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Effects of glass powder on the characteristics of concrete subjected to high temperatures

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Abstract. This paper presents an experimental investigation on the performance of concrete with and without glass powder (GP) subjected to elevated temperatures. Mechanical and physicochemical properties of concretes were studied at both ambient and high temperatures. One of the major environmental concerns is disposal or recycling of the waste materials. However, a high volume of the industrial production has generated a considerable amount of waste materials which have a number of adverse impacts on the environment. Further, use of glass or by-products in concrete production has advantages for improving some or all of the concrete properties. The economic incentives and environmental benefits in terms of reduced carbon footprint are also the reason for using wastes in concrete. The occurrence of spalling, compressive strength, mass loss, chemical composition, crystalline phase, and thermal analysis of CPG before and after exposure to various temperatures (20, 200, 400, and 600℃) were comprehensively investigated. The results indicated that, the critical temperature range of CPG was between 400℃ and 600℃.

Keywords: powder glass; concrete; high temperature, spalling, mass loss

1. Introduction

Concrete is the most important substitute in building material. Strength, cost and durability of building is highly depending upon it (Gautam *et al*. 2014). But concrete is generally considered to have an acceptable resistance to fire in comparison with other construction materials such as wood or steel. When concrete remains exposed for long time to high temperatures, mechanical losses of its properties take place (Sangluaia *et al*. 2013).

Laboratory experiences show that in case of concrete not protected the mechanical properties decrease drastically for temperatures above 300℃. They are attributed to the microstructure transformations occurring in cement paste and aggregates, and the volume changes induced by thermal stresses. After fire the assessment of deterioration of the structure is needed in order to identify the level of damage induced by the chemical transformation and the cracking, both

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contributing to losses in mechanical strength (Kulkarni 2014).

Recent examples of fires happened in the Channel and Mont Blanc tunnels have revealed gaps in the understanding of the phenomena of chipping and bursting of concrete thus motivating the scientific community to investigate further the behavior of concrete at high temperature. The first difficulty of the study lies in the complexity of the structure of the cement binder material and its highly heterogeneous nature. With mixing proportions of aggregates with cement and water have a major impact on mechanical properties of concrete with temperature because, usually important chemical and physical changes occur, with loss of cohesion, that leads to its degradation (Khoury 2000).

When concrete is under fire, it usually causes a build-up of pressure within it after exceeding 1000℃. When the temperature reaches about 400℃, the calcium hydroxide in the cement will begin to dehydrate, generating more water vapor and also bringing about a significant reduction in the physical strength of the material. The material behavior during heating is nonlinear itself according to its deterioration with temperature (Bazant and Kaplan 1996, Sangluaia *et al.* 2013).

It is important to note that concrete properties and their variation as a function of the various influencing factors, such as loading, temperature and moisture, depend on the properties of the concrete constituents. It can be stated that concrete microstructure derives its macro-properties through the combination of the properties of the aggregates and the cement paste. The large number of different aggregate types can be used within concrete at ambient temperature. However, under fire conditions, when concrete is heated, many changes occur in physical structure, chemical composition and fluid content. Therefore, the mechanical properties of concrete, in particular strength and stiffness, are significantly altered when exposed to high temperature (Burlion *et al.* 2003).

Tebbal *et al.* (2017) determined the combined effect of silica fume and additive on the behavior of high performance concretes subjected to high temperatures. The tested concretes are formulated with 5% silica fume and two dosages of super plasticizers in the ratio of (2%, 2.5%) the weight of cement after having been exposed to four maximum temperatures, 200℃, 400℃, 600℃ and 900℃ without any imposed load during the heating. The results obtained show that the mechanical resistance at 28 days increases with the degree of temperature compared to that measured at 20℃. On the contrary, a clear decrease is observed between 600℃ and 900℃. However, material composition seems to have great influence on the mechanical strength.

Shayan and Xu (2004) concluded that waste glass has a potential of being used as aggregate and pozzolan in concrete that could potentially replace traditional pozzolans such as fly ash and silica fume in concrete. This research explores the synergistic use of two waste materials, i.e., waste glass and demolished concrete (as recycled aggregate) and emphasizes the novel concept of using milled waste glass to overcome the limitations of recycled aggregate and consequently recycled aggregate concrete. When milled waste glass is used in recycled aggregate concrete as partial replacement of cement, it interacts with calcium hydroxide available in the attached mortar/paste clinging to aggregate surface to form calcium silicate hydrate (C-S-H) which is the key binder among cement hydrates. This reaction can enhance the quality of the remnant cement paste on recycled aggregates, thus benefiting the strength, durability and dimensional stability of recycled aggregate concrete.

