EFFECT OF COOLING METHODS ON RESIDUAL COMPRESSIVE STRENGTH AND CRACKING BEHAVIOUR OF FLY ASH CONCRETES EXPOSED AT ELEVATED TEMPERATURES

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Abstract. *This paper presents the effects of cooling methods on residual compressive strength and cracking behaviour of concretes containing four different class F fly ash contents of 10, 20, 30 and 40% as partial replacement of cement at various elevated temperatures. The residual compressive strength of above fly ash concretes are measured after exposed to 200, 400, 600 and 800^{0}C temperatures and two different cooling methods e.g. slow cooling and rapid water cooling. Results show that the residual compressive strengths of all fly ash concretes decrease with increase in temperatures irrespective of cooling regimes, which is similar to that of ordinary concrete. Generally, control ordinary concrete and all fly ash concretes exhibited between 10% and 35% more reduction in residual compressive strength due to rapid cooling than slow cooling except few cases. Cracks are observed over concrete specimens after being exposed to temperatures ranging from 400˚C to 800˚C. Samples that are slowly cooled developed smaller cracks than those rapidly cooled. At 800˚C, all fly ash concretes that are exposed to rapid cooling showed the most severe cracking. X-ray diffraction (XRD) analysis shows reduction of $Ca(OH)_2$ peak and formation of new calcium silicate peak in concretes containing 20% and 40% fly ash when subjected to 800^{o}C in both cooling methods. Thermo gravimetric analysis (TGA) and differential thermal analysis (DTA) results show increase in thermal stability of concrete with increase in fly ash contents. The existing Eurocode also predicted the compressive strength of fly ash concretes with reasonable accuracy when subjected to above elevated temperatures and cooling methods.*

Keywords: fly ash, concrete, elevated temperatures, residual compressive strength, TGA/DTA, XRD, Cracking.

1 INTRODUCTION

The use of fly ash as partial replacement of cement in structural concrete is now a common practice around the world due to environmental concerns. Extensive research on different engineering properties of fly ash concretes have been conducted and still going on. Generally, fly ash concrete shows slow pozzolanic reaction at early ages and has significant improvements of long-term mechanical and durability properties. As a result, engineers and concrete producers have gained confidence in its use in construction.

Fire can result from anything ranging from a natural event to human error. Many civil engineering structures particularly those made of reinforced concrete experience fire during their lifetime. When designing and implementing structural materials it is important to understand all of its mechanical properties to ensure safe design. Due to the unpredictability of fire, the residual mechanical properties become very important in the design process. Despite extensive research on fly ash concretes, results on its behaviour at elevated temperatures during fire are not widely reported.

The early study of fly ash concretes at elevated temperatures dates back to late seventies, where Nasser and Marzouk [1] tested concrete containing 25% fly ash by weight at various elevated temperatures up to 232ºC. They observed an increase in compressive strength in the temperature range between 121 and 149ºC. Sarshar and Khoury [2] tested OPC-fly ash paste containing 30% fly ash by weight at different temperatures up to 650ºC and reported 12% and 27% reduction of ambient compressive strength at 450ºC and 600ºC, respectively. Nasser and his co-researcher [3] also investigated the combined effects of elevated temperature (21–232ºC) and pressure (5.2-13.8 MPa) on compressive strength of concrete containing 20 and 60% fly ash and 10% silica fume by mass of the total binder. A gradual decrease in the compressive strength was observed with increasing temperature in their study. Xu et al. [4] studied the effects of 25% and 55% fly ash as replacement on the residual compressive strength of concrete at elevated temperatures. An increase in strength was observed at 200ºC. Poon et al. [5] investigated the residual compressive strength of normal-strength concrete incorporating 30 and 40% fly ash at elevated temperatures up to 800 ◦C for 1 h duration. It was found that the compressive strength marginally increased only at 200ºC and beyond that temperature the compressive strength decreased gradually. The effects of different elevated temperatures on the residual mechanical properties of concretes containing natural pozzolan and fly ash are also evaluated at different holding periods after heating [6]. Significant loss of mechanical properties of concrete containing fly ash and natural pozzolan than ordinary concrete at 200^0C are observed. However, at higher elevated temperatures the differences are reduced. It can be seen in above studies that different fly ash contents are used by different researchers. Moreover, in all the above studies a slow cooling method was applied to evaluate the residual strength.

