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Effect of heating and cooling technique on residual compressive strength and weight loss of grout containing High volume fly ash

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Abstract. Grouting technology is commonly used in building and bridge construction as structural repair and gap filling material. However, the parent concrete is often mixed with conventional OPC cement-sand grout. In the event of fire, both concrete material and the grouts in structures are likely exposed to high temperatures which could results in premature failure to both materials. Research studies clearly indicated fire performances of cementitious material are affected by the mineral additives like fly ash. However, currently very little information is available on the performance evaluation of high volume fly ash grout exposed to elevated temperature. An understanding of various factors influencing fire performance will aid in developing appropriate solutions for mitigating spalling and enhancing fire resistance of concrete grouting materials. This paper discusses the fly ash grout's characteristics under elevated temperatures and effect of different types of cooling method, curing durations and fly ash content. The specimens were water cured for 28, 90, 180 and 360 days. Loss of weight and compressive strengths of OPC and fly ash grouts (10, 20, 30, 40, 50% of fly ash) exposed to high temperatures (200, 400, 600, 800 and 1000ºC) and cooled in air and water were obtained. It was observed that the compressive strength of the air cooled grout increased with an increase in the temperature up to 400 ºC; while the water cooled grout reduces strength with increase in temperature. Fly ash grout exhibited better resistance to strength loss when exposed to elevated temperature. The type of cooling affects the intensity of cracks formation, residual weight and also the compressive strength, the effect being more pronounced as the fly ash and temperature increases. These results were also supported by scanning electron microscope (SEM) studies which reveal the formation of ettringites in OPC mortar post exposure. The overall result indicated that fly ash grout could be benefiting in the construction due to better resistance to high temperature.

1. Introduction

The strength behaviour of building material needs proper evaluation when the service environment is critical, for example in a condition such as fire where the material would go thru a duration of large flux of heat. Current knowledge covers the condition of concrete exposure to elevated temperature. However, in reality, various other cementitious building materials are being used for strengthening,

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repair and construction works such as mortar, grout which will be equally exposed in case of fire. The existing literature indicates that no work has been conducted to study the influence of temperature on the strength of grout containing high volume fly ash that was cured for long duration, and cooled using both air and water. In this paper, an attempt has been made to study the temperature response of cement-sand grout with the inclusion of high volume of fly ash (up to 50%) in the laboratory. Grout are integral part of building materials assortment. This repair material can be applied in many sectors of reinforced concrete constructions (column strengthening, tunnels, bridges, cooling towers, residential buildings, waste water sumps etc.) Generally, the grout with OPC are used in the repair works. When repairing constructions with assumption of higher fire risk the application of grout is one of the possibilities where resistance of common cement grout is considerably limited. Previous studies have shown that the cement composites fire performance are linked to the material used in the manufacturing, for example the aggregates, cement and the admixtures.

The degradation mechanism happens at different level of temperature. As soon as the temperature reaches 100°C, the material first goes thru simple decomposition. However, at higher temperature around 400°C Portlandite disintegrates which with complete disintegration to 800°C [1]. However, the cement matrix also consists of C-S-H gel that goes thru some changes under certain temperature, namely at 600°C where it undergoes some disintegration, which often accompanied by volume change [1]. Also, other components such as calcites will disintegrate around 700°C. It is important to note that, all the disintegration will release gaseous substances [2]. Additional to the decomposition of major components, the residual material from fire reaction can react with water and results in the formation of $Ca(OH)2$. This hydrating reaction results in volume growth and internal stress to occurs in already degraded structure of the cement composite. This is generally results water cooling to be more detrimental to building structure compared to air cooling [3].