In 2014, Singh *et al*. carried out a critical study of effectiveness of waste glass powder in concrete. This work examines the effect of using waste glass powder as a cement and sand replacement material into concrete. Glass constitutes about 5% of the municipal solid waste stream, but only a small percentage of it is recycled. Therefore, tapping its potential as a cement replacement material is imperative. Waste glass contains about 72.5% S_iO_2, when it is ground to the fineness of around 600 μm; S_iO_2 in it reacts with alkalis in cement to form cementitious products. Such products help contribute to strength and durability in concrete. Glass powder was partially replaced as 5%, 10%, 15%, 20%, 25% with sand and tested for its compressive strength, slump, workability and alkalis test and compared with those of conventional concrete. From the result obtained it is found that replacement of 20% glass can be more beneficial and capable to increased strength by 45%. Recently, the research mainly focused on the effect of GP's (glass powder) fineness on its pozzolanic activity while the effect of heat treatment on its pozzolanic activity and the mechanism of GP in cement-based materials under different curing temperatures were not clear. It is necessary to fully recognize the acting mechanisms of GP in cement-based materials and their influencing factors to guarantee its optimum utilization (Liu *et al.* 2015).

Recent researches have shown that concrete made with recycled glass aggregate have shown better long term strength and better thermal property of the glass aggregate. When concrete contains waste glass powder it gives high percentage of C_3S, low C_3A, C_4A_4, C_3S/C_2S contents which result in production and offer greater resistance to the sulphate attack. Glass powder content SiO_2 when it reacts with alkalin in cement (pozzolanic reaction) to form cementation product such product helps contribute to strength and durability in concrete (Sorousshian Nassar 2012).

The influence of curing conditions on the high temperature performance of SCGC was investigated by Ling *et al*. In this research for each curing regime, five SCGC mixtures were prepared with recycled glass (RG) which was used to replace natural fine aggregate at the level of 0%, 25%, 50%, 75% and 100%. The test results indicate that regardless of the exposure temperature, all the water cured specimens had higher residual strengths and mass losses while the water porosity and water sorptivity values were lower as compared to the corresponding air cured specimens. The incorporation of RG in the concrete mixes helped to maintain the concrete properties after the high temperature exposure due to the melting and resolidification of the recycled glass in the concrete matrix (Ling *et al.* 2012).

In 2017 Oluwarotimi *et al.* carried out a study on the effect of elevated temperature on the strength of concrete containing glass powder (GWP) as ordinary Portland cement replacement. The cement was partially replaced by 0, 15, 18, 21, 24, 27 and 30% of GWP and samples were prepared at constant water-binder ratio of 0.5. The cube samples after curing in water for 90 days were exposed to 60, 150, 300 and 500℃ temperatures increased at a heating rate of 10℃/min. Compressive strength values were measured on the unheated samples and after air-cooling period of the heated samples. The results indicate a decrease in the compressive strength with increasing temperature, and significant alteration was observed in the concrete matrix and interface from the SEM analyses. However, the results indicate that concrete samples containing 21% GWP exhibit higher strength compared the control samples (Oluwarotimi *et al*. 2017).

Sarkawt *et al.* (2016) also found that the optimum percentage of glass waste content in SCC (Self Compacting Concrete) as coarse aggregate replacement is 25%. On the other hand, the increase of temperature leads to decrease of these strengths of SCC. In addition, the properties of fresh SCC study and results show that the workability of SCC decreases slightly with increasing glass waste content in the mixes (Sarkawt *et al.* 2016).

The main objective of the current study is to evaluate the effect of glass powder on the characteristics of concrete subjected to high temperature, XRD patterns, ATG, and ATD of concrete subjected to high temperature.

