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An Emphasis Study on Residual Engineering Properties of Fly Ash Concrete Elevated Temperature

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ABSTRACT

The paper examines the properties of fly ash as partial cement replacement in normal strength concrete at ambient and elevated temperature. The properties of fly ash class F was chemically investigated. Behaviour of concrete containing fly ash subjected to elevated temperature 200 ºC up to 600 ºC at temperature rate of 2.78 ºC/min is investigated mechanically and microstructurally. Physically, fly ash concrete changed colour to brown at 400 ºC and to whitish grey at 600 ºC, which is similar to normal concrete. At 600 ºC, the residual compressive strength of fly ash concrete was slightly higher than normal concrete with a strength reduction of 13.18% and 45.22%, respectively. At 400 ºC, fly ash concrete has better splitting tensile strength than normal concrete with strength reduction of 28.81% and 43.26%, respectively. Thus, the use of fly ash as cement replacement in fly ash concrete improves concrete performance after exposed to elevated temperatures. Based on Field Emission Scanning Electron Microscopy (FESEM) and Energy Dispersive X-Ray (EDX), fly ash concrete performed better thermally proven by the formation of ettringite. In conclusion, the utilization of fly ash as a replacement for Ordinary Portland cement (OPC) at elevated temperatures is a good innovation for concrete performance.

Keywords: *Concrete; Fly Ash; Chemical Properties; Mechanical Properties; Elevated Temperature*

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Introduction

The construction industry has invoked innovations in concrete mixes through cement replacement using environmental and economic friendly alternatives as well as by-products and waste materials such as fly ash, silica fume, slag and fine limestone which act as cementitious materials [1]–[4] Adding supplementary cementitious materials into concrete mixture lead to microstructure refinement, resulting in increment in mechanical strength [5], [6]. The utilization of coal combustion by-products, namely fly ash, can be an alternative to industrial resources and improve concrete properties [7]. In Malaysia, Class F fly ash has been widely used as addition or replacement to cement and concrete products, structural fills, cover materials, road and pavement. Disposal processes in landfills require proper handling as fly ash particles are considered highly containimating due to the enrichment in toxic trace elements that significantly impact pozzolanic reactivity [8].

Fly ash generated from the combustion of coal for energy production is recognized as an environmental pollutant. Fly ash is also known as fuel ash and millions tons of ash and related by-products were generated in 2018 - 2019 [9]. The current annual production of coal ash worldwide is estimated to be around 600 million tons, with fly ash constituting about 75 – 80% of total ash production. Based on its chemical composition, two general classes of fly ash can be defined: low-calcium fly ash (Class F) produced by burning anthracite or bituminous coal and high-calcium fly ash (Class C) produced by burning lignite or sub-bituminous coal. Class F fly ash is categorized as a normal pozzolan containing silicate glass, modified aluminium and iron [10] which formed cement in the presence of water (pozzolanic reaction). Due to the pozzolanic reaction, the strength increment for fly ash concrete continues for a longer period of time than conventional concrete [11]. Figure 1 shows the particle size distribution of fly ash (FA), ground granulated blast furnace slag (GGBS), silica fume and Ordinary Portland cement (OPC) [12]. It is shown that fly ash and GGBS are among the finest particle size distribution, followed by silica fume and OPC.

However, microstructure refinement also lead to a reduction in permeability in concrete mixture. The impermeabile microstructure of fly ash concrete (FC) at elevated temperature exposure results in an incremental built-up in pore pressure, thereby intensifying crack propagation [13]. Thus, fly ash concrete is prone to spalling effect. The majority of studies on fly ash concrete behaviour at high temperature have been slanted towards some typical mechanical features (compressive, flexural and tnsile strength), overlooking other properties of concrete. Previous studies have considered the utilisation of fly ash as cement replacement in geopolymer concrete [1], [14]–[16]. Jiang found 30% fly ash replacement of cement in normal strength concrete as optimum for the concrete's porosity [17]. Fly ash used as cement replacement occupies a high volume in concrete and significantly influences its behaviour at elevated temperature. Ashish studied the effect of Class F fly ash on the durability of concrete at ambient temperatures to find that the compressive strength of concrete cylinder at 28 days curing sharply decreased [11]. However, for 30% and 40% fly ash, compressive strength of the concrete increased gradually up to 180 days and at 360 days curing the results showed a constant compressive strength. Khan [18] studied the performance of high-volume fly ash concrete after exposure to elevated temperature with 0%, 25% and 40% fly ash replacement. It was found that an optimum level of cement replacement with fly ash is 40% based on residual compressive strength of concrete after a single heating-cooling cycle of elevated temperature ranging from ambient to 400 ºC at an interval of 200 ºC.

