

## Effect of Firing Temperature on the Properties of Granulated Slag Pozzolanic Cement Pastes

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### ABSTRACT

The effect of thermally treated temperature on blended cement pastes containing ground blast furnace slag (WCS) was studied. After moulding, the cement pastes were cured under tap-water for 28 days and dried at 105°C for 24 hours. The cubes were subjected to heat treatment at rate of 10°C/min and then kept for 3 hours at the heating peak exposure temperature to establish a stable temperature at 105, 250, 450, 600 and 800°C, then cooled to room temperature in the furnace. The specimens were covered with plastic film and kept into desiccators in order to avoid the influence of humidity and the carbonization of thermally treated pastes. pozzolanic cement pastes have weight loss higher than OPC up to 600°C. At higher temperature, 800°C, the weight loss of OPC is higher than that of slag pozzolanic cement pastes. The pozzolanic cement pastes containing granulated slag is more resisting for fire than OPC. All pozzolanic cement pastes are more durable than ordinary cement paste.

**KEY WORDS:** Firing temperature, blast furnace slag, pozzolanic cement pastes.

### 1. INTRODUCTION

Fire is one of the hazards that attack the building. The damage occurring takes place due exposure to fire. Chemical processes stimulated by increase in temperature on cement phases have a significant influence on the thermal deformation, cracking, spalling and compressive strength losses. In these processes, the water loss, therefore, adsorbed and chemically water effects on capillary and gel porosity as well as on the microstructure to collapses CSH gel were removed (Rostásy *et al.*, 1980 & Galle, and Sercombe, 2001). Between temperature range (100-300°C), free and bound water from CSH gel are removed. Above 300 °C a reduction in strength in the range of 15-40% is occurred. At 550°C, reduction in strength in the range 55-70% is occurred due to dehydroxylation of Ca(OH)<sub>2</sub> to produce CaO.

Pozzolanic materials were used to enhance strength and reduce permeability. The pozzolanic material depletes Ca(OH)<sub>2</sub> liberated from the OPC hydration to produce additional CSH, therefore, they enhance the fire resistance. Furthermore, pozzolanic materials are expected to play a significant role in the self-healing process of high strength concrete, when damaged through transformation of Ca(OH)<sub>2</sub> deposited in cracks into CSH.

The hardened pozzolanic cement pastes were tested for fire resistance cured at 28 days under tap water, dried for 24 hours at 105°C, then subjected to thermal treatment for 3 hours at 105, 250, 450, 600 and 800°C, then cooled to room temperature in the furnace. The weight loss, bulk density, total porosity and compressive strength were determined. The phase composition was determined by XRD. The results show that additional hydration of unhydrated phases, recrystallization, deformation and transformation of CSH phases was occurred (Gustafero, 1983). The change in physical state of cement paste was studied by measuring the deterioration in the compressive strength with temperature.

The work aimed to study the effect of thermally treated temperature on granulated slag pozzolanic cement pastes.

### 2. EXPERIMENTAL

The materials used in this investigation were ground granulated blast-furnace slag (WCS), which provided from iron & steel company; Helwan, Egypt, and ordinary Portland cement (OPC) from Suez cement Company. The chemical analyses of these raw materials are shown in Table (1).

Table (1): Chemical composition of starting materials, (wt%).

Oxides	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	MnO	TiO <sub>2</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	BaO	SO <sub>3</sub>	S <sup>-</sup>	L.O.I
Materials													
WCS	37.48	12.86	0.40	36.70	2.45	6.24	0.72	1.84	0.71	5.31	0.01	0.75	
OPC	21.51	5.07	4.39	65.21	2.00	0.15	-	0.23	0.29	-	0.25	-	2.40

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The dry constituents of OPC and WCS were mechanically mixed for one hour in a porcelain ball mill using three balls to attain complete homogeneity, then kept in airtight containers until the time of cement preparation. The mix compositions and W/C ratio and initial as well as final setting time are given in Table (2).

Table (2): The Mix Composition of Specimens, Water of Consistency and Initial as well as Final Setting Time of Cement Pastes.