However, although the use of fly ash is common in the concrete application, however the use is still limited in the grouting material, particularly cement-sand grout that used in repair works in building, bridge and tunnels. Fly ash that is available due to preferences of power plant operators to use coal as fossil fuel. Investigations carried out in the past have revealed that fly ash can be utilized as an important constituent of concrete [4,5]. Fly ash is utilized in different volumes as a replacement of cement in the concrete. In general, a concrete in which 40–60% of the cement is replaced with that of the fly ash is classified as high-volume fly ash concrete. Chen et al. [6] studies shows that there is strong influence of the duration of curing, temperature and type of cooling on the residual strength of concrete. The type of cooling (water / air) was found to affect the level of recovery of strength. Various studies have shown that the use of fly ash has been promising in the concrete application. Generally, it was found that 30% fly ash have shown a highest compressive strength compared to 10, 20 when the material is heated up to 800°C [7, 8]. It was also observed by several researchers that concrete specimens had in increase in the compressive strength with the increase in temperature [10, 11, 12]. All the studies reported that the strength increases up to 250°C only, in concrete and limited to 30% of fly ash only.

The highest strength results were obtained from the specimens with 30% fly ash. The fly ash was observed to prevent the decrease of concrete strength against high temperature due to the interfacial properties mainly by the pozzolanic effect [13]. The above investigations revealed that even though a considerable work has been carried out to study the effect of varying temperatures on fly ash concrete, however, there are only a few studies in which the investigation of fire resistance of grout, particularly considering the effect of varying temperatures, long term curing and cooled using different methods. Structures repaired with grout are being exposed to high temperature in the power, nuclear and oil industries. It is, therefore, important to understand the behaviour of such material under such conditions for the safe design of structures. The effect of high temperature on the compressive strength of high-volume fly ash grout has not yet been fully understood. The present study involving the exposure of high-volume fly ash grouts to varying levels of temperature will, therefore, be helpful in understanding the behaviour of high-volume fly ash grouts at elevated temperatures.

2. Experimental program

2.1. Material and sample preparation

Grout mixes were made by replacing cement with 10, 20, 30, 40 and 50% fly ash by weight. The water to binder ratio (W/B) was kept constant at 0.40 and all the mixes contained 0.5% superplasticizer relative to the cement mass. The fly ash used in this study was obtained from a coal-fired power plant located in Peninsular Malaysia. A naphthalene sulphonate based super-plasticizer (SP) with 93% solid content and specific gravity of 0.66 was used in this study. The use of plasticizers is important because it can help to disperse cement particles that are exposed to aggregation due to colloidal interactions. The chemical compositions of the cement and fly ash are shown in table 1.

All the constituents (cement, fly ash, sand, water, and other additives) were mixed with water in a mechanical mixer at 285 rpm. On completion, grout cubes are casted using a 50mm x 50mm cube according to ASTM C109. Since the type of grout used is a non-shrink grout, the cube mould is restrained as required by ASTM C109. After 24 hours, the cubes were removed from the mould and cured in water for 28 days at room temperature of 25±2°C. The mix proportion is presented in table 2. The range of variation of compressive strength for the grout (13.0–51.0 MPa) brings the experimental parameters of the present research within the realm of the actual situation and the outcome of the experimental work is expected to closely fit-in the real life situation.

Table 1. Chemical properties of OPC and Fly ash.

Elements	Chemical Symbols	Cement (%)	Fly Ash (%)	ASTM C618, (2012) Requirement (Class F)
Silicon dioxide	SiO_2	18.24	49.97	
Aluminium oxide	Al_2O_3	5.340	28.36	Min Total
Ferric oxide	Fe_2O_3	3.214	7.04	70 %
Calcium oxide	CaO	60.16	5.17	-
Magnesium oxide	MgO	0.5550	2.07	-
Sulphur trioxide	SO_3	2.137	0.46	Max 5 %
Sodium oxide	Na_2O	0.0397	2.11	-
Potassium oxide	K_2O	0.5755	1.50	-

Table 2. Mix proportions of grout.