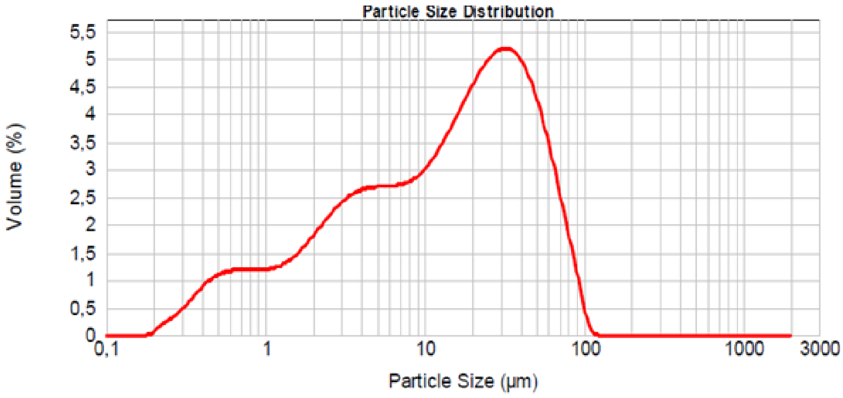


Fig. 1 Particle size distributions of cement

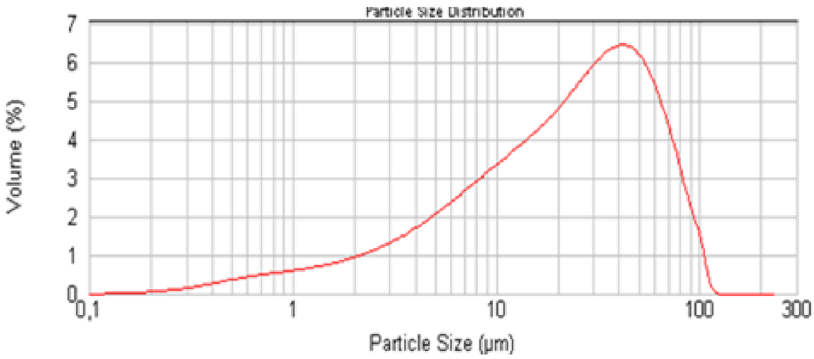


Fig. 2 Particle size distributions of Glass powder

2. Materials and methods

The Portland cement type CEM II/A 42.5 from Hammam Dalâa local factory was used in this experimental study. The chemical composition of the cement is shown in Table 1.

The glass powder is made of collected waste glass bottles, which are crushed and ground for 0.5 h in a ball mill after being cleaned and dried. Different from the spherical fly ash particles, GP particles show irregular singular, blocky, and classic shapes with smooth surface morphology. Most glass powder particles are smaller than 20 μm, which may contribute to activating its reaction activity and reducing the risk of alkali-silica reaction (ASR). The chemical compositions of GP and cement used are listed in Table 1.

The physical properties and particle size which are done by laser granulometer (Master-sizer 2000) of cement and glass powder are shown in Table 1 and Figs. 1-2.

The natural fine aggregates used were dune sand with particles ranging from 0.08 mm to 5 mm in size, with a fineness modulus, M_f of 2.44. This natural sand was taken from the region of Boussâada, (250 km east of Algiers). The sieve analysis is performed according to the European standard NF EN 933-1. After the treatment, process allows eliminating a significant portion of clay minerals impurities. The mineralogical composition determined by X-ray diffraction shows that the siliceous sand is more than 95% of quartz and calcite traces.

The coarse fraction of aggregate is gravel (G_1) of size 3/8 mm and gravel (G_2) of size 8/15 mm.

Table 1 The chemical and physical properties of cement and Glass powder

	Cement (%)	Glass powder (%)
SiO_2	18.51	72.84
Al_2O_3	04.88	0.98
Fe_2O_3	02.96	0.55
CaO	58.30	9.66
MgO	01.85	1.76
SO_3	2.39	0.25
CL	0.016	-
K_2O	-	0,43
Na_2O	-	12,69
P_2O_5	-	0,01
T_iO_2	-	0,04

Mineralogical composition of cement

	C_3S	C_2S	C_3A	C_4AF
Item Content	59	14	6	10

Physical properties of cement and glass powder

	Absolute density (kg/l)	Apparent density (kg/l)	Porosity (%)	Specific surface area (cm^2/g)	Pozzolanic activity
Cement	3.10	1.90	41.96	3410	
Glass powder	2.7	-	-	8530	28,5 g $Ca(OH)_2$/g (PV)

The tap water used all through the study from mixing was taken from the laboratory of civil engineering.

2.1 Mix design

The concrete mix design was proposed according to DREUX method for control concrete. The replacement level of sand to glass powder was used in the term of 0%, 5% and 10% in concrete.