Mix No	OPC	Slag	Water of consistency %	Setting time (min.)	
				Initial	Final
M <sub>0</sub>	100	—	25.2	180	244
MS <sub>1</sub>	90	10	25.2	176	245
MS <sub>2</sub>	80	20	25.5	187	250
MS <sub>3</sub>	70	30	25.6	191	265

The mixing was carried out on the cement powder with the required water of standard consistency (ASTM Designation: C191, 2008). The blended cement was placed on a smooth non-absorbent surface and a crater was formed in the center. The required amount of water was poured into the crater by the aid of a trowel. The dry cement around the outside of the crater was slightly troweled over the remaining mixture to absorb the water for about one minute. The mixing operation was then completed by continuous vigorous mixing for about three minutes by means of ordinary gauging trowel. At the end of mixing, the paste was directly moulded in (2×2×2cm) stainless steel moulds. Freshly prepared cement paste was placed in the moulds into two approximately equal layers, compacted and pressed until homogeneous specimen was obtained. The moulds were then vibrated for a few minutes to remove any air bubbles to give a better compaction of the paste, and then the moulds were cured in a humidity chamber at 100% relative humidity at constant temperature of 25 ± 1°C for the first 24 hours. The samples were cured under tap-water for 28 days and dried at 105°C for 24 hours. The cubes were heated at a rate of 10°C/min and then kept for 2 hours at peak exposure temperature up to 800°C to establish a stable temperature (Heikal, 2006). The pastes were kept for 3 hours at 105, 250, 450, 600 and 800°C, then cooled to room temperature in the furnace and taken out for testing. After cooling, the specimens were covered with plastic film and kept into desiccators in order to avoid the influence of humidity and the carbonization of thermally treated pastes. The total porosity of the cement pastes was determined according Egyptian specification. The compressive strength was carried out on four samples as described by ASTM Specifications (ASTM Designation: 150, 2007).

X-ray diffraction technique was carried out on some selected hydrated and fired cement pastes. The samples were finely ground to pass from a 200 mesh sieve. The samples were carried out using X-ray diffraction using a Philips diffractometer with a scanning speed of 20°/min. (Ni-filtered CuK<sub>α</sub> radiation). The identification of all samples were conformed by computer-aided search of the PDF data obtained from joint committee on powder diffraction standards International Center for Diffraction Data (JCPDS-ICDD), 2001.

### 3. RESULTS AND DISCUSSION

#### 3.1 Weight loss

The weight loss of hardened OPC and pozzolanic cement pastes (MS<sub>1</sub>, MS<sub>2</sub> and MS<sub>3</sub>) as a function of thermally treatment temperature at 105, 250, 450, 600 and 800°C are shown in Figure (1).

Generally, the weight loss increases with thermally treatment temperature due to the decomposition of some hydration products (Heikal, 2006). The free water was removed below 105°C, partially decomposition of calcium silicate, aluminite, sulpho-aluminite and aluminite-silicate hydrate takes place below 200°C. The decomposition of gehlenite hydrate (C<sub>2</sub>ASH<sub>8</sub>) is occurred at 200-250°C. Above 300°C, the decomposition of hydrogarnet is up to 400°C. The dehydroxylation of Ca(OH)<sub>2</sub> at temperature of thermally treatment 420-520°C (Morsy *et al.*, 1998) was occurred. The weight loss increases up to 800°C, due to the decomposition of CSH, CAH, calcium sulphoaluminite hydrates as well as gehlenite and hydrogarnet hydrates and Ca(OH)<sub>2</sub>. It was shown that MS<sub>1</sub>, MS<sub>2</sub> and MS<sub>3</sub> have weight loss higher than OPC up to 600°C. At higher temperature, 800°C, the weight loss of OPC is higher than that of slag pozzolanic cement pastes. The pozzolanic cement pastes have lower combined water than OPC paste. Also, the increase of weight loss of OPC at 800°C may be due to the higher content of CaCO<sub>3</sub> in OPC pastes. These pastes give higher values of free lime which tend to be carbonated.

Increase the treatment temperature up to 800°C the weight loss of OPC pastes shows the higher values due to the increase of the amount of Ca(OH)<sub>2</sub> decomposed and converted to CaO. The pozzolanic cement pastes have lower amount of Ca(OH)<sub>2</sub> as well as CaCO<sub>3</sub> contents.