Materials	Fly ash content (%)					
	0%	10%	20%	30%	40%	50%
OPC	55.95	50.36	44.76	39.17	33.57	27.98
Fly Ash	0.00	5.60	11.19	16.79	22.38	27.98
Sand	43.00	43.00	43.00	43.00	43.00	43.00
W/Binder Ratio	0.40	0.40	0.40	0.40	0.40	0.4

2.2. Heating and cooling procedure

Before placing the specimens in the furnace, all the specimens were dried in an oven at 100°C for 24 hours. An electric muffle furnace with a maximum operating temperature of 1,000°C was used to heat the specimens. The furnace was capable to accommodate only six specimens at a time. 6 cubes from single mixture of grout specimens were heated at a time in the furnace. At the time of placing the specimens in the furnace, the inside temperature of furnace was at the room temperature. The average rate of heating was kept at 5°C/min and after reaching the peak the temperature was kept constant throughout the exposure duration of 1 h. The furnace was completely closed during the heating process. The furnace was then switched off and the specimens were removed from the furnace for cooling. 3 specimens were air cooled to reach atmospheric temperature which was 27±2◦C. The remaining 3 cubes were water cooling by placing the specimen in basin filled with tap water of a temperature of 27±2°C. The heating and cooling of specimen pattern are depicted in figure 1. The heating rate was kept slow for avoiding cracking of grout due to high temperature gradient between the core and the outer surface. Any sudden exposure of concrete to temperature either high or low causes the specimen to rapid expansion or contraction resulting in disintegration of the material. Thus the cooling duration was taken as 3 h (minimum).

The peak temperature (100, 200, 400, 600, 800 and 1000°C) values are considered keeping in view of thermal environments such as clinker silos and nuclear reactors etc. and the fire conditions. The total number of cubes tested in the study is thus 6 cubes × 6 mixes × 5 testing durations = 180 cubes. A close examination of the specimens was done for possible changes in the colour and sign of disintegration. The crack propagation and weight loss of the specimens were also studied. Subsequently, the specimens were tested for determining their compressive strength so as to ascertain the effect of elevated temperature on high-volume fly ash grouts. The set-up of the heating equipment is shown in figure 2.

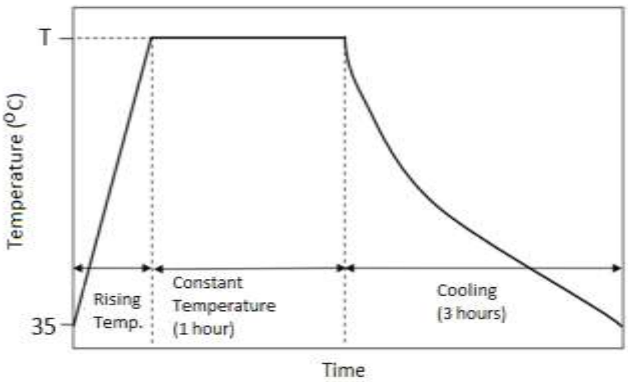


Figure 1. Heating and cooling regime.

Figure 2. Set-up for heating of specimens.

3. Results and discussions

3.1. Color change

Previous researcher has reported that the cementitious material, particularly concrete does change colour when exposed to elevated temperature and the colour change is related mainly to the intensity of the temperature [14]. The major changes in colour, including in the current study are related the intensity of the temperature that the specimen are exposed. The changes in colour is an important indicator as it provides first-hand information about the in-situ condition of the material after exposed to elevated temperature. Generally, any OPC mixture in concrete, mortar or grout are known to be in grey in colour, and sometimes darker in colour based on the mineralogical composition of the cement, such as the magnesium content. Meanwhile, fly ash can be dark grey or brownish, depending on its chemical and mineral constituents. A brownish colour is typically associated with the iron content. A dark grey to black colour is typically attributed to unburned carbon content. Fly ash colour is usually very consistent for each power plant and coal source. The fly ash used in current study is brownish in colour. Therefore, the grout colours changes from greyish to more brownish as the fly ash content increases. The visual comparison of the grout specimens is shown in figure 3.