It is important to note that in real situation fire is often extinguished rapidly by spraying water, which causes rapid cooling of the concrete structures. Therefore, the effect of rapid cooling in terms of water spray on degradation of strength and cracking behaviour of ordinary concrete and fly ash concretes and its comparison with slow cooling will provide useful information in the design of structures. The effect of cooling on residual mechanical properties of concrete after exposed to elevated temperatures is, however, received relatively little attention. Kowalski [7] reported the residual compressive strength of ordinary concrete due to different cooling periods in water after exposed to elevated temperatures. Results show that the residual compressive strength reduced with increase in cooling periods in water. In another study Lee et al. [8] also reported reduction of residual compressive strength of ordinary concrete due to cooling after exposure to elevated temperatures. However, no study so far reported the effect of different cooling methods on the residual mechanical properties of concretes containing different fly ash contents.

This paper presents the residual compressive strength of concretes containing four different contents of class F fly ash such as 10, 20, 30 and 40% as partial replacement of cement at various elevated temperatures of 200, 400, 600 and 800^0C. The residual compressive strength of above fly ash concretes are measured after two different cooling methods namely slow and rapid cooling. This study also evaluated the physical changes in terms of surface cracking of above fly ash concretes due to two cooling methods. Existing Eurocode method to predict the residual compressive strength of fly ash concretes is also evaluated in this study. X-ray diffraction (XRD) analysis of mortar samples of above fly ash concretes were also used to identify any changes of different phases of fly ash concretes and interpret the observed behaviour. Thermogravimetric analysis (TGA) and differential thermal analysis (DTA) of mortar sample of above fly ash concretes was also conducted to study their thermal stability.

2 EXPERIMENTAL DETAILS

A total of five series of mixes were considered in this study. In the first series a control mix was used which contained no fly ash. In other four series 10, 20, 30 and 40% fly ash were used as partial replacement of cement. For each series twenty seven cylinder specimens were cast. Three were used to measure the compressive strength at ambient temperature, while the rest were exposed to elevated temperatures. At each temperature three cylinders were used for slow cooling and the other three for rapid cooling. Thus, all together 135 cylinder specimens were cast and tested in this study.

2.1 Materials

The cement used in this study was general purpose Portland cement which corresponds to ASTM type I. The class F fly ash used in this study was obtained from Collie Power Station in Western Australia. The chemical compositions of cement and fly ash are shown in Table 1. The coarse aggregates used in this study were 10 and 20mm in size. Both aggregates were crushed granite rock and prepared to saturated surface dry (SSD) condition.

2.2 Mix proportions, mixing and curing

Mix proportion of all mixes is shown in Table 2. The mixing of control and fly ash concretes was carried out in a pan mixer. First, the aggregates and cement and fly ash were dry mixed for approximately 5 minutes and then water was slowly added into the mix and continued to mix for another 5 minutes. The cylinders were then filled with concretes and compacted on a vibrating table. The concrete cylinders were demoulded after 24 hours and stored in the curing tanks where they were subjected to standard wet curing for 56 days. Longer curing time was chosen due to slow pozzolanic reaction of fly ash in concrete. Before putting the specimens in the kiln, they were dried in an oven at 105^0C for 24 hours to remove any free water from the concrete. This is to prevent the specimens from exploding in the kiln during the heating process, as a result of extremely high pore water pressure from the superheated water.

To cast the concretes, cylinders of 200mm in height and 100mm in diameter were prepared. Some cylinders were modified in order to install 20mm long and 2mm diameter removable pins so that holes would be cast into the specimens for the thermocouples to be installed.

