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The Effect of Nanosilica in Compensating the Strength Loss Caused by Using High Volume Fly Ash in High Strength Mortars

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Abstract--- Fly ash has been widely used as a cement replacement in building construction for the purpose of reducing cost and the environmental impacts of cement however, using a high volume of fly ash results in a reduction in strength. In this research study, large volumes of cement have been replaced by fly ash, and nanosilica was used to compensate for the strength loss. The objective was to produce high strength mortars containing high fly ash volumes to reduce the amount of cement in high strength mortar mixes. The compressive and flexural strength results showed that replacing up to 35% of the cement with fly ash could enhance the strength after just 28 days, while at earlier ages, the strength reduces. Further replacement results in a significant reduction in strength. The incorporation of nanosilica compensates the strength loss caused from replacing cement by fly ash for all replacement percentage sat earlier ages, and results in a significant increase in strength at later ages. The maximum compressive strength of 91.75 MPa and flexural strength of 11.185 MPa were recorded by replacing 45% of the cement with fly ash and nanosilica. The micro-structural investigations of the systems by using SEM, XRD and TGA techniques supported the enhancement of strength by nanosilica, and the porosity tests showed the ability of nanosilica to refine the pore structure in the system.

***Index terms*---High volume fly ash, Nanosilica, strength, Microstructure, XRD, TGA, BET Porosity.**

I. INTRODUCTION

The most widely used construction material in the world is concrete. Annually, more than one meter cube per capita is produced [1]. This leads to an increasing demand of using Portland cement which its production is not only highly energy intensive, but is also an obvious contributor to the emission of carbon dioxide [2]. To produce 1 ton of Portland cement, approximately 2.5 tons of raw materials (including fuel) is needed, and this process produces approximately 1 ton of green-house gases [3], which primarily consists of carbon dioxide. For increasing the sustainability of concrete, replacing large amounts of cement with by-product materials is essential [4]. There are a vast amount of researches regarding the partial replacement of cement by supplementary materials [2, 5-8]. Among the supplementary materials, fly ash plays a dominant role due to its high performance in enhancing the properties of concrete in addition to its low price with respect to other supplementary materials.

Fly ash is a byproduct material produced from electrical power plants as a result of burning pulverized coal [9]. Using this material in concrete brings many advantages like; enhancing workability, durability and strength as well as lowering the price [4]. Using a high volume of fly ash in concrete return to early 1980s in Canada, where fly ash was used as a Portland cement replacement in percentages of 55% to 60% during the production process of Portland cement [2]. The national practice codes in UK recommends replacing 35% of the cement by fly ash [10], while the test method of ASTM C311-94a recommends only 20% replacement for the strength measurement of mortars [9]. Experimental researches showed that replacing up to 30% of the cement with fly ash results in a comparable strength or even an increase in strength [11, 12], while higher replacement percentages caused a significant reduction in strength [13, 14].

As the concrete technology develops, new materials and novel application of these materials are used in construction sector. One of the materials that recently have been used in concrete is nanomaterials. Nanomaterials are materials optimized at a nanoscale (1-100nm). At this scale, the classic theories of quantum mechanics are no longer applicable and a wide variety of surprising properties is possible [15]. In the last 10 years, a number of attempts were made in using nanomaterials in concrete. However most of these attempts remained in the research field, the only nanomaterial used in concrete as a construction purpose was nanosilica. Nanosilica has shown to be more effective in enhancing the strength of concrete [16]. Although one of the basic materials for producing high strength concrete is silica fume [9], as it enhances the concrete's strength, researchers have reported that nanosilica has superior performance to silica fume in enhancing the compressive strength of concrete[17, 18] while, its price is comparable to that of silica fume[19].Many researchers emphasized that nanosilica particles improve the microstructure by increasing the process of hydration and because of their high surface area; they increase pozzolanic activity more readily than performing as filler for stuffing calcium silicate hydrate structure [20-22]. The beneficial effects of nanosilica in improving the microstructure and strength qualifies it to be one of the most widely used nanomaterials in the construction sector[16].

The main hydration products from the reaction of water with cement are calcium silicate hydrate and calcium hydroxide; the former is responsible for the binding properties in cement paste. These materials are produced from the reaction of water with both tricalcium silicate and dicalcium silicate[23]. For detecting changes in the hydration products through the addition of pozzolanic materials, X-ray diffraction (XRD) and Thermal gravimetric analysis (TGA) are best methods [24, 25]. A well-established method for detecting the material's hydration is the porosity test. The hydration of the cement begins when it contacts water, and the hydration products occupy the voids that filled with water. The pores in the hydrated cement paste are of nano metric to micrometric scales [26].The size and distribution of these pore are highly affect durability and performance of concrete [27].