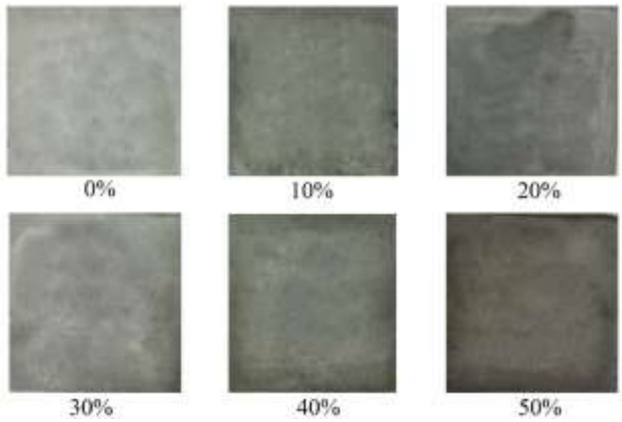


Figure 3. Colours of specimen before heating.

Therefore, it is important that the original colour of the specimen must be factored when considering the thermal induced colour change. The influence of temperature in the current study shows that the colour of specimens was found to be greyish (for grout up to 10% fly ash) and light brownish for rest of specimen when the peak temperature was up to 400°C. However, the specimens attained light brown colour for peak temperature of 600°C. Moreover, at 1000°C peak temperature the colour of the specimens again changed to dark brown. The higher the fly ash content, the darker the brown appearance noticed. The variation of colour with peak temperature is shown in figure 4. It can be concluded from the above results that the colour of grout is indicative of the intensity of temperature exposure and corresponding duration to which it has been exposed.

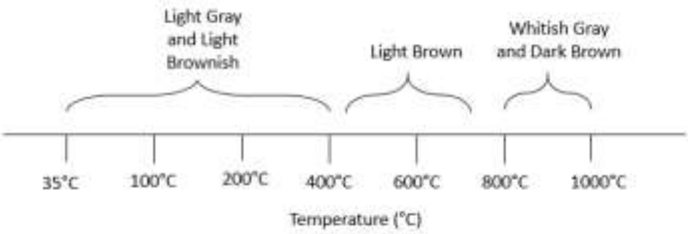


Figure 4. Changes in colour due to heating.

3.2. Cracking

Cracking has been observed for nearly all the grout exposed to high temperatures. Additionally, it was observed in current study that the type of cooling affects the cracking of grout. Grouts specimens that were taken out from the furnace and cooled by air has less cracks compared to those rapidly cooled using water. The intensity of the crack was larger on the specimens that were water cooled. This is due to the reason, which is known as thermal shock. Grout being a composite cementitious building material, has a poor thermal conductivity. Therefore, the thermal gradient which occurs during the heating and cooling process results in the cracks formation in the grouts.

Beside the type of cooling, three other factors, the fly ash content, specimen maturity, and maximum temperature found be influencing the intensity of the crack. Inspection made on the specimen cured for different duration (28, 90, 180 & 360 days) shows that when the specimens cured for longer duration, the crack reduces. For examples, for air cooled specimen, it can be seen in figure 5 and figure 6 that a 28 days specimen exposed to 1000°C has slightly lesser micro-cracks on the surface compared to the those aged up to 360 days. While, for water cooled specimen as shown in figure 7 and figure 8, grout exposed to 1000°C at 28 days had a very severe deterioration while the grout tested at 360 days was fully disintegrated.

In-term of fly ash content, specimens with lesser fly ash has higher presence of crack on the surface. For example, the grout without any fly ash and with 10% of fly ash has the most number of cracks. When the specimen was exposed to lower temperature, i.e. 600, 400, 200 and 100°C, the effect of cooling on the cracking is not noticeable. However, when the heating temperature is as high as 1000°C, the rapid cooling results in a serious deterioration of the grout with any ash, but the grout with 50% fly ash stayed in-tact.

It is known that during the heating process, pore pressure develops inside the cement matrix that results in cracking. This scenario is different compared to concrete as in concrete it is known that the thermal compatibility between the aggregate and the cement paste often results in the development of large crack at temperature as low as 800°C. However, since the grout consist of smaller fine aggregate, the expansion of the aggregate is found to be not as significant as concrete. Thus, grouting material probably a better alternative for structure at high risk for fire deterioration.