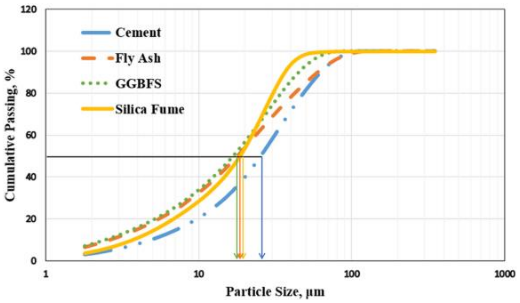

Figure 1: Particle size distribution of fly ash (FA), rice husk ash (RHA) and Ordinary Portland cement (OPC) [12]

Wang studied the thermal conductivity of 30% fly ash concrete to elevated temperatures and an effect of micro-environment relative humidity of 20%, 45%, 75%, and 100%. For the micro-environment relative humidity of 45%, the thermal conductivity of ordinary concrete was constantly greater than fly ash concrete; however, at 100% humidity, the thermal conductivity of fly ash concrete (1.603 W/m.K) was higher than normal concrete (1.599 W/m.K) [19]. Rahel [20] studied the fire resistance of high-volume fly ash mortars with nanosilica additions and found that high strength mortars had an equivalent residual strength before and after exposure to elevated temperatures as high-volume fly ash and colloidal nanosilica replacement concretes. This was proven by Field Emisson Scanning Electron Microscope (FESEM) analysis, with magnifications up to 20 KX. After exposure to 400 ºC, an increase in calcium silicate hydrate was noticed. Thus, from all recent studies, fly ash is expected to offer better performance than Ordinary Portland cement (OPC) at elevated temperatures. In this paper, the utilisation of fly ash as a partial cement replacement for concrete after exposure to elevated temperature was investigated. This paper presents the influence of elevated temperature on pozzolanic reaction and optimum temperature for pozzolanic reaction for conrete containing 30% replacement of fly ash. In addition, this paper presents further study on microsturctural analysis for optimum temperature and destructive temperature for fly ash concrete after exposure to elevated temperature based on mechanical properties of fly ash concrete.

Methodology

Materials

In this study, fly ash and ordinary Portland cement (OPC) were used as a binder concrete ingredient with a specific gravity of 3.15 and 2.30, respectively. For this study, fly ash was obtained from Tanjung Bin Power Plant, Kukup Johor. For coarse aggregates, portions less than 10 mm and greater than 5 mm were used in accordance with BS EN 933 (2012)[21]. The river sand was composed of portions passing 4.75 mm and a density of 2650 kg/m³ in Ordinary Portland cement (OPC) was used in this study in both normal concrete and a percentage in fly ash concrete mix. The tap water used in this concrete mix followed the requirements of BS3148:1959, which means it was free from contaminants and particles.

Mixture proportions

In the experiment, to achieve 30 MPa concrete strength, a water-cement ratio of 0.54 was used. Table 1 shows the mixture proportions of the concrete speciemens. The fly ash concrete replaced 30% of OPC with fly ash.

Table 1: Mixture proportion of concrete specimen

Ratio of Fly Ash (%)	Cement (kg/m³)	Fly Ash (kg/m³)	Water (kg/m³)	Sand (kg/m³)	Gravel (kg/m³)	Fly Ash replacement (%)	Water cement ratio
0	431	-	233	619	1054	0	0.54
30	301.7	129.3	233	619	1054	30	0.54

Experimental program

The chemical properties of fly ash were determined through X-ray fluorescence (XRF) testing in accordance with ASTM E1621-13 (2013) [22]. Fly ash was tested under XRF to determine its chemical composition. The machine irradiated samples using an intense X-ray beam from a radioisotope source. This machine used an energy-dispersive system with elements ranging from sodium to uranium (Na to U). The fly ash sample was placed in the machine for 5 minutes.

