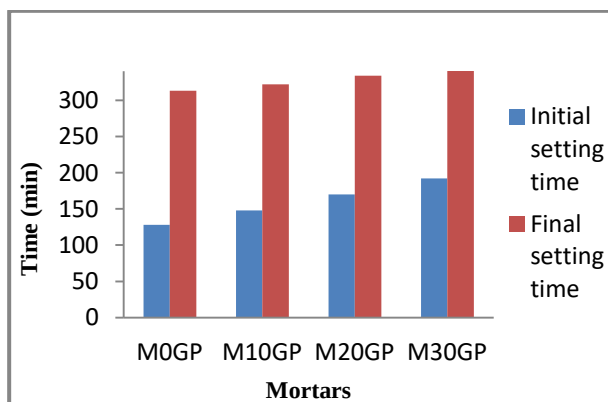


Figure 7. Initial and Final setting time according to % of GP with VICAT apparatus (a)

3.2 Evaluation of Poazolanic Activity Index

From the chemical characterization of the glass powder illustrated in Table 2, it is found that the sum of the three components: alumina Al_2O_3 , silica oxide SiO_2 and iron oxide Fe_2O_3 is about 74.2% which exceeds the limit value required by ASTM C618 (ASTM C618 2008) the glass powder would be a pozzolanic material if it has a minimum of 67% as an Activity Index at 28 days. Figure 8 shows the evolution over time of the

activity index obtained with the three cement mass substitution rates by the glass powder (10%, 20%, and 30%).

We obtain at 28 days of age an Activity Index higher than the normative limit (67%) for the three percentages in glass powder studied, we can conclude that the glass powder has pozzolanic reactivity. We also observe an increase of this Index of Activity over time for the three mortars; it goes to a maximum obtained at 365 days, which is 112.7% for the M20GP mortar. This expected evolution is attributed to the development of the pozzolanic reaction.

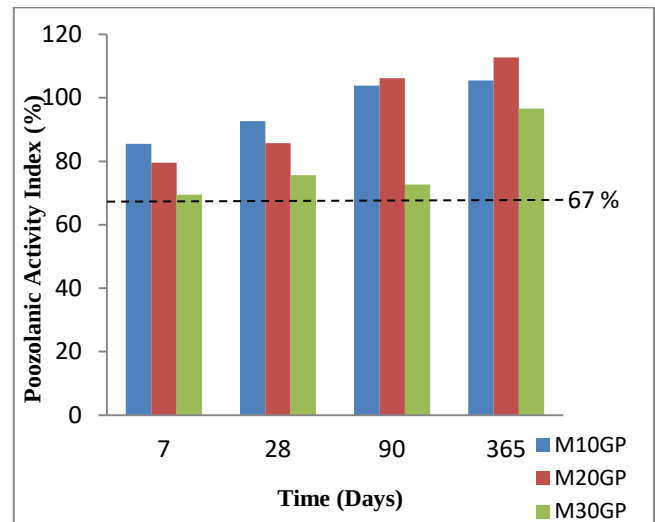


Figure 8. Poazolanic Activity Index with different percentage of glass powder at all ages

3.3 Compressive strength of mortars cured in fresh water

The evolution of the mechanical behavior in compression is followed as a function of time for the different types of mortar studied with CEM I 52.5 cured in fresh water; the results for the specimens are shown by Figure 9.

The results show that the compressive strength propagates as a function of time and deteriorates with the increase in the percentage of the glass powder at 7 and 28 days compared to the control mortar M0GP. Intile 28 days, the compressive strength of mortars with glass powder increased (90 and 365 days) to even exceed the strength of our control mortar M0GP, which is explained by the pozzolanic reaction (Idir et al., 2011) of the glass powder which manifests at long term. With further progression of reaction at the age of 365 days, recycled glass mortars with 10 and 20% GP substitution (M10GP and M20GP) provided mean compressive strengths exceeding the control mortar M0GP and 20% cement replacement gave the highest value among them (89.2MPa). Except 30% glass addition, 365 day test results indicated that the differences in compressive strengths between recycled glass M30GP and control mortars M0GP were little significant (3MPa). This indicates the optimum reaction period of glass replaced mortar (Nassar and Soroushian, 2011). At this stage 10 and 20% cement substituted by glass powder gave higher compressive strength than the control mortar 90 and 365 days.