Additionally, grout with high volume fly ash can be used as structural elements as compared to a normal OPC grout that could easily fail during fire and when water was used as extinguishing medium. In concrete, it has been reported by some researchers that the increase in fly ash reduces concrete resistance to high temperature. This loss of strength is suspected which could be due to the thermal incompatibility that breaks the bond between the aggregate and the binder phases and thus developed internal pressure within the concrete matrix. The reason behind the strength loss with the increase of fly ash is due to a reduction in the thermal conductivity causes a greater thermal incompatibility [15].

Furthermore, previous studies [16, 17] have shown that cracks forms around 600°C as a result of decomposition of calcium hydroxide (Ca(OH)2), results in the shrinkage within the cement matrix. The initial cracking is usually due to thermal expansion of cement paste causing local breakdowns in bond between the cement and the aggregate. As the maximum exposure temperature rises, drying shrinkage eventually becomes much greater than thermal expansion as water is driven off. These two opposing actions progressively weaken and create cracks in the cement composites [18].

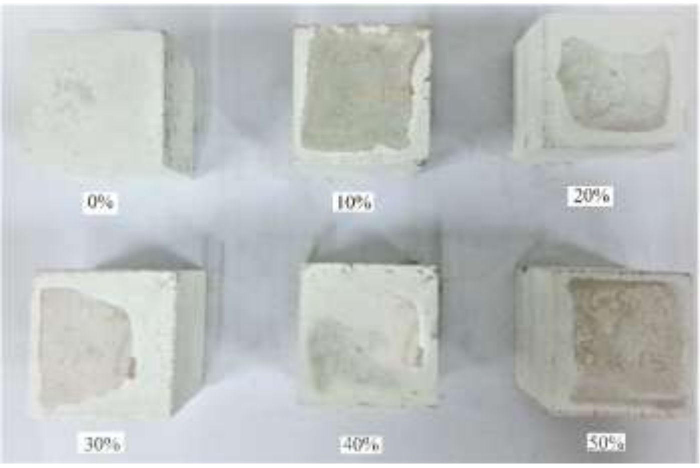


Figure 5. Air cooled grouts micro-cracks on surface at 1000°C at 28 Days.

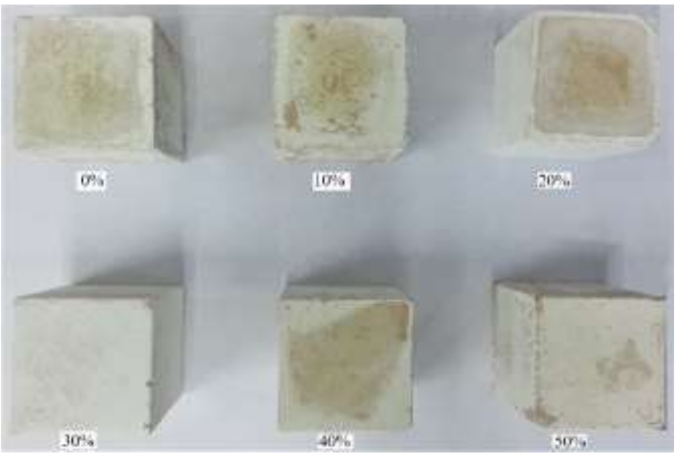


Figure 6. Air cooled grouts micro-cracks on surface at 1000°C at 360 Days.

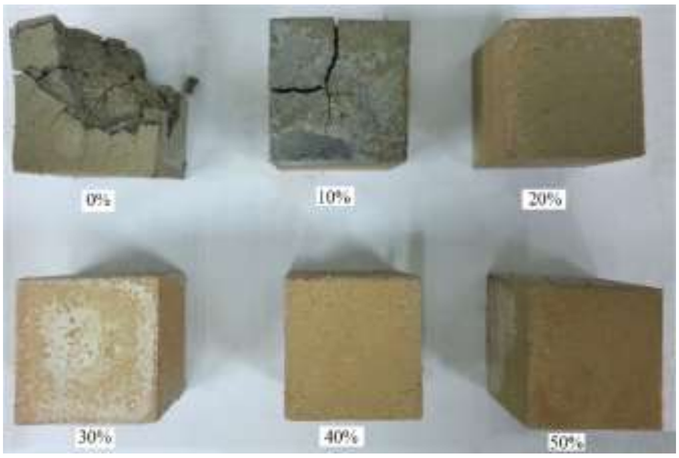


Figure 7. Water cooled grouts cracks on surface at 1000°C 28 Days.