2.3 Heating regime

The kiln used to heat the cylinders was a locally manufactured Kiln with a maximum temperature rating of 1200˚C. It was a small capacity kiln capable of fitting a maximum of 6 cylinders at any one time. Details regarding the kiln can be found in another paper [9]. The heating rate of 8˚C per minute was applied as this was the maximum rate that could be achieved in the kiln. To monitor the temperatures four "type K" thermocouples shown in Fig. 1 were set up in different positions inside the kiln (Fig. 1) and in the cylinders (Fig. 2). Two thermocouples were set up to monitor the ambient temperature inside the kiln (see Fig. 2). One was positioned 50mm from the top of the interior and another was set 50mm from the bottom of the kiln. The other two thermocouples were inserted in to a sacrificial cylinder to monitor the core temperature of the sample. All four thermocouples were then connected to a data logger to monitor the temperatures inside the kiln and cylinder. Irrespective of the target temperature the testing method remained the same throughout. Oven dried samples were put into the kiln and immediately exposed to an 8˚C per minute heating rate. This heating rate was continued until the ambient temperatures inside the kiln reached the target temperature. At this point the target temperature was maintained and the concrete was held in the kiln for a further two hours before being subject to different cooling methods.

2.4 Cooling regime

Two cooling methods were chosen, namely rapid cooling achieved from water quenching and slow cooling at ambient air. After the samples had been in the kiln for the correct duration of time the kiln was then turned off and the door kept open. The area around the kiln was roped off for safety and then left overnight giving ample time for the heat to slowly permeate out of the kiln and allow the concrete to gradually return to ambient temperature.

A 50L plastic bucket was prepared for water quenching. Firstly, the base of the bucket was filled approximately 100mm deep with 20mm aggregates to prevent melting of bucket's base. Secondly, unlike other studies, a 20mm diameter hole was punched through the side of the bucket approximately 1/3 of the way up. The purpose of this hole was to allow a continuous flow of hot water out of the bucket. The reasoning behind this is that during a fire, water is sprayed onto the concrete surface and it drain quickly due to gravity; this means that fresh cold water is continuously being sprayed onto the concrete. After the full heat treatment process was complete, the concrete samples were immediately removed from the kiln using a long pair of crucible tongs, and placed into the bottom of the bucket. A large hose was then used to rapidly spray water on cylinder into the bucket for 15 minutes. During this time the bucket completely filled up and there was a continuous flow of water out of the hole (see Fig 3). The samples were then removed from the water and left in ambient laboratory conditions overnight to allow the core temperature to return back to ambient temperature.

2.5 Testing details

The compressive strength of concretes were determined according to ASTM C39 [10] using a loading rate of 0.33 MPa/s. For each temperature and cooling method three specimens were tested and the mean value of these measurements was reported.

To study the strength change due to various temperatures and cooling regimes, X-ray diffraction (XRD) analysis were performed on powder mortar samples obtained from selected series. Mortar samples from individual specimen were collected using a pestle making sure to remove course aggregates as they appeared. They were then manually grinded until all that remained was 15mg of powder that could pass through a 0.15mm sieve. The power samples were then individually bagged and sent to the labs for analysis. The XRD analysis was performed on a D8 Advance Diffractometer (Bruker-AXS) using copper radiation and a Lynx eye position sensitive detector. The diffractometer were scanned from 3 to 70 (2h) in steps of 0.02using a scanning rate of 0.5/ min. XRD patterns were obtained by using Cu Ka lines (k = 1.5406 A). A knife edge collimator was fitted to reduce air scatter.

A Mettler Toledo TGA 1 star system analyser was used for TGA analysis. Samples of 25 mg were placed in an alumina crucible and tests were carried out in Argon atmosphere with a heating rate of 10^{o}C/min from 25 to 1000^{o}C.