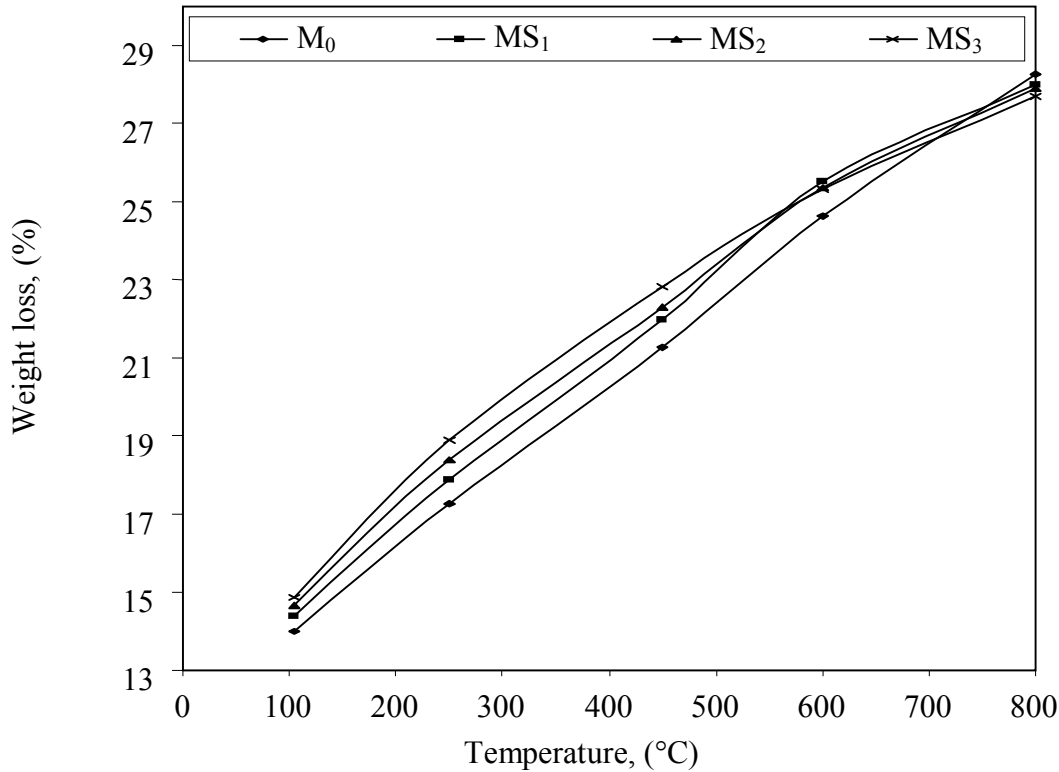


Figure (1): Weight loss of OPC and slag-cement pastes at different heat treatment temperature up to 800°C

### 3.2 Bulk density:

Bulk density of thermally treated OPC and pozzolanic cement pastes containing blast furnace slag (WCS) is shown in Figure (2) as a function of thermally treatment temperature from 105°C up to 800°C

It is clear that the bulk density decreases with curing temperature up to 250-450°C. First the removal of free or adsorbed as well as some combined water contents comes from structural CSH or CAH. The change of bulk density of different thermally treated cement pastes are proceeded in three steps. Generally, the apparent density decreases with the treatment temperature up to 250°C (first step). The second step, from 250-600°C the density has almost the same value. The third step (from 600-800°C) is accompanied by an increase in the density for all pozzolanic cement pastes. OPC cement paste has higher apparent density than the other pozzolanic cement pastes. This is mainly attributed to the formation of more hydration products leading to a denser structure.

### 3.3 Total porosity:

The variations of porosity of OPC and pozzolanic cement pastes are graphically plotted as a function of treatment temperature in Figure (3)

It is clear that the porosity increases with treatment temperature, due to the formation of internal cracks. At (105-250°C) MS<sub>3</sub> has the lowest values of porosity than other pozzolanic cement as well as OPC pastes. This is due to the pozzolanic activity of slag which increases with temperature.