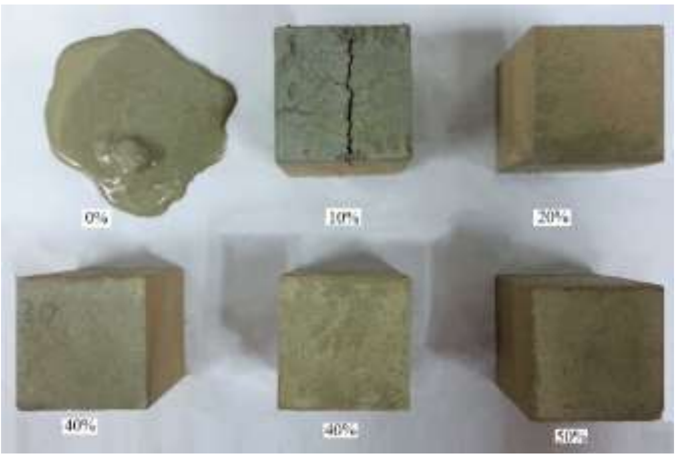
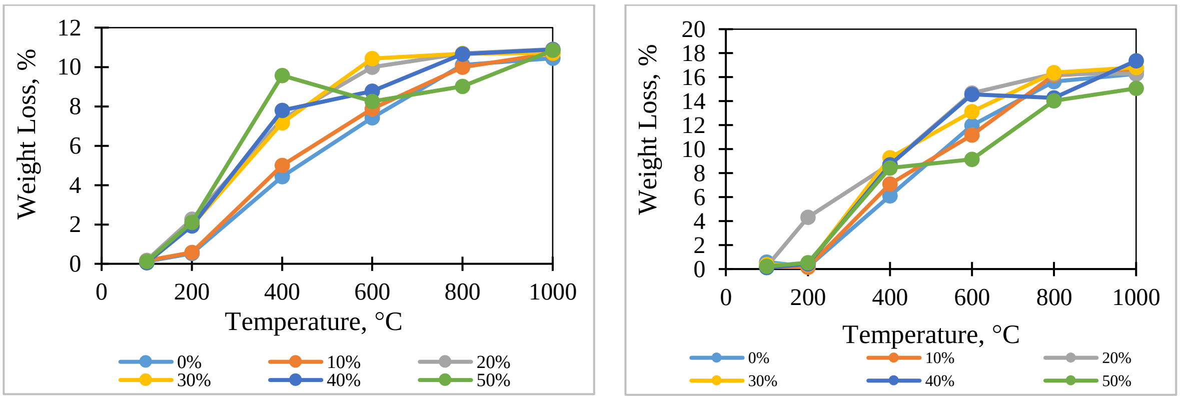


Figure 8. Water cooled grouts cracks on surface at 1000°C 360 Days.

3.3. Loss of weight

The weight loss of specimen after exposed to high temperature and air cooled are shown in figure 9. Generally, it can be observed that throughout all the curing duration, the loss of weight increases as the exposed temperatures increases. The loss of weight is only applicable for air cooled specimens. 28 days old specimens exposed to 100 and 200°C shown no significant weight loss. But, when the specimens exposed to temperature of 400 and 600°C, it was observed that the weight loss of the fly ash specimens were generally higher than the OPC specimens. At 800 and 1000°C, the 30% and 20% fly ash specimens had the highest weight loss. The increased weight loss at higher temperature of 800°C and above was due to the decomposition of materials, whereas the weight loss at 200°C was only due to the loss of moisture from inner grout structures. Furthermore, at a given peak temperature, the loss of weight increases with an increase in the fly ash content. This increase may be due to difference in pore structures of the specimens with different fly ash content. 28 and 90 old specimens with 20% of fly ash suffered the maximum strength loss when exposed to 1000°C, with a value 10.9% and 12.33% respectively.