Fresh concrete mixes were prepared in a modified laboratory mixer. The concrete specimens with dimensions 100 mm×100 mm×100 mm were preserved in their moulds in a wet place at a temperature of 20℃ and 95% relative humidity (RH) during 24 hours. After demoulding, they were immersed in water at 20℃ until the age of testing.

The physical and mechanical characteristics of the concretes with and without the addition of glass powder have been compared. The glass powder is added at dosages of 5% and 10% of cement weight respectively. The final compositions of concrete with addition, after optimization is reported in Table 2.

For all the concrete made, workability was measured by the Abrams cone slump test in accordance with NF EN 12350-2. The axial compressive strength was tested at 28 days according to NF EN 12390-4 for the concrete at 20 C that was not subjected to high temperatures.

After 28 days, specimens are dried in an oven (at 100℃), until stabilization of their mass. All specimens are subjected to high temperatures: 200℃, 400℃ and 600℃ according to the time-temperature schedule of ASTM E 119-00. After cooling, they were subjected to compression tests.

Phase compositions of these concretes were investigated on the fine powders using *x*-ray

Table 2 Compositions of concrete with and without glass powder

Type of concrete	Composition of concrete					
	Cement kg/m^3	Glass powder kg /m^3	Water kg /m^3	Sand kg /m^3	Gravel (3/8) kg /m^3	Gravel (8/16) kg /m^3
CR	350	-	215.36	651.83	142.63	968.94
GP 5	315	17.5	215.36	651.83	142.63	968.94
GP 10	284	31	215.36	651.83	142.63	968.94

CR: control concrete without glass powder; GP 5: Concrete dosed with 5 % of glass powder;
GP10: Concrete dosed with 10% of glass powder.

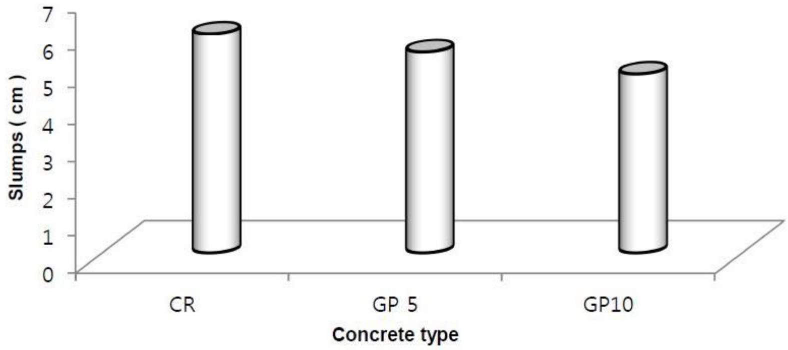


Fig. 3 Variation of slump of concrete with cement replacement by glass powder

diffraction method. The powder samples of concretes heat treated aggregates at 200 and 400℃ were collected after abrasion. *X*-ray diffraction analysis was performed on an *x*-ray diffractometer (X'Pert) coupled to a computer system. The essential purpose of this analysis is to identify different crystalline phases present in a sample. Gravimetric and differential thermal analyzes (ATG and ATD) make it possible to quantify portlandite. These techniques are used to characterize degradations.

3. Results and discussion

3.1 The effect of GP on concrete workability

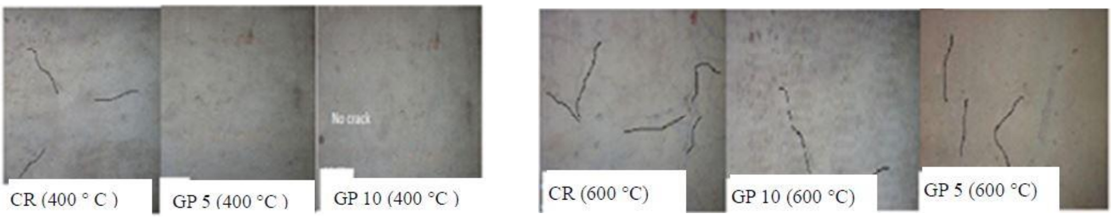
The effect of dosing fillers on concrete workability was evaluated by the Abram's cone immediately after mixing.

Fig. 3 shows the results of workability of concrete in which the glass powder is added at dosages of 5% and 10% of cement weight. It seems that workability of concrete decreases as the glass content increases.