3 RESULTS AND DISCUSSION

3.1 Temperature profile of cylinders at different target temperatures

The rate of temperature increase in the kiln and in the cylinder is shown in Fig. 4. The two dotted lines are for the thermocouples measuring the air temperature inside the kiln, while the two solid lines are the readings for the concrete cylinder. For both pairs of thermocouples in the concrete and in the kiln air there was no more than a 30°C temperature difference within the pairs on average. As can be seen in the figure, that there is some difference between the core temperature of concrete cylinder and air temperature inside the kiln, particularly for the 200^{0}C and 400^{0}C temperature profiles. This is due to the heat capacity of the concrete specimens and the rate at which they are able to absorb heat. However, the difference between kiln temperature and cylinder at 600^{o}C and 800^{o}C gradually reduced. The difference between the temperatures in the kiln air and in the concretes were to ensure rapid heating which would better reflect the heating within a house fire. Up until 500°C, the kiln was able to maintain a reasonably constant heating rate, there after the heating rate decreased.

3.2 Residual compressive strength

The effects of different elevated temperatures on residual compressive strength of fly ash concretes are shown in Fig. 5. The effects of different cooling regimes on the residual compressive strength of above concretes are also shown in the same figure. It can be seen that in the case of slow cooling, the residual compressive strength of all concretes increased at 200^{o}C except concretes containing 10% and 30% fly ash (Fig. 5a). With further increase in elevated temperatures, the residual compressive strength of all concretes decreased at all elevated temperatures. However, in the case of rapid cooling the scenario is different, where no such increase in residual compressive strength is observed at 200^{o}C and even at other elevated temperatures the residual compressive strengths are lower than those of slow cooling (Fig. 5b).

3.2.1 Residual compressive strength at 200^{o}C

It can be seen in Figs. 6-7 that the residual compressive strength of most fly ash concretes due to slow cooling is higher than that of rapid cooling. The possible reasoning behind the strength gain at this temperature range is the formation of tobermorite. Tobermorite is a calcium silicate hydrate mineral that is formed from a reaction between unhydrated fly ash particles and lime at elevated temperatures [11]. Tobermorite improves the adhesion between the aggregates and the paste.

There was considerable difference between concrete samples that were slowly and rapidly cooled. As can be seen in Fig. 7, samples that were rapidly cooled exhibited higher strength losses than slow cooling process. One justification as to why such a difference occurred in this temperature range is thermal shock. Thermal shock is caused by a sudden fluctuation in temperature due to quenching of water on concrete specimens [12]. Thermal shock induces tensile stress inside the concrete promoting cracking, and as clearly shown in Fig. 7, results in a fairly significant reduction in strength.

3.2.2 Residual compressive strength at 400^{o}C, 600^{o}C and 800^{o}C temperatures

As presented earlier, the concrete specimens experienced significant drop in compressive strength between 400^{o}C and 800^{o}C for both cooling methods. However, rapid cooling is more severe than slow cooling, where residual compressive strength is reduced between 26 and 34% at 400^{o}C, between 48 and 59% at 600^{o}C and between 80 and 87% at 800^{o}C (see Fig. 7). As discussed, the thermal shock due to sudden fluctuation in temperatures due to rapid cooling could be a cause for such significant drop in the residual strength capacity.

Thermal shock is not the sole reason for the strength loss in those temperatures, the development of thermal incompatibilities is also a contributing factor. Thermal incompatibilities refer to the different rates that a concrete's constituent either expand or contract from the exposure to different elevated temperatures [5]. When subject to 200˚C, the 10mm and 20mm aggregates expand at a slow rate and similarly so does the fly ash paste. As both constituents are expanding at a similar rate there is no real stress from thermal incompatibilities. And due to this reason no significant reduction in residual compressive strength of fly ash concretes are observed at 200^{o}C for both cooling methods. At 400˚C and beyond, the 10mm and 20mm aggregates continue to expand at a similar rate, but the fly ash paste, however, stops expanding and starts rapidly contracting due to further evaporation of moisture. This difference in volumetric changes creates an incompatibility between aggregates and paste causing micro cracking between the two. The formation of this micro cracking damages the cementitious bonds between paste and aggregate and reduces compressive strength.