The behaviour of fly ash in concrete at ambient temperatures was studied, using compressive, splitting tensile and flexural strength tests after 7, 14, 28, 56, and 120 days of curing. The optimum percentage of fly ash cement replacement in the cement concrete mix was determined using porosity, chloride diffusivity and thermal properties [16], [21]-[22]. Two batches of concrete consisting of 0 percent (normal concrete) and 30 percent (fly ash concrete) fly ash were casted. Each batch consisted of 18 cubes for compressive strength testing, 18 cylinders for splitting tensile test and 18 prisms for flexural strength test with dimensions of 100 mm x 100 mm x 100 mm, 100 mm x 200 mm and 100 mm x 100 mm x 500 mm, respectively.

At elevated temperatures, residual mechanical property tests such as compressive, splitting tensile and flexural strength tests were conducted for specimens after 28 days of curing. Two batches of normal concrete and fly ash concrete consisting of 30 cubes for compressive strength testing, 30 cylinders for splitting tensile testing and 30 prisms for flexural strength testing with dimensions of 100 mm x 100 mm x 100 mm, 100 mm x 200 mm and 100 mm x 100 mm x 500 mm, respectively, were used. All specimens were exposed to elevated temperatures of 100 ºC – 1000 ºC (100 ºC increments) with a slow heating rate of 2.78 ºC/min in an electric furnace at Structure Laboratory Universiti Teknologi Malaysia as shown in Figure 2. After the heating process, the samples were left to cool for 24 hours. Then, mechanical property tests (compression test, tensile test and flexural test) were conducted on the specimens.

Figure 2: Laboratory electric furnace in Universiti Teknologi Malaysia

Field Emission Scanning Electron Microscope (FESEM) images were taken at magnifications up to 20 KX to study the micro-structure of the fly ash concrete materials after elevated temperature exposure. Figure 3 shows the equipment used to analyze the FESEM and Energy Dispersive X-ray (EDX) data.

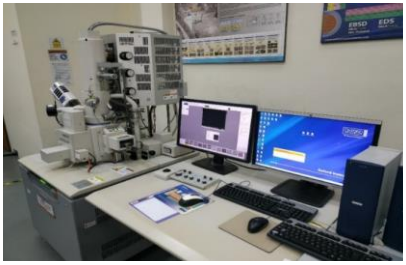

Figure 3: FESEM equipment at Universiti Teknologi Malaysia (UTM)

Casting and curing procedure

Specimens were casted with three layers of vibration to ensure no voids were left in the concrete. After casting, the specimens were left at ambient temperature for 24 hours. On the next day, the samples were demoulded and cure for the 7, 14, 28, 56, and 120 days for compressive testing. For the residual mechanical property test, the samples were cured for 28 days before being exposed to elevated temperatures.

Results and Discussion

Chemical properties of fly ash

Figure 4 summarises the outcomes for the X-ray defractions (XRD) test for fly ash. It can be noticed that the most common elements in fly ash were silicon dioxide, aluminium trioxide, iron (iii) trioxide, and calcium oxide. Generally, silicon dioxide in fly ash produces a calcium silicate hydrate (C-S-H) gel when reacted with calcium hydroxide, which is a product from first hydration process as shown in Equation (1), in the pozzolanic reaction as shown in Equation (2) [25].