Based on the experimental results presented in Fig. 3, the incorporation of mineral additions affects the workability of concrete negatively. Indeed, glass content increases (i.e., cement content decreased) workability decreases. As there is a reduction in fineness modulus of cementations' material, quantity of cement paste available is less for providing lubricating effect per unit surface area of aggregate. Therefore, there is a restrain on the mobility (Raju and Kumar 2014).

(a) At 200℃ (b) At 600℃

Fig. 4 Concrete samples exposed to high temperature (200℃ and 600℃)



(c) Cracking behaviour of CR, GP 5 and GP 10 exposed to 400℃

(d) Cracking behaviour of CR, GP 5 and GP 10 exposed to 600℃

Fig. 5 Cracking behaviour of concrete samples exposed to high temperature (400℃ and 600℃)

3.2 *High temperature effects on surface properties of concrete*

As noticed in Fig. 4, some color changes happened on surface of concrete samples due to high temperature effects.

As a result of these color changes, it can be absolutely appraised the range of temperature values. It can be seen that cracking (illustrated by Fig. 5), rupture, and color changes happened much more in the concrete samples exposed to 600℃ than the ones to 200℃ and 400℃.

The surface of specimens has been observed after exposed to different temperature levels and cooled to ambient temperature. Concrete GP 5 and GP 10 did not detect any visible cracking at higher exposure temperature of 400℃ and developed only one or two minor cracks at 600℃.

On the other hand, CR specimens started developing hair line cracks at 400℃ itself and when the temperature was increased to 600℃, an increased number of wider and distributed cracks developed. Figure 5 depicts the cracking behavior of GP 5, GP 10 and CR concrete specimens after they were exposed to different temperature levels of 400℃ and 600℃.

3.3 Loss of concrete's mass related to temperature

Fig. 6 shows the evolution of mass loss during the heating cycle of the studied specimens.

The mass of concretes decreases with increasing temperature due to loss of moisture. The retention in mass of concrete at elevated temperatures is highly influenced by the type of aggregate (Kodur 2014). Here are some facts for this.

√ Before 200℃, the mass change is very low. The mass loss in this temperature range corresponds generally to the water escape from concrete pores.

√ Between 200 and 600℃, a strong mass loss for all concrete specimens tested. All concrete

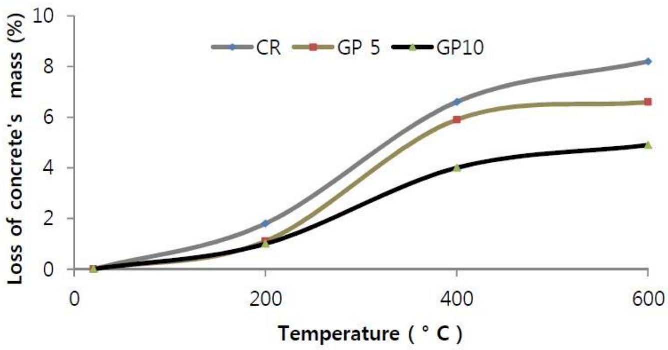


Fig. 6 Evolution of mass loss related to temperature

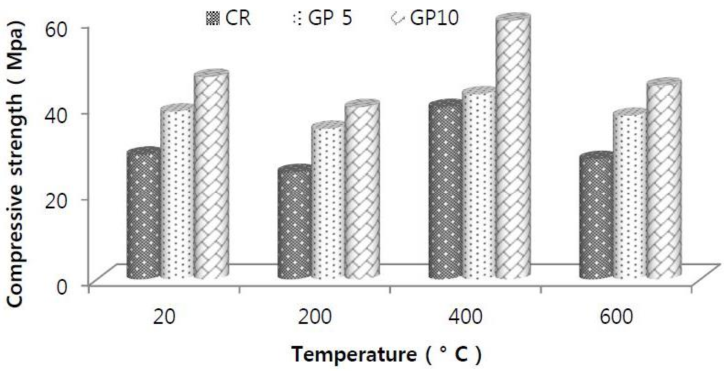


Fig. 7 Compressive strength after cooling relative to the initial strength

lost 4% to 9% of its original mass. Maximum of water in each concrete specimen evaporated during heating between 200 and 400℃.