Increase of the treatment temperature (450-800°C) illustrates that the porosity of OPC has the lowest values than all pozzolanic cement pastes. Also, in this range of temperature the porosity increases with WCS content. This is due to the increase of water of consistency and the slow rate of hydration of slag cement pastes.

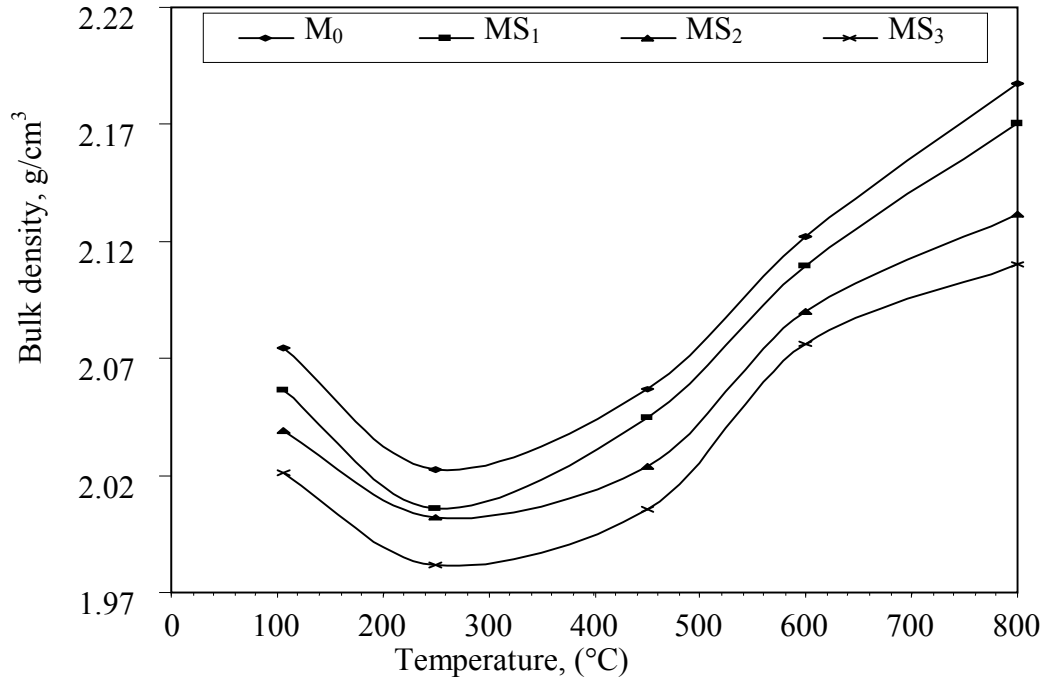


Figure (2): Bulk density of OPC and slag-cement pastes at different heat treatment temperature up to 800°C

### 3.4 Compressive strength:

Compressive strength of thermally treated OPC and pozzolanic cement pastes containing WCS namely MS<sub>1</sub>, MS<sub>2</sub> and MS<sub>3</sub> as a function of thermally treated temperature from 105 up to 800°C is graphically represented in Figure (4). The compressive strength of OPC pastes increases up to 250°C due to self autoclaving of the cement pastes (Piasta, *et al.*, 1984). The increase of temperature from 250 up to 600°C results in a decrease in compressive strength, then increase the temperature up to 800°C, the OPC and MS<sub>1</sub> specimens were completely destroyed without any compressive loading.

The temperature range between 100-300°C, leads to additional hydration products from the anhydrous cement grains. The compressive strength increases with the addition of WCS from 450 up to 800°C. 30 wt% slag pozzolanic cement pastes is more heat durable at 250°C than the other pozzolanic cement pastes. This is due to the reaction of liberated lime with the pozzolanic materials to form additional amounts of calcium silicate, aluminates and aluminosilicate hydrates. These hydrates deposited within the pore system which increase the compressive strength.