Hydration Process;

$$Cement + H_2O \rightarrow C\text{-}S\text{-}H\ gel + Ca(OH)_2 \quad (1)$$

Pozzolanic Reaction;

$$SiO_2 + Ca(OH)_2 \rightarrow C\text{-}S\text{-}H\ gel \quad (2)$$

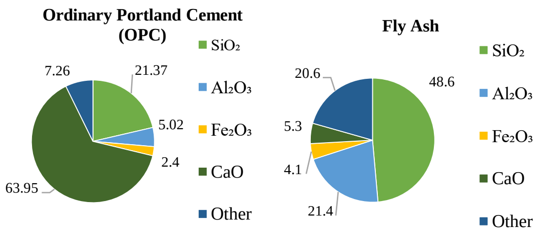

Figure 4: Chemical composition of fly ash and ordinary portland cement

Compressive strength at ambient temperature

Figure 5 displays the compressive strength for all specimens after 7, 14, 28, 56 and 120 days of curing. A increase in fly ash concrete compressive strength was noticed when replacing 30 percent OPC with fly ash. After 7 ,14, 28 and 56 days, the decrease in compressive strength was 33.72% , 32.54%, 15.68% and 1.74%, respectively. After 120 days, an increase in compressive strength of 14.73% was shown and can increase in further curing period. However, the increase in compressive strength of fly ash concrete, known as latent strength, required long time period. This is attributed to the pozzolanic reaction showed in Equation (2), which requires calcium hydroxide ($Ca(OH)_2$) which is a product of the hydration process shown in Equation (1). Hence, fly ash concrete has better performance as its strength increases over time. The increase in compressive strength in fly ash concrete is due to fine fly ash particles filling voids within the concrete, thus making the final product more durable and compact.

Table 2 presents summarized data on fly ash concrete based on previous studies and results from research study at ambient temperature [4], [20], [26], [27]. Based on data, a study can be done in order to improve the concrete containing fly ash. This is because an additional material such as nano silica and recycle aggregate seems to improve concrete properties.

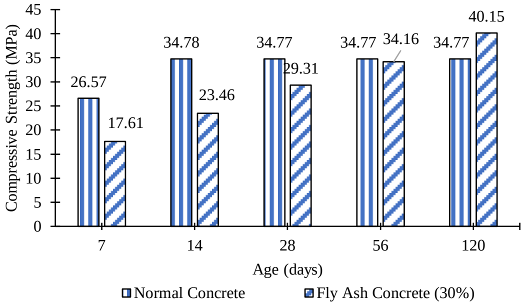

Figure 5: Compressive strength of normal concrete and fly ash concrete

Table 2: Summary of mechanical properties at ambient temperture

Concrete Specimens	Compressive Strength (MPa)	Splitting Tensile Strength (MPa)	Flexural Strength (MPa)	Residual Porosity (%)	References
Fly Ash (40%)	20.76	1.85	2.41	-	[26]
Fly Ash (30%) + Recycle Concrete Agregate	45.1	-	-	10	[27]
Fly Ash (25%)	31.0	2.70	-		[4]
Fly Ash + Nano silica	56.28	-	7.43	-	[20]
Fly Ash (30%)	33.9	2.98	4.35	2.02	Experimental Test

Elevated temperature test

All specimens were exposed to temperatures of up to 1000 ºC with a slow heating rate of 2.78 ºC/min. Three cubes, three cylinders and three prism samples were used for each 100 ºC increment. This paper presents physical properties such as colour changes, cracks, and spalling effects. Moreover, mechanical properties such as residual compressive strength, residual splitting tensile strength and residual flexural strength are presented in this section.

Physical changes in fly ash concrete subjected to elevated temperature

Table 3 shows the physical changes in 30FC and NC after being subjected to elevated temperatures. The colours and surface texture of the 30FC and NC specimens remain unchanged until the temperature of the specimens reached 200 ºC due to a chemical composition that contained siliceous aggregates. At 300 ºC, the colour of fly ash concrete became yellowish while the normal concrete turned to reddish or brownish colour with no observed crack. This change in colour from normal-grey to reddish or brownish was due to the oxidation of iron contained in river sand [28]. Upon further heating at 400 ºC, the colour of fly ash concrete changed to brownish while normal concrete turned to a whitish grey colour with no observed cracks. This change in colour to whitish grey at 400 ºC was due to the decomposition of C-S-H gel in cement paste [29]. A change in colour to whitish grey for both fly ash concrete and normal concrete at 500 ºC and 600 ºC without observed crack was due to the decomposition of C-S-H gel in cement paste [29]. Upon further heating up to 700 ºC, both fly ash concrete and normal concrete turned a strong whitish grey. It was observed that fly ash concrete experienced a massive spalling effect from fly ash, while normal concrete experienced crack and explosion. The formation of cracks and explosions was because of the dissociation of elements within the concrete, thereby increasing pore pressure and particle stress due to a slow heating rate [29]–[31].