√ The increase is almost linear up to a temperature of 600℃. This is due to the evaporation of water and the progressive dehydration of CSH gel. Noumowé *et al*. and other researchers confirm that beyond 600℃ there is no more water in the concrete specimen (Noumowé and Galle 2001, Sabeur and Colina 2015).

√ The mass loss is minimal for (PG10) concretes up to about 600℃. However, the type of aggregate has significant influence on mass loss in concretes beyond 600℃. In the case of glass powder concrete, mass loss is little significant above 600℃ compared with the control concrete. This higher percentage of mass loss in concrete is attributed to dissociation of dolomite in carbonate aggregate at around 600℃ (Dwaikat and Kodur 2010).

In this part of the research, we want to highlight the influence of glass powder on the mechanical behaviour of concrete. After the passage in the oven, the concrete specimens were cooled for 24 hours in the laboratory at a temperature of 20±5℃ before submitting to compressive strength test. The compressive strength of all concretes mixtures at ambient temperature and after heating to 200℃, 400℃ and 600℃ is illustrated in Fig. 7.

As expected, the replacement of cement by 5% to 10% of glass powder increased the compressive strength by approximately 33% at 28 days. This is due to the reaction of the glass powder with calcium hydroxide formed during the hydration of cement that caused the formation of calcium silicate hydrate (CSH) as well as filler role of very fine particles of glass. Generally, it can be concluded that concretes containing GP had significantly higher strength than those of CR concretes at room temperature. After exposure to 200℃, reductions occurred in the compressive strength of concretes without GP. Results showed the strength recovery of 20% for the concretes GP 10 after heating to 400℃ when compared to 200℃. These gains at 400℃ are attributed to the increase in the forces between gel particles (Van der Walls forces) due to the removal of water content (Castillo and Durrani 1990).

According to the many researches, the high temperature can accelerate and promote the process of cement hydration and GP's pozzolanic reaction at the same time, both of which will produce calcium silicate hydrate (C-S-H) and generate strength. According to chemical equilibrium theory, it is reasonable that the chemical reaction rate and reaction degree, including cement hydration reaction and GP pozzolanic reaction, increase with the rising of reaction temperature (Malhotra and Mehta 1996, Federico 2013).

In the range of 400-600℃, severe strength losses occurred in all concretes CR, GP 5 and GP 10. In this range, cement paste contracts, whereas aggregates expand. So, the transition zone and bonding between aggregates and paste are weakened. After heating to 600℃, the compressive strengths of CR were lower than those of the concrete GP 10. This is due to the presence and quantity of glass powder in concretes that formed very denser transition zone between aggregates and paste attributed to its ultra-fine particles as filler and its pozzolanic reactions. In accordance with Hager's studies (2013), when temperature increases beyond 400℃, the concrete strength decreases more rapidly due to the degradation of calcium-silica-hydrate (C-S-H). Second phase of the C-S-H decomposes in the temperature range from 600 to 800℃, forming β-C2S.

At 600℃, one noticed that quick losses in compressive strength for GP 10 concretes are attributed to the dense microstructure in this type of concretes, which caused the build-up of higher internal pressure due to the water vapor transition of the interlayer water. Thus, this process as well as chemical decomposition of hydration products causes severe deteriorations and strength losses in concrete after subjecting to high temperatures. The greatest relative residual strength losses of concrete GP 5, GP 10 and without glass powder were observed at 600℃, which were 2%, 24% and 25% respectively.

It appears that the dosage of glass powder has no significant effect on the compressive strength at 200℃. However, between 200℃ and 400℃, the quantity of 10% GP has significant effects on the residual compressive strength.

3.5 Thermo-gravimetric and differential thermal analysis (TG-ATD)

Internal structure. Thermo-gravimetric and differential thermal analysis, ATG, and ATD of concrete subjected at high temperature are shown in Figs. 8 and 9. A test sample of 200 mg of the concrete was analyzed according to linear heating from ambient temperature to 1100℃ with a speed of 10℃/min.

Six endothermic peaks were observed: 110 -125℃, 180℃, 400℃, and 450-550℃, 573℃ and 800℃. These thermal flux peaks are essentially related to the phase exchange temperatures of the different hydrates of the cement paste. Some facts for this are given as follows.