From the compressive strength test results in mortar at 365 days, the optimum glass powder content was found to be 20% (M20GP) for which compressive strength was achieved 83.2 MPa higher than the control mortar by working with our CEM I

class 52.5, but the work of show that the compressive strength of his mortar has reached approximately the 37 MPa at 90 days for 20 % GP and by working with the CPA class 42.5, at the same time replacing 20% cement will reduce production and release of 18% CO₂ in the environment (Sadiqul et al., 2017).

3.4 Effect of hardening acceleration (heat treatment) on compressive strength

The results obtained for the effect of heat treatment are shown in the figure 10. The rise in temperature is beneficial for improving strength at an early age (Shi et al., 2005), which is valid for mortars M10GP and M20GP as shown in the figure 10

and not valid for the control mortar M0GP which are based on CEM I cement.

We also note that we have the same results in terms of behavior of the glass powder; we observe lower strengths at a young age for mortars based on glass powder by comparing with M0GP but this strength increase for exceed that of the control mortar at 365 days except for the M30GP mortar, which remains below M0GP. Stoving probably has to accelerate the hardening, but comparing to the results of the figure 9 we note that the results has considerably affected the phenomenon of development of strength of CEM I cements with glass powder or noting that the compressive strength of all mortars which have been steamed are inferior to those of the same mortars preserved with fresh water at different ages.

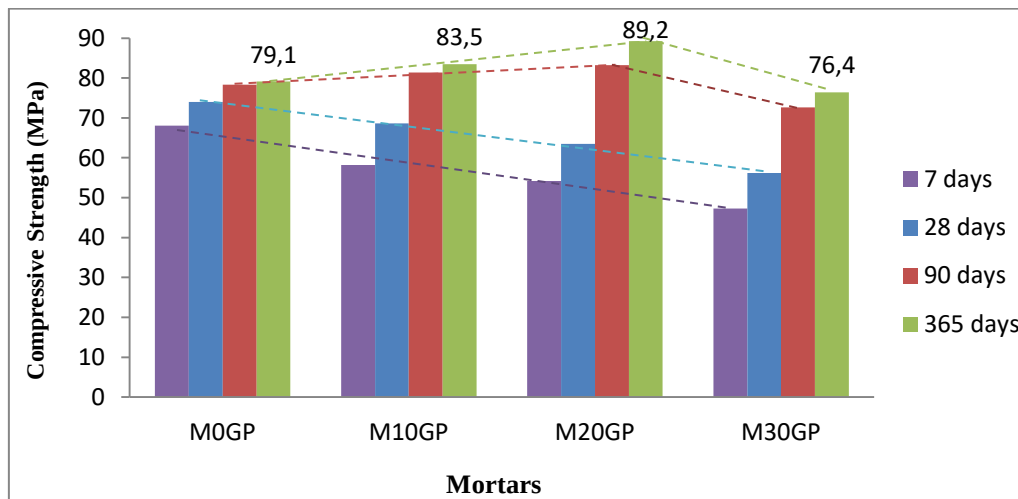


Figure 9. Influence of the addition dosage on the compressive strength at different ages