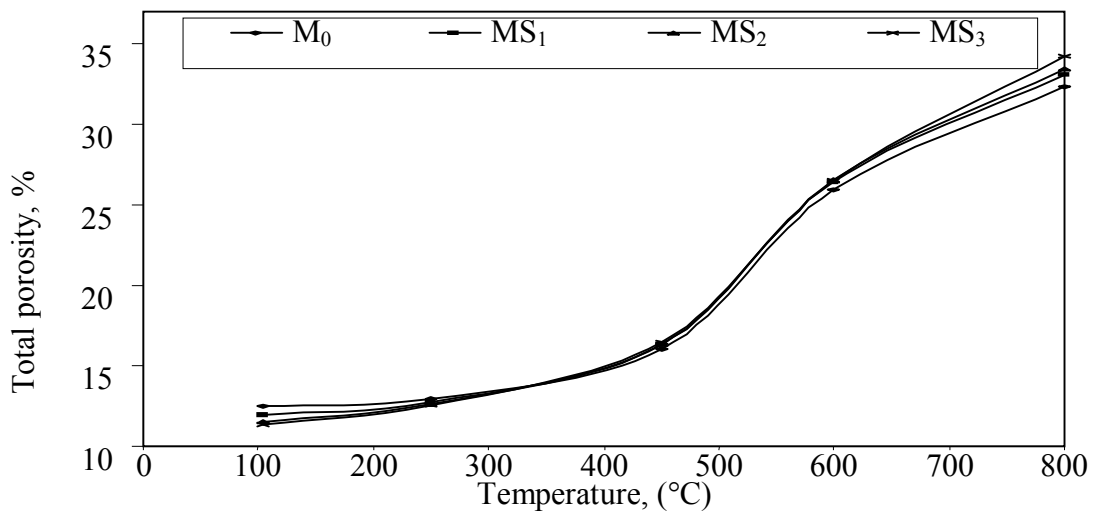


Figure (3): Porosity of OPC and slag-cement pastes at different heat treatment temperature up to 800°C

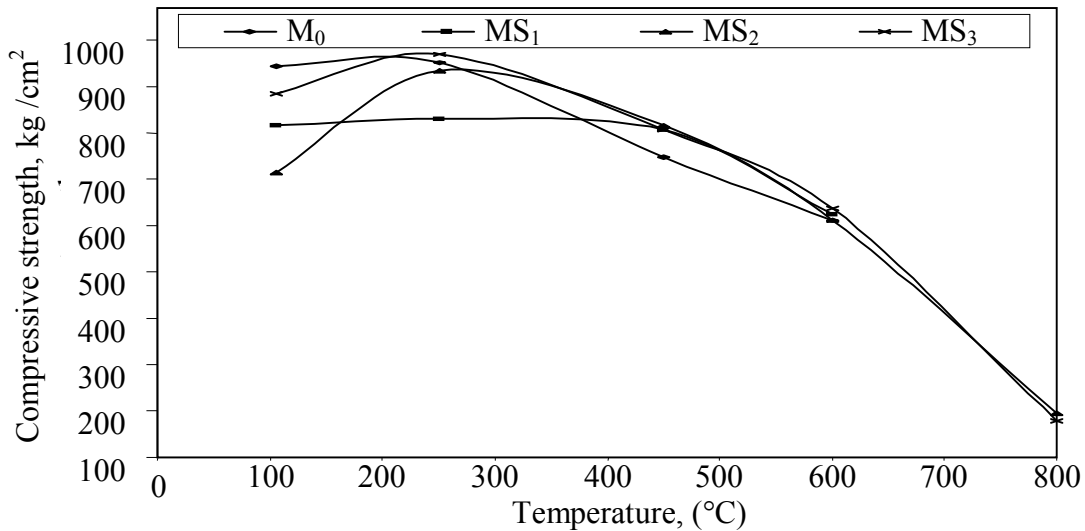


Figure (4): Compressive strength of OPC and slag-cement pastes at different heat treatment temperature up to 800°C

### 3.5 X-Ray diffraction analysis:

Figure (5) illustrates the XRD patterns of pozzolanic cement pastes containing 0, 10, 30 WCS (M<sub>0</sub>, MS<sub>1</sub>, MS<sub>3</sub>) thermally treated at 600°C. The intensity of calcium hydroxide (CH) peaks decreases in the diffractograms of MS<sub>3</sub> (30% slag) due to the pozzolanic reaction between slag and Ca(OH)<sub>2</sub> as well as the decrease of OPC content. The intensity of CSH peak is the highest in M<sub>0</sub> (100% OPC) due to the higher content of hydrating species. The increase of the peak of CSH in the case of OPC paste is mainly due to the increase of CH. The CH may be carbonated. The line of CC is overlapped with that of CSH.