For explaining the obtained strength results in the present research, the authors focused on the SEM, XRD and TGA techniques for investigating the micro structural changes in the hydration products through the incorporation of nanosilica. Moreover, the Brunauer, Emmett, and Teller (BET) porosimetry technique was used for determining the pore size distribution of the mortars at sub nano-metric scales.

The main objective for this study is to produce mortars having high strength and containing high volumes of fly ash by using nanosilica. This approach addresses the increasing demand to produce sustainable and more environmentally friendly concrete. The present report focuses on the maximum quantity of cement to be replaced by fly ash and on compensating for the strength loss or even generating a higher strength by the addition of colloidal nanosilica.

II. EXPERIMENTAL STUDY

A. Materials

The cement used in this study was obtained from the Tasek Cement Company. The fly ash was collected from the Kelang power station near Kuala Lumpur, and it matches the class F fly ash according to ASTM C618-00:2000. The chemical composition of the cement and the fly ash is shown in Table 1. Colloidal nanosilica that contains 50% amorphous nanosilica having the average particle size of 50 nm, with a 1050 kg/m³ bulk density and a pH of 10 (obtained from Akzo Nobel Company) was used. The fine aggregate used in this study was natural siliceous sand with a fineness modulus of 2.65 and passing through a 4.75 mm sieve. The super plasticizer was a naphthalene formaldehyde sulphonate type under the trade mark of Sikament-NN.

B. Methods

Mixtures with three different fly ash levels 25%, 35% and 45% and three different (nanosilica + fly ash) levels of 2.5%+22.5%, 5%+30% and 37.5%+7.5% were casted next to the ordinary Portland cement mix. The fly ash and nanosilica were used as cement replacements. The mixes had a water/binder ratio of 0.4 and for fixing this rate in the mixes containingnanosilica, the water in the used colloidal nanosilica was reduced from the total mixing water amount. The ratio of cement to sand was 1:2.75. Details of the mix proportions are shown in Table 2.

Mixing of the materials was carried out according to ASTM C305-06. The ASTM C1437-07 standard was used for measuring the consistency for the mixtures using the flow table specified in ASTM C230/C 230M. The consistency of the mixes was adjusted to (110±5%) by using super plasticizer.

Specimens having dimensions of 40×40×160 mm were prepared. The ASTM C348-08 standard was used for casting and testing the specimens. Except for the specimens containing fly ash, which stayed in the molds for 2 days, all of the specimens were taken out from the molds after 24 h and allowed to cure for 3, 7 and 28 days in lime-saturated water. After the curing process, at least three specimens from each mixture were surface dried and tested in flexure by a universal testing machine using the ASTM C348-8 standard. For the compression test, the broken portions from flexure were used in accordance with ASTM C349-08.

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For the XRD test, control paste specimens and cement paste specimens containing 45% fly ash and 45% (fly ash+nanosilica) were prepared. After 28 days of moist curing, the specimens were dried, ground to pass through a 63 µm sieve and then subjected to XRD. TGA test was conducted on control and paste specimens containing the combination of 45% fly ash plusnanosilica. The porosity test was performed on 28-day cured mortar specimens by using a Sorptomatic 1990 series pore analyzer machine and the BET method, which was used to determine the size distribution of the nano-scale pores via liquid nitrogen. Specimens were subjected to pre-treatments of drying and vacuuming for 24 h, and then placed in the machine.

Table 1.Chemical composition of cement and fly ash

Items	Clinker (%)	Cement (%)	Fly ash (%)
SiO2	21.66	21.28	44.16
Al2O3	5.8	5.6	24.6
Fe2O3	3.68	3.36	12.5
CaO	65.19	64.64	5.34
MgO	2.86	2.06	2.5
TiO2	–	–	4.1
K2O	–	–	1.5
SO3	0.2	2.14	0.3
Total Alkalis	0.07	0.05	–
Insoluble Residue	0.1	0.22	–
Loss on Ignition	0.27	0.64	5.0

Table 2.Mix proportions of the mixtures

Mix Code	w/b ratio	water (g)	Cement content (g)	Sand (g)	Nano silica (g)	Fly ash (g)	super plasticizer (g)
P	0.4	175	440	1210	0	0	8
F1	0.4	175	330	1210	0	110	8
F2	0.4	175	286	1210	0	154	8
F3	0.4	175	242	1210	0	198	9
FS1	0.4	175	330	1210	11	99	10
FS2	0.4	175	286	1210	22	132	13
FS3	0.4	175	242	1210	33	165	15.5

III. RESULTS AND DISCUSSION

A. Compressive and Flexural Strengths

The compressive strength for specimens is shown in Figure 1. For F series specimens, replacing up to 35% of the cement by fly ash resulted in a comparable and/or even a slight increase in strength for 28-day cured specimens, while the strength for early age specimens was less than that of the control specimen [11]. The only exception to this behavior was 3-day cured specimens with 25% fly ash. The reduction in strength at early ages is due to slow pozzolanic activity of type F fly ash that contains lower amount of CaO. For 28-day cured specimens containing 35% fly ash, the strength loss was compensated because at this age, the specimens developed a denser calcium silicate hydrate structure. The filler effect of the fly ash contributed to compensating for the strength loss as well. Replacing 45% of the cement by fly ash resulted in a significant decrease in strength due to the low quantity of Portland cement, which negatively affects the hydration process. Less calcium silicate hydrate gel will be produced to bind the materials together, and the excessive amount of fly ash is just acting as filler without contributing in strength gain due to the lowCaO content in the used fly ash [9].