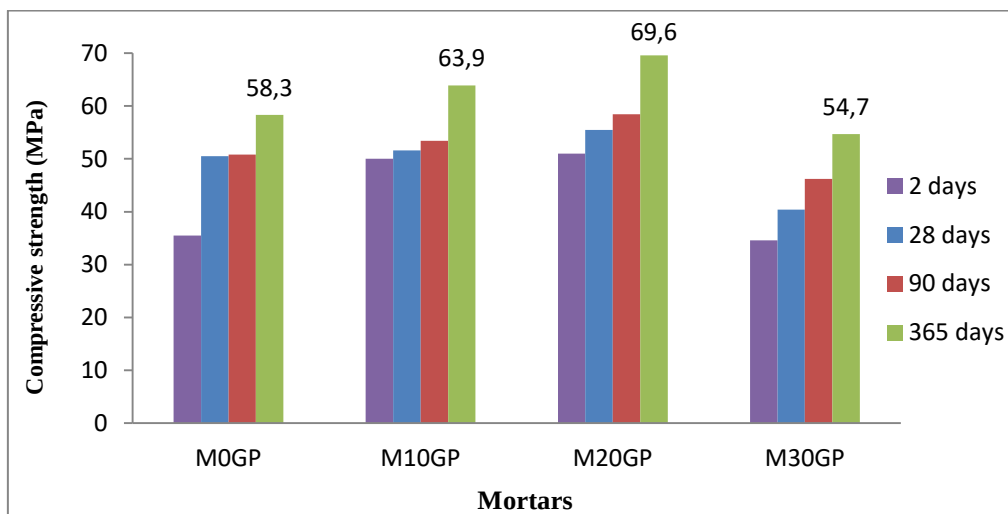


Figure 10. Influence of heat treatment on the compressive strength at different ages for different mixtures

4. CONCLUSION

The paper discusses the influence of glass powder on the mechanical properties of cement CPA without additions while varying the percentage of glass powder by 0, 10, 20 and 30%.

→ The setting times are influenced by the substitution of Glass Powder, they increase slightly with the increase in the

addition of this glass powder, so the use of glass powder usually reduces the demand for water. The workability increases with the increase of the substitution rate of the cements CEM I by the glass powder; it is reflected by the variation of the specific surface of the pasta thanks to the great fineness of the glass powder (3600 cm²/g) compared to the cements and therefore a decrease in demand for water.

- The results obtained show that an addition of 10% and 20% of Glass Powder improves the mechanical compressive strength of the mortars studied, the compressive strength is considerably deferred, a decrease in the strength as a function of the percentage increase in Glass Powder and a shelf life increase for CEM I which shows that the glass powder exerts a physical and chemical effect (pozzolanic and alkali-silica reaction). The control mortar M0GP at 28 days appears more strength than M10GP, M20GP and M30GP, this may be to the substitution of a percentage of cement by the Glass powder, which affects the hydration and thus the strength; but this strength increases beyond 28 days to reach values which may even exceed that of the mortar M0GP (365 days). Stoving affected the strength of mortars based CEM I cement with glass powder, the strength of all mortars which have been stoved are inferior to those of the same mortars preserved with fresh water at different ages.
- The mineralogical characterization of the glass powder shows that the sum of $Al_2O_3 + SiO_2 + Fe_2O_3 = 74.2\% > 70\%$, the evaluation of the pozzolanic activity index of the glass powder allowed us to conclude that this glass powder is a pozzolanic material. In fact, we obtain at 28 day pozzolanic activity indices $> 67\%$.
- The best rate of replacement of the cement with glass powder is 20% following the results obtained for the compressive strength of the mortar M20GP as well as those of index of activity, nor at least one can neglect the result obtained with 30 % replacement of cement with glass powder, which is about 3 MPa less than M0GP, but it has a gain in cement which is favorable to the performance of companies producing cement products.
- However, the analysis of the results in terms of strength shows that there are products formed during the reaction between glass and lime which would probably be hydrated calcium silicates (HSC) and which develops in the long term (365 days) something that can be confirmed by DRX and ATG analysis tests that we will take care to complete in our future work to better understand the results of analysis of the microstructure.

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List of notation

C	is the cement
CEM I	is the cement without additions (95% clincker + 5% gypsum)
GP	is the glass powder
B	is the binder; noted $B=C+GP$
W	is the water
$W/C= 0.35$	is used to determinate the point saturation on superplasticiser for cement CEM I
$W/B= 0.35$	is used to produce high performance mortar