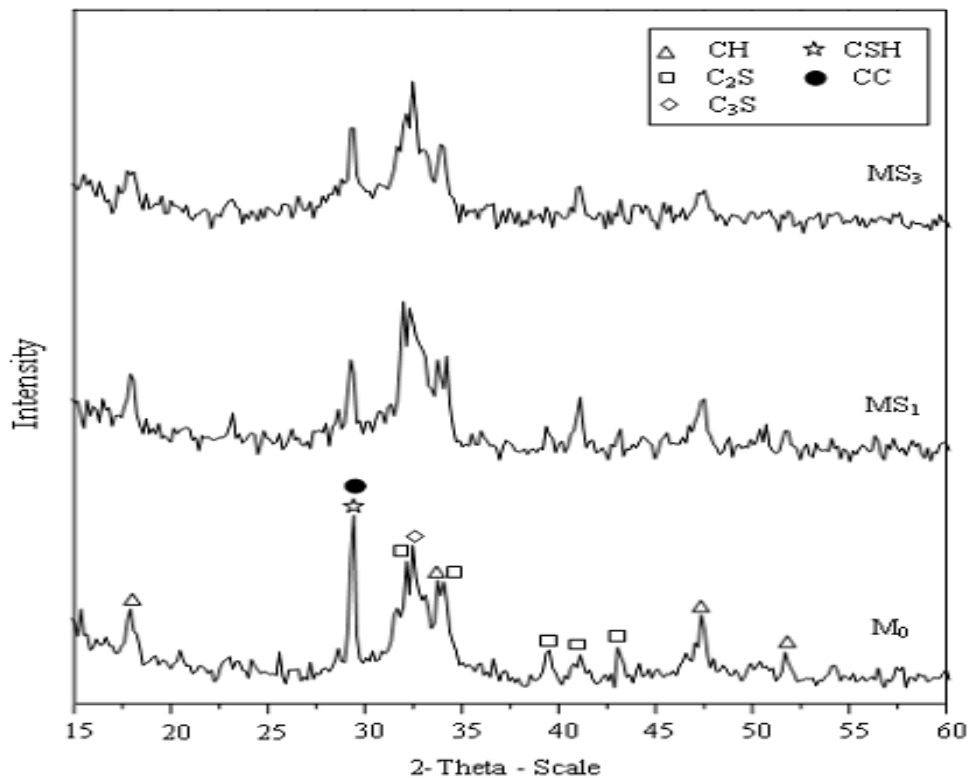


Figure (5): XRD patterns of blended cement pastes containing 0, 10 and 30% WCS thermally treated at 600°C

Figure (6) illustrates the XRD patterns of pozzolanic cement pastes containing 30% WCS (MS<sub>3</sub>) and thermally treated at 250, 450, 600 and 800°C. The intensity of characteristic lines for Ca(OH)<sub>2</sub> (4.90, 2.62, 1.92 and 1.79 Å<sup>2</sup>), decreases with the treatment temperature. The intensity of Ca(OH)<sub>2</sub> is nearly the same up to 450°C due to the crystallization and acceleration of hydration with heat treatment. At 600°C the lines of Ca(OH)<sub>2</sub> are