Residual Compressive Strength

Figure 6 displays the relative reduction of the residual compressive strengths of all specimens after 28 days of curing at different elevated temperature. At ambient temperatures, the compressive strength of fly ash concrete was lower than normal concrete. However, at 100 ºC, the residual compressive strength of 30FC increased by 29.48% while NC decreased by 3.19%. Upon further heating to 400 ºC, the residual compressive strength of 30FC and NC decreased by 4.01% and 4.30%, respectively. Fly ash concrete and normal concrete experienced a sudden drop in strength when exposed to 600 ºC with a reduction of 13.18% and 45.22%, respectively. At an 100 ºC, the residual compressive strength of 30FC increased by 29.48% since this elevated temperature accelerated the alkaline activation of calcium hydroxide ($Ca(OH)_2$) [32], which accelerated the pozzolanic reaction and speed up the formation of the reaction product. Meanwhile, at 200 ºC, the reduction of strength of concrete was due to the dehydration of water from the calcium silicate hydrate (CSH) gel [33].

Table 3: Summary of colour change with respect to tempurature exposure

Temperature / Sample	100 ºC	200 ºC	300 ºC
Fly Ash Concrete	 (1:100)	 (1:95.2)	 (1:100)
Normal Concrete	 (1:95.2)	 (1:100)	 (1:100)
Temperature / Sample	**400 ºC**	**500 ºC**	**600 ºC**
Fly Ash Concrete	 (1:95.2)	 (1:100)	 (1:95.2)
Normal Concrete	 (1:95.2)	 (1:100)	 (1:100)
Temperature / Sample	**700 ºC**		
Fly ash Concrete	 (1:80)	Spalling	
Normal Concrete	 (1: 80)	Crack and explode	

The results show that 30FC offered slightly better residual compressive strength at elevated temperatures than NC especially after exposed to 100 ºC. Fly ash performs better under elevated temperatures as it is more stable and loses strength slower than OPC [33]. Hence, further microstructural study on fly ash concrete after exposure to elevated temperature at 100 ºC is investigated and presented in next topics.

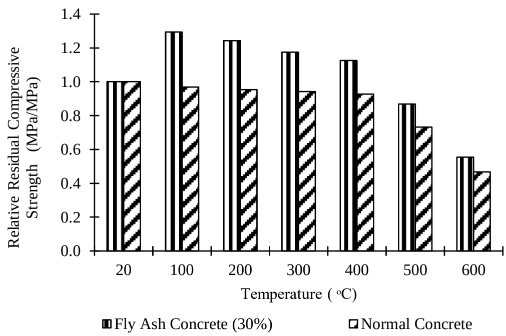

Figure 6: Relative residual compressive strength of normal concrete and fly ash concrete at elevated temperatures

Residual splitting tensile strength

Splitting tensile tests were conducted on fly ash and normal concrete after exposure to elevated temperature as shown in Figure 7. From the results, it is observed that at 100 ºC the strength of 30FC increased by 1.04%, while NC decreased by 8.87%. At 200 ºC, the residual splitting tensile strength of 30FC began to gradually drop, unlike NC. Upon further heating to 400 ºC, the residual splitting tensile strength of 30FC and NC decreased by 28.81% and 43.26%, respectively. At 600 ºC, the residual splitting tensile strength of 30FC and NC decreased by 78.42% and 73.41%, respectively. The results show 30FC offers improvement in residual splitting tensile strength at various elevated temperatures compared to NC.