The specimens containing both fly ash and nanosilica (FS series) exhibited better performance than the control specimen's exhibit, and by increasing the nanosilica content, better strength was recorded for all curing regimes. Nanosilica enhanced the strength of fly ash specimens at early ages and a considerable increase in strength was obtained by increasing the nanosilica content to 7.5%. This behavior is primarily due to nanosilica's high performance in strengthening the calcium silicate hydrate structure [13]from one side, and strengthening the

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transition zone between the paste and the fillers (aggregate, fly ash) from the other side, rather than simply acting as a nano-filler.The maximum 28-day compressive strength of 90 MPa was recorded for specimens containing 37.5% fly ash plus 7.5% nanosilica.

Specimens in flexure exhibited comparable performance to that of the compressive strength as shown in Figure 2. Moreover, the rate of strength enhancement in flexure was roughly similar to that of the compression. The strength decreased by increasing the fly ash content and compensated and/or enhanced by adding nanosilica to the fly ash. The maximum flexural strength of 11 MPawas recorded for the specimens containing 37.5% fly ash plus 7.5% nanosilica.

The 28-day compressive strength (f'_c), flexural strength (f_f) and the rate of $f'_c$to f_f of control and nanosilica contained specimens are shown in Table 3. By increasing fly ash content from 25% to 35 the f'_c / f_f ratio increased to 8.28, while increasing the fly ash content to 45% brought down the f'_c / f_f ratio to as low as 7.58, which is the same ratio for control specimen. This phenomenon shows that, replacing cement by 25% fly ash has roughly the same effect in enhancing both compressive and flexural strengths with respect to control specimen. Whereas, by increasing the replacement rate to 35% the effect of the fly ash in enhancing the compressive strength becomes more significant. The decrease of f'_c / f_f ratio for the specimens containing 45% of fly ash corresponds to the decrease in both compression and flexural strengths. The addition of 2.5% nanosilica to 22.5% fly ash as a cement replacement resulted in a slight decrease in f'_c / f_f ratio, while for higher fly ash plus nanosilica replacement percentages the f'_c / f_f ratio increased significantly. Nanosilica enhanced the compressive and flexural strengths for specimens containing high volumes of fly ash in different ways. For instance, replacing cement by 22.5% fly ash plus 2.5% nanosilica had a better effect on the flexural strength enhancement than the compressive strength. On the other hand, the previous phenomenon reversed by increasing fly ash plus nanosilica percentages.

B. Microstructure

SEM: The SEM images for control (P), 45% fly ash (F3) and combination of 37.5% fly ash plus 7.5% nanosilica (FS3) contained specimens are shown in Figures 3 a, b and c respectively. In Figure 3 b, large amount of different sized, spherical-shaped fly ash can be seen filling the pores in the system [3], while less binder material of calcium silicate hydrate can be detected to hold together this large amount of fly ash. For the FS3 specimen the contribution of nanosilica in strengthening the boundaries between the pastes with fly ash particles can be detected, and due to the reaction of nanosilica with portlandite, an adequate amount of calcium silicate hydrate was formed to hold the fly ash in the system. The promoted effect of nanosilica in increasing the pozzolanic activity in the presence of fly ash as well as its filler effect resulted in strengthening the microstructure for the mortars. This phenomenon is the cause of strength enhancement in mortars containing a combination of fly ash and nanosilica.