nearly decomposed or disappeared and the lines of CSH and tobermorite still present. There are new lines appear due to anhydrous calcium silicate phases such as  $\beta$ -C<sub>2</sub>S at 800°C. The increase of Ca(OH)<sub>2</sub> lines from 250°C up to 450°C due to the self autoclaving condition, which transformed the anhydrous grains to hydrated phases in the presence of water vapour and temperature up to 450°C. This results of formation of additional calcium silicate hydrate and tobermorite phase, from the hydration reaction of slag grains hydrothermally with Ca(OH)<sub>2</sub> liberated from the hydration of OPC phases. The lines of CSH hydrated phases show a maximum at 600°C. At 800°C the most hydrated phases were completely disappeared, this mainly replaced by crystalline phases with structure similar to C<sub>2</sub>S (Alonso and Fernandez, 2004) at diffraction lines (2.84698, 2.74999, 2.18954 and 2.03751). The formation of  $\beta$ -C<sub>2</sub>S as shown in the figure started at 600°C, this is due to the decomposition of CSH and tobermorite phases (Heikal, 2006). The origin is from the decomposition of Ca(OH)<sub>2</sub> and CaCO<sub>3</sub>. This is also present in all heated specimens and the same for C<sub>2</sub>S (Alonso and Fernandez, 2004). Increasing the heat above 500°C, the cement pastes show that Ca(OH)<sub>2</sub> and calcite decomposed into CaO. But by thermally treated up to 250-450°C an increase of Ca(OH)<sub>2</sub> is found due to progress of hydration of residual anhydrous components. This hypothesis is sustained by the parallel increase in CSH, indicating higher content of CSH gel.

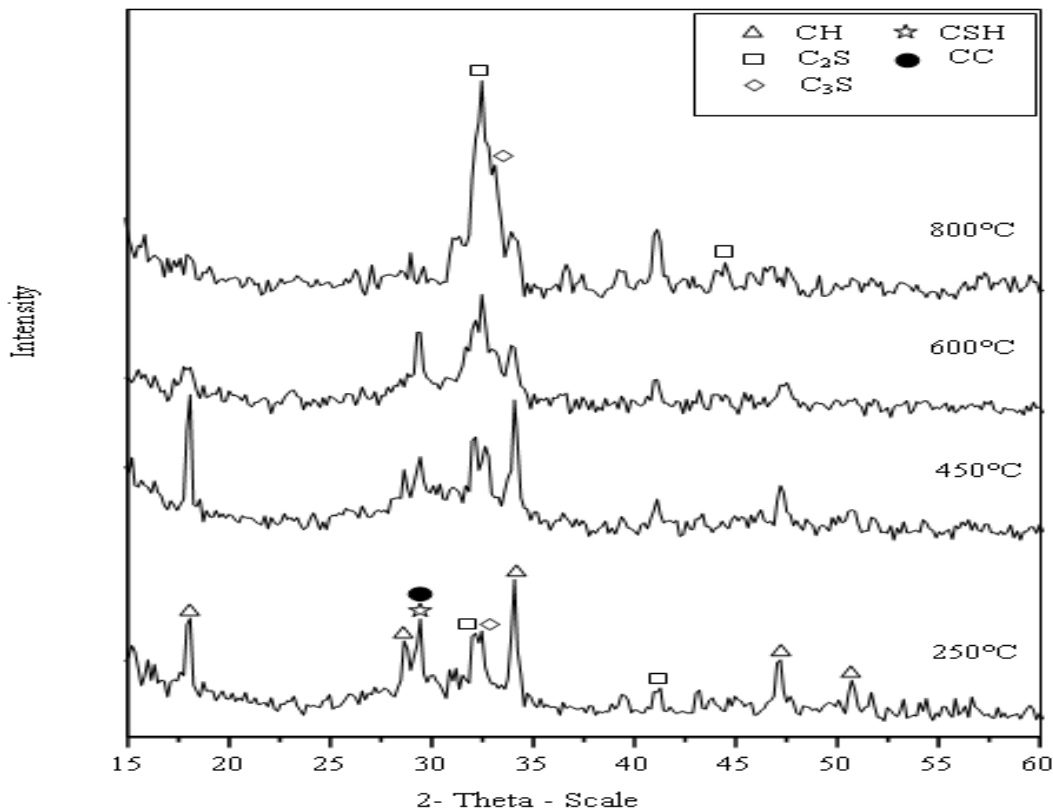


Figure (6): XRD patterns of pozzolanic cement pastes containing 30% WCS thermally treated at 250, 450, 600 and 800°C

## CONCLUSIONS

From the above findings it can be concluded that:

1. Pozzolanic cement pastes containing WCS have weight loss higher than OPC up to 600°C. At higher temperature, 800°C, the weight loss of OPC is higher than that of slag pozzolanic cement pastes
2. OPC cement paste has higher apparent density than the other pozzolanic cement pastes.
3. The pozzolanic cement pastes containing granulated slag is more resisting for fire than OPC.

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