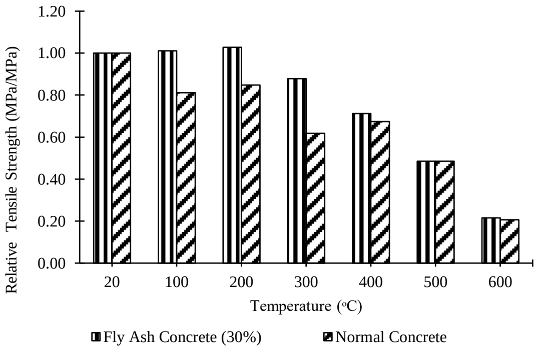

Figure 7: Relative of residual splitting tensile strength of concrete at elevated temperatures

Residual flexural strength

The residual flexural strength of all specimens is shown in Figure 8. The results show that the flexural strngth of the concrete specimens gradually decreased when exposed to elevated temperature. At 100 ºC, the residual flexural strength of 30FC and NC decreased by 12.10% and 12.74%, respectively. At 200 ºC, the reduction of strength for 30FC is 12.10% since the concrete experienced the dehydration of water from CSH gel, causing reduction of residual flexural strength. Upon further heating to 400 ºC, the residual flexural strength of 30FC and NC decreased by 45.13% and 22.38%, respectively. Above 400 ºC, the flexural strength dropped drastically. For flexural specimens, since the exposure surface was larger than other specimens, the specimens were at risk of explosive spalling due to steam pressure from water released during heating combining with compressive thermal and static stresses at the fire exposed surface [30]. Hence, the results show both of the concrete specimens offer slightly different residual flexural strengths when exposed to elevated temperatures.

Microstructural properties

Figure 9 shows the FESEM results for Fly ash concrete (30FC) at ambient temperature after 28 days of curing in tap water. The image illustrates the formation of gaps within concrete and fly ash particles and most of fly ash particles in the figure were still smooth. This means that the reaction for fly ash particles had not yet started compared to Figure 9 and Figure 10, in which both of the fly ash particles in the images showed rough surface. It is clear in the image that there is a gap around the fly ash particle as fly ash required a certain period to undergo pozzolanic reactions (second hydration process).

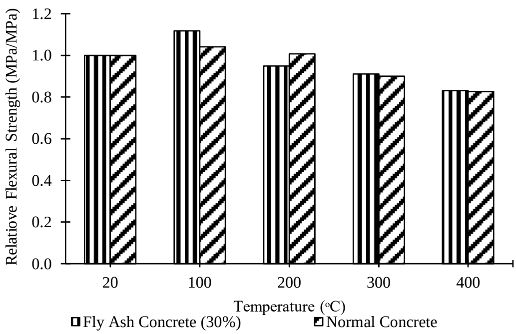

Figure 8: Relative residual flexural strength of concrete at elevated temperature

It is clearly shown in Figure 10 that microstructure image of normal concrete at ambient temperature seems to be denser with products of hydration process (ettringite, calcium hydroxide and calcium silicate hydrate) after curing for 28 days. Figure 11 shows the results of 30FC at ambient temperature after 28 days of curing using standard curing, in which the specimens were cured in a room at 20 ± 2 °C and more than 95% relative humidity [17]. Most fly ash particles in the image continued to present smooth surface. It means that most fly ash particles had not reacted yet [17].

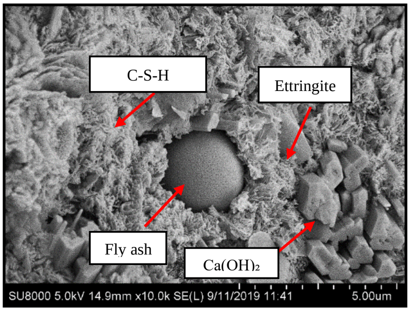

Figure 9: FESEM morphology after 28 days of curing at ambient temperature of fly ash concrete (30FC)

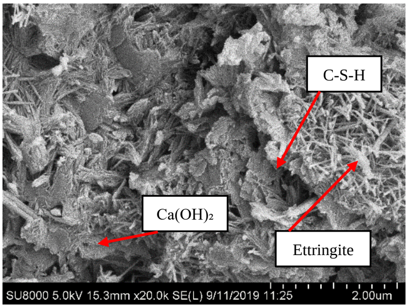

Figure 10: FESEM morphology after 28 days of curing at ambient temperature of normal concrete