- The free water starts evaporating rapidly, which explains the presence of double peak at 110 and 125℃.
- In the temperature range from 80 to 150℃, dehydration of éttringite takes place, followed by the decomposition of gypsum between 150 and 170℃ (Hager 2013).
- Between 200℃ and 300℃, slight variations in flux to the continuous dehydration of C-S-H, a

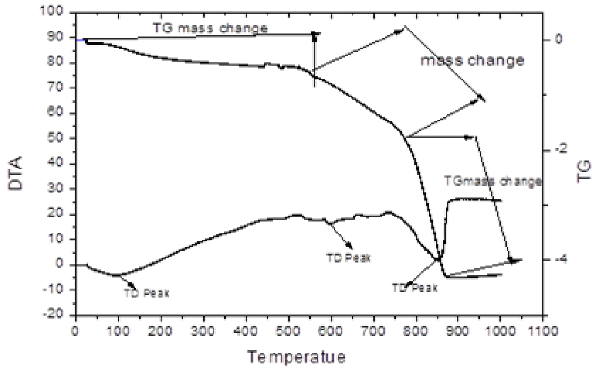


Fig. 8 ATG and ATD powder of CR at 600°C

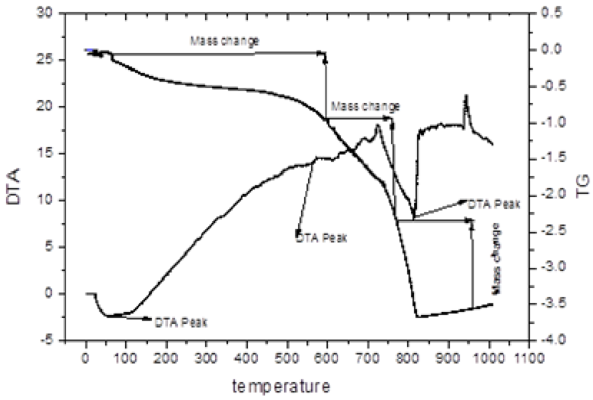


Fig. 9 ATG and ATD powder of GP 10 at 600°C

so-called “water plug” develops in concrete pores (Rao *et al.* 2013).

• At 400°C a minor peak was observed on cement pastes. Sha *et al*. (1999) attribute this change of crystalline state or dehydration to a solid solution of Fe_2O_3 but other sources (Persy and Deloye 1986) attribute this peak to the decomposition of brucite ($Mg(OH)_2$).

• The peak corresponding to the decomposition of the free limestone $Ca(OH)_2$ CaO was observed between 450°C and 550°C (Platret 2002).

• The allotropic transformation of the quartz-α quartz-β accompanied by a phenomenon of expansion . It was attributed at 573°C (Tufail *et al*. 2017).

• C-S-H decomposes and transforms into a new form of hydrates less rich in water and donation without it being formed of anhydrous compounds. These are mainly bi-calcium silicates (B-C_2S) and β-wollastonite (β-CS) between 600°C and 700°C (Platret 2002).

• At 700°C to 900°C , the limestone decomposes, so this peak indicates the decomposition of calcium carbonates ($CaCO_3$), also known as “calcite” by releasing Lime accompanied by a release of CO_2 (Khoury 1992) according to the highly endothermic reaction which is as follows

$$CaCO_3 \longrightarrow CaO + CO_2 \quad (1)$$

In the last of this study, we can say the results are consistent with a range of research done by Liu *et al.* (2014), through which he confirmed that, TG-DTA tests indicate that part of CH is consumed during the pozzolanic reaction of GP. The more the amount of GP is, the less the CH content is. High curing temperature can contribute to the pozzolanic reaction of GP, leading to more consumption of CH.

4. Conclusions

Based on experimental observations, the following conclusions are drawn:

- As the percentage of glass powder increases the workability decreases. To maintain the workability with a ratio of cement to water restricted, it is necessary to use a super plasticizer;
- Compressive strength increases with increase in percentage of glass powder up to 5% replacement.
- Very finely ground glass has been shown to be excellent filler and may have sufficient pozzolonic properties to serve as partial cement replacement.
- 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. It can offer a theoretical basis for the application of GP in precast concrete products, mass concrete or even concrete construction built during the hot season.
- The HPC specimens containing glass powder have high compressive stress compared to the concrete specimens without glass powder (CR).
- The critical temperature, which causes maximum attenuation properties of different compressive strengths and mass losses between 400℃ and 600℃.
- Color changes were observed on concrete under the effect of high temperature.

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