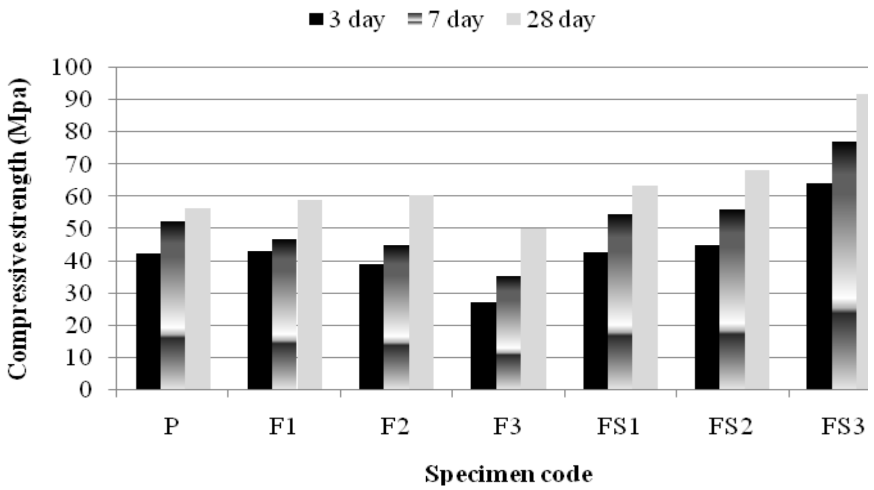

Fig. 1.Compressive strength for the specimens at different curing ages. P (control), F1 (25% fly ash), F2 (35% fly ash), F3 (45% Fly ash), FS1 (22.5% fly ash plus 2.5% nanosilica), FS2 (30% fly ash plus 5% nanosilica) and FS3 (37.7% fly ash plus 7.5% nanosilica)

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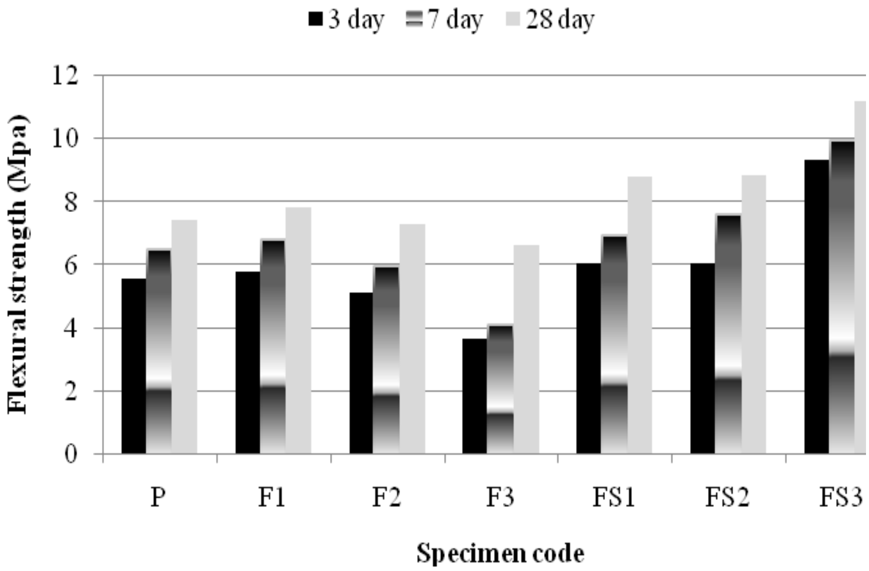

Fig. 2.Flexural strength for the specimens at different curing ages. P (control), F1 (25% fly ash), F2 (35% fly ash), F3 (45% Fly ash), FS1 (22.5% fly ash plus 2.5% nanosilica), FS2 (30% fly ash plus 5% nanosilica) and FS3 (37.7% fly ash plus 7.5% nanosilica)

Table 3.The rate of 28-day compressive strength to flexural strength for control, fly ash and fly ash plus nanosilica contained specimens

Specimens	f'_c (MPa)	f_f (MPa)	f'_c/f_f
P (Control)	56.28	7.43	7.58
F1 (25%FA)	59.06	7.83	7.54
F2 (35%FA)	60.36	7.29	8.28
F3 (45%FA)	50.24	6.63	7.58
FS1 (22.5%FA+2.5%NS)	63.50	8.82	7.20
FS2 (30%FA+5%NS)	68.23	8.88	7.69
FS3 (37.5%FA+7.5%NS)	91.75	11.19	8.20

XRD: To detect the changes in the compounds from replacing cement with fly ash and incorporating nanosilica into the system an XRD test was performed on controlled cement paste, pastes containing 45% fly ash and pastes containing 7.5% nanosilica + 37.5% fly ash. Cement paste compounds having a crystalline nature, such as calcium hydroxide, calcium silicate and silica, can easily be detected, while amorphous compounds such as calcium silicate hydrate can rarely be distinguished by this technique [3, 28].

The XRD diagrams for the specimens are shown in Figure 4. Replacing cement by fly ash resulted in a slight decrease in the calcium hydroxide peaks due to the pozzolanic reaction, while the incorporation of nanosilica decreased the amount of calcium hydroxide significantly [17, 29]. This phenomenon supports the idea of increasing calcium silicate hydrate (the binder material) due to conversion of calcium hydroxide to calcium silicate hydrate by nanosilica, which by turn explains the significant increase in strength for specimens containing nanosilica. Furthermore, a decrease in the silica peak in specimens containing nanosilica and fly ash indicates the effect of nanosilica in accelerating the reaction of coarser particles of silica with calcium hydroxide to produce larger amounts of high-density calcium silicate hydrate.

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