The weight loss was found to increase with the aging of the specimen. This is suspected due to the fly ash particles that participated in the Pozzolanic reaction to form C-S-H that has decomposes in high temperature. At 180 days, it was found that the grout specimen with 30% fly ash had a higher weight loss of 12.77% and 13.40% at 800°C and 1000°C respectively. However, at 360 days, it was found that at 800°C, grout with 30% has the highest weight loss of 16.38%, while at 1000°C grout with 40% fly ash has the higher loss with a value of 17.37%. Although the weight loss is affected by the curing duration and fly ash content, the increase in fly ash has shown an increased in weight loss, additionally as the grout matures, the weight also found to be increasing, particularly in high temperatures.



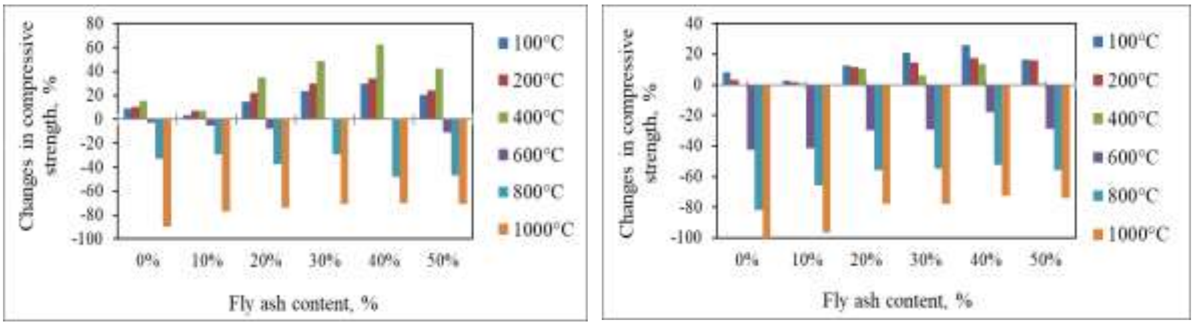
a) 28 days b) 360 days

Figure 9. Weight difference after exposed to elevated temperature, difference between 28 and 360 days old specimens.

3.4. Changes in compressive strength

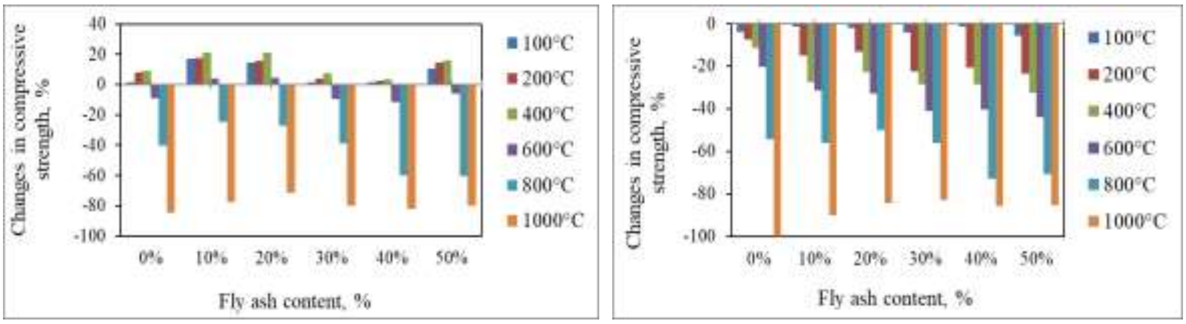
Compressive strength is one of the most important criteria when comes to fire exposure. As shown in figure 10, it was observed on 28 days old specimens that the compressive strength of the specimens increases with the increase in temperature up to 400°C regardless of being cooled by air or water. 30% fly ash specimens that were heated up to 400°C, had the highest strength gain of 48.73%, and the lowest value was observed for grout specimen with 10% fly ash with a loss of 6.90%. For water cooled specimen, 40% fly ash specimen had the highest gain of 13.64% while the lowest was OPC with 0% gain. At temperature above 600°C to 1000°C, it was found that the grouts started to lose strength in both water and air cooled specimen. For air and water cooled specimens, OPC grout had the most loss with a value of 89.39% and 100% respectively.