The suspected reasoning behind this sharp drop in residual compressive strength is also due to the dehydration of calcium hydroxide ($Ca(OH)_2$). $Ca(OH)_2$, although not found in fly ash, is abundant in cement and as the cementitious paste is comprised of mostly OPC its dehydration will be a likely contributing factor. As found in research by Mendes, et al. [10], when concrete passes the critical temperature level of 400˚C $Ca(OH)_2$, which is the main hydrate in cementitious paste, dehydrates into calcium oxide (CaO). This change of phase causes the paste to shrink and crack. Furthermore, when subject to rapid cooling, CaO rehydrates rapidly back into $Ca(OH)_2$ which causes Dissociation due to re-expansion of OPC paste. Dissociation is essentially the splitting of one ionic compound into smaller particles reducing strength even further [13].

Total evaporation of free water occurs as exposure temperatures increases. Further loss of chemically bound water also occurs and this bound water evaporates when concrete is exposed to extreme high temperatures [7]. The evaporation of the remaining water in the concrete not only induces more micro cracking but it is also the potential reason why, during testing, the concrete subject to 800˚C crumbled into powder. In order to ensure the repeatability of the measured calculated strengths standard deviations (SD) are also calculated and listed in Table 3. Generally a smaller SD represents that the results are very close to the mean. The larger the SD the more variance in the results. By looking into the SD values of all concretes it can be seen that they are generally low in the range of 0.43 to 2.77 and hence, ensure their repeatability.

3.3 Cracking behaviour

Figs. 8 and 9 show the effects of different cooling methods on cracking behaviour of ordinary concrete and fly ash concretes containing 10% and 20% fly ash at various elevated temperatures. When subjected to 200˚C, irrespective of cooling rate and fly ash content, there was no apparent sign of cracking in any of the concrete samples. When exposed to temperatures of 400˚C, tiny hairline cracks no greater than 20mm long began to appear. These hairline cracks were inconsistent throughout the specimen making a common trend unpredictable. At 600˚C exposure temperature, obvious cracking formed on the surfaces of all three concrete specimens. It was at this temperature range where the effect of cooling rate on cracking started to become evident. Concrete samples that were slow cooled produced hairline cracking similar to that produced in the 400˚C range, whereas concrete that was subject to rapid cooling experienced cracking that appeared to not only be wider but have an increased length, 30-40mm. Consistent with the results found by Arioz [14], concrete exposed to 800˚C was noted to experience a further increase of cracking compared to that exposed to only 600˚C. The degree of cracking was so much more significant that the cracks started to join up and completely span around the entire surface area of the cylinder. Although control samples containing no fly ash exhibited large amounts of cracking, they were visibly smaller than cracking patterns formed on the surface of concrete containing fly ash. It was noted that the 800˚C temperature was the only range that showed a variance in the level of cracking between the control and fly ash concrete. When comparing Figs. 8 and 9 one can see that cooling rate did have an effect on the severity of cracking at 800˚C. Slow cooling, although inducing a large amount of cracking, did not induce the size of cracking exhibited by the rapidly cooled samples. It was noted that the cracking formed on the surface of the rapidly cooled samples was approximately 1mm wide, significantly wider then that formed on the slow cooled concrete.

3.4 Thermal and microstructural analysis

TGA/DTA and XRD analysis were performed to study the thermal stability and changes in hydration phases of mortar matrix of different fly ash concretes when exposed to elevated temperatures. Figs. 10 and 11 show the TGA and DTA analysis results of mortar samples of fly ash concretes, respectively. Results show that all fly ash matrixes exhibited sharp decrease in mass (about 10-12%) before about 150^oC and the lower the fly ash contents the more is the mass loss. The decrease in mass is peaked at around 100^oC as indicated by the DTA curve in Fig. 11. This is attributed to the dehydration of chemically bound water from the CSH gel. The result is quite similar with that of pure cement matrix where researchers reported about 6-8% mass loss around that temperature [14, 15]. The slightly high mass loss in fly ash matrix can be attributed to the formation of more CSH gel due to pozzolanic reaction of fly ash in those concretes during 56 days wet curing. At other higher temperatures, such after 400^oC and 800^oC mass loss is also observed, however, not at that high rate, which is also evident from the peaks in DTA curves in Fig. 11. The drop in mass at those two temperatures can be attributed to the decomposition of $Ca(OH)_2$ and $CaCO_3$ in the matrix [16].

XRD results of concretes containing 20% and 40% fly ash after exposure to 800^oC are shown in Fig. 12. The effects of slow and rapid cooling are also shown in the same figure. The quantitative phase abundance analysis of XRD results is also shown in Table 4. A number of potential crystalline phases are identified in the samples. The results show that after exposing to 800^oC temperature Portlandite [$Ca(OH)_2$] and Quartz [SiO_2] phases in both concretes are decreased at both cooling methods. The reduction of those phases is high for concrete containing 40% fly ash. This indirectly indicates that SiO_2 reacted with $Ca(OH)_2$ and formed additional CSH hydration products in fly ash concretes. However, the measured compressive strength results do not agree with this observation. Moreover, new peak of calcium silicate (Ca_2SiO_4) is formed in both concretes at around 2θ angle of 32^o after exposed to 800^oC for both cooling. This intensity is slightly higher in slow cooling than rapid cooling.

The formation of calcium silicate phase indicates the decomposition of CSH at elevated temperatures.

3.5 Comparison of experimental results with prediction

Eurocode EN1994:2005 [17] provides guideline to predict the compressive strength of OPC concretes at different elevated temperatures when they are exposed to fire. In this study, the measured compressive strength of fly ash concretes and OPC concrete at various elevated temperatures are compared with that predicted by the Eurocode EN1994:2005 and are shown in Fig. 13. It can be seen in the figure that the measured compressive strengths of OPC concrete at various elevated temperatures are very close to the predicted values except at 200 and 400^{o}C due to slow cooling. In the case of fly ash concretes, it is observed that the prediction by Eurocode EN1994:2005 is valid for all temperatures and cooling regimes, where the reduction of experimentally measured strength follows the same trend to that predicted by the code. Based on this present study, it can be concluded that the Eurocode EN1994:2005 can be used to predict the compressive strength of fly ash concretes with reasonable accuracy for all temperatures upto 800^{o}C.

4 CONCLUSION

This paper presents the results on the effects of slow and rapid water cooling on the residual compressive strength of ordinary concrete and fly ash concretes when subjected to various elevated temperatures. Based on experimental results the following conclusions can be drawn:

- Concrete containing 10% fly ash exhibited similar reduction (about 8-9%) to its ambient compressive strength when subjected to both slow and rapid cooling after exposure to 200^{0}C. However, at other elevated temperatures, the residual compressive strength of 10% fly ash concrete is lower due to rapid cooling than slow cooling. The concrete containing 20% fly ash exhibited about 9% and 30% less residual compressive strength at 200^{0}C and 400^{0}C, respectively, when subjected to rapid cooling than slow cooling. Surprisingly, at 600^{0}C and 800^{0}C this concrete showed about 5% and 12% higher residual compressive strength due to rapid cooling than slow cooling. The concretes containing high fly ash contents, such as 30% and 40%, showed higher reduction in residual compressive strengths at all elevated temperatures for rapid cooling compared to slow cooling. It was also observed that at all elevated temperatures the difference in residual compressive strengths between rapid and slow cooling decreased with increase in fly ash contents.

- Cracks were occurred sporadically over concrete samples after being exposed to temperatures ranging from 400˚C to 800˚C. Samples that were slowly cooled developed smaller cracks than those rapidly cooled. At 800˚C, all fly ash concretes that were exposed to rapid cooling showed the most severe cracking. Results also showed that existing Eurocode methods to predict the compressive strength of ordinary concrete subjected to elevated temperatures can still be applied for concretes containing fly ash.
- XRD results revealed new calcium silicate peaks in concretes containing 20% and 40% fly ash after exposure to 800ºC and different cooling indicating the decomposition of CSH due to heating. The reduction of $Ca(OH)_2$ and SiO_2 peaks in XRD results indicates the decomposition of above two compounds in hydration products and unreacted fly ash particle, respectively. The TGA/DTA results show that the thermal stability of concrete containing high fly ash content is higher than that containing low fly ash.

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