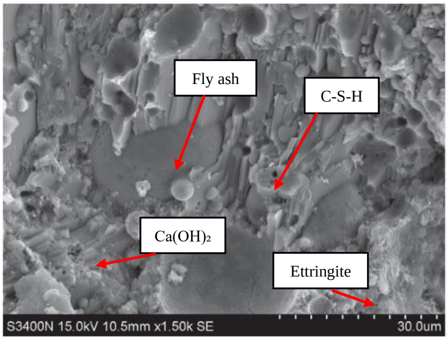

Figure 11: SEM morphology for fly ash concrete cured under standard curing procedure at ambient temperature [17]

Figure 12a shows the results of 30FC after exposure to 400 ºC; the formation of microcracks and voids within the concrete along ettringite and fly ash particle was observed. Fly ash particles exhibit a rough surface and there is irregular flake around the particle indicating reaction had occurred. The images ilustrate the formation of crystal calcium-silicate-hydrate (C-S-H) and calcium hydroxide ($Ca(OH)_2$) through cement hydration and second hydration (pozzolanic reaction) from fly ash.

Figure 12b shows morphology of pozzolanic mortar contained high volume fly ash and nano silica after exposed to 400 ºC temperatures [20]. An increase in calcium silicate hydrate can be noticed and rough surface of fly ash particles indicated high temperature can stimulate the acceleration of pozzolanic reaction of fly ash and silica to convert calcium hydroxide to calcium silicate hydrate. Then, the formation of microcracks and voids within the concrete along ettringite and fly ash particle was observed as most of the ettringite and calcium hydroxide destruct because of dehydration process.

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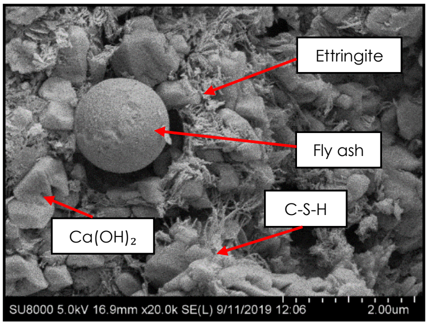
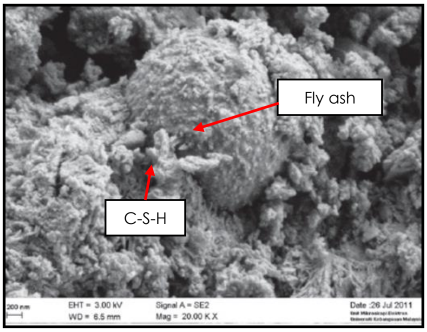
In general, the formation of C-S-H was the predominant product for concrete strengthening. The production of $Ca(OH)_2$ from hydration processes enables pozzolanic reactions for second hydration. The formation of ettringite was observed in 30FC at ambient temperature and after exposure to elevated temperature (100 ºC and 400 ºC). Ettringite was formed because of gypsum and other sulphate compounds reacting with calcium aluminate in the cement [31]. Generally, ettringite forms within 7 days of hydration and disappears when cement hydration continues beyond 7 days [34]. Then, it will destruct due to dehyration process at higher temperature exposure.



(a)



(b)

Figure 12: (a) FESEM morphology 400 ºC temperatures for 30FC (fly ash contained specimens) and (b) SEM morphology for fly ash + nano silica contained specimens after exposed to 400 ºC temperatures [20]

Energy dispersive X-Ray

Figures 13a and 13b summarize the outcomes of EDX analysis for specified morphology from FESEM test of fly ash concrete (30FC) before and after exposure to 100 ºC. As at 100 ºC, the concrete gains significant increment in strength based on compression and splitting tesntile tests, which means pozzolanic reaction is occured rapidly at 100 ºC. This can be proved using EDX analysis. In general, Energy Dispersive X-ray (EDX) analysis was performed to gather element and quantitative composition information from the gathered images. Based on Figure 8, the calcium (Ca) content of the concrete increased by 10.10% after the concrete was exposed to elevated temperatures up to 100 ºC. Calcium is a product of the hydration process and as calcium content increased, the ability of elevated temperatures to activate pozzolanic reactions in hydration process also increased. Silica (Si) content was reduced by 12.01% as a result of the hydration process. In additon, based on morphology images shown in Figure 8, fly ash particle of fly ash concrete after exposure to 100 ºC illustrated a rough surface. It indicates that higher accumulated temperature can promote the reacton of fly ash particles even at same age of curing. It is known that fly ash particles start to undergo reaction after 28 days curing process [17]. Hence, a reduction in silica content shows improvements in the hydration process.