At 360 days, the initial heat related strength gain was found to be lesser to 28 days. On air cooled specimen, strength gain noticed on 10% and 20% ash grout up to 600°C. However, all the specimen losses strength when cooled using water regardless of the temperature. Interestingly, at the maximum peak temperature of the present study (i.e. 1000°C), OPC specimens got fully disintegrated and hence could not be tested for their compressive strength. The increase in fly ash content, generally resulted lower deterioration particularly at high temperature like 1000°C. The results are shown in figure 11. Nasser and Marzouk [19] have also reported an increase in strength of concrete containing fly ash when it was subjected to temperatures up to 149°C. It appears that the thermal incompatibility results in the breakdown of interfacial bond between aggregates and the surrounding paste which causes loss of strength of concrete at higher temperature. Peng and Huang [20] also reported that the C-S-H decomposition started at 560°C but become significant only above 600°C.



a) air cooled b) water cooled

Figure 10. Changes in compressive strength between 28 days air and water cooled samples.

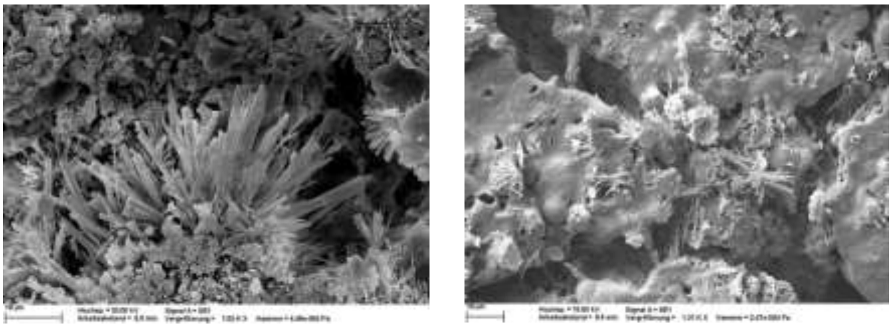


a) air cooled b) water cooled

Figure 11. Changes in compressive strength between 360 days air and water cooled samples.

3.5. Microstructural analysis

The microstructural analysis between the specimen containing high volume fly ash and without ash is shown in figure 12. It was noticed that at highest exposed temperature of 1000°C, formation of ettringite is significant in grout without any fly ash. The formation of expansive such ettringite is often expansive thus results the cracking of the grout specimen. While the high volume fly ash grouts, shown a rather plain flat surfaces.



a) OPC grout specimen (b) 50% fly ash grout specimen

Figure 12. Microstructural comparison between OPC and 50% fly ash grout after exposed to 1000°C.

4. Conclusion

The following conclusions may be drawn from the present experimental investigation on the behavior of high-volume fly ash concrete at varying temperatures.

1. The color of the grout specimen changes with the fly ash content. The grout specimen's changes to brownish with the replacement of fly ash with OPC.
2. The color also noticed to be changing with the intensity of the exposure temperature. Higher temperature found to change the specimen to whitish surface. However, when its water cooled. The specimen with 20% fly ash and above turns to soil like brownish color.
3. The weight loss increased with the aging of the specimen. This is suspected due to the fly ash particles that participated in the Pozzolanic reaction to form C-S-H that has decomposed in high temperature.
4. The strength loss is lesser with the introduction of fly ash. The water cooled specimen has suffered more severe strength loss compared to air, however as the % of the fly ash increases, the strength loss is lesser.

Therefore, it can be concluded that the inclusion fly ash in in grouting materials makes it to be more resistant to elevated temperature. Additionally, it has shown a superior performance when the material is water cooled. This material will make it suitable for structures designed for temperature exposure of up to 300◦C. Such concretes may thus be used as a liner in Clinker storage silos, reinforced concrete chimney and containment vessels of power plant etc.

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