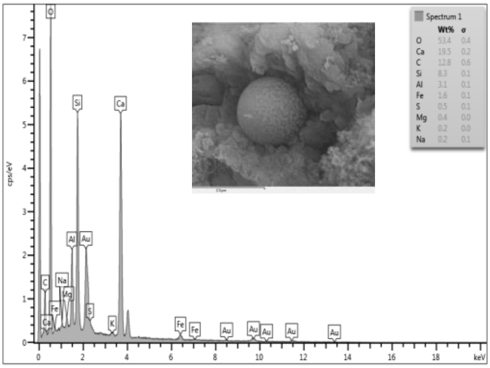


Figure 13: (a) EDX results for 30FC based on morphology image gathered from FESEM testing after exposure to 27 ºC

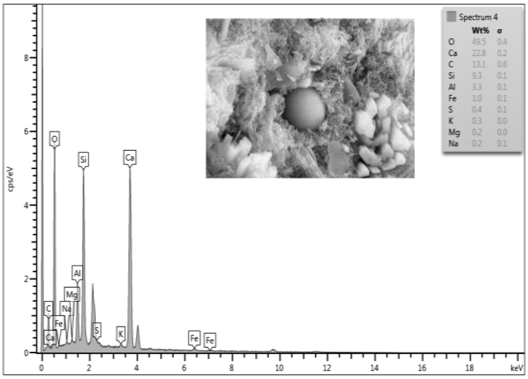


Figure 13: (b) EDX results for 30FC based on morphology image gathered from FESEM testing after exposure to 100 ºC

Conclusion

The properties of 30% replacement fly ash in cement in and normal strength concrete were successfully examined at ambient and elevated temperatures. This concrete can be considered cost-effective since fly ash is a waste by-product of coal power plants. Due to its similar properties, this product can reduce the use of cement and minimize environmental problems. As fly ash shows better performance at elevated temperatures, it is recommended for concrete in fire structures engineering. In conclusion, fly ash can be potentially used as a 30 percent cement replacement for concrete production. The residual compressive strength of 30FC increased by 17.32% while NC decreased by 3.11% at 100 ºC elevated temperature exposure. Fly ash has a lower initial strength, but the incorporation of fly ash benefits conventional concrete in terms of later strength development. In addition, thermal load increased pozzolanic reactivity which increase development of bonding between concrete particles. Hence, the lower early strength for fly ash concrete will not give bad impact on the concrete fire performance. A similar trend was seen in a study conducted on 25% and 40% fly ash replacement on the OPC, where the rate of compressive strength development for fly ash concrete was higher compared to plain concrete with OPC [18]. The tensile splitting strength of 30FC increased by

1.04%, while NC decreased by 8.87% at 100 ºC which is similar trend as residual compressive strength.

FESEM morphology of 30FC after exposure to 100 ºC was compact and homogenous compared to exposure prior to elevated temperature. This result is supported by EDX analysis results where the calcium (Ca) content of the concrete increased by 10.10% after the concrete was exposed to elevated temperatures up to 100 ºC. Hence, it can be concluded that concrete containing fly ash experience an increment in mechanical properties after exposure to elevated temperature, which is supported by the FESEM morphology and EDX results.

Conflict of Interests

Nurizaty Zuhan received financial support provided by ZAMALAH Scholarship, Universiti Teknologi Malaysia (UTM). Mariyana Aida Ab. Kadir has received a research grant from Ministry of Education Malaysia: Grant Q.J130000.2522.12H44. The remaining authors have no conflicts of interest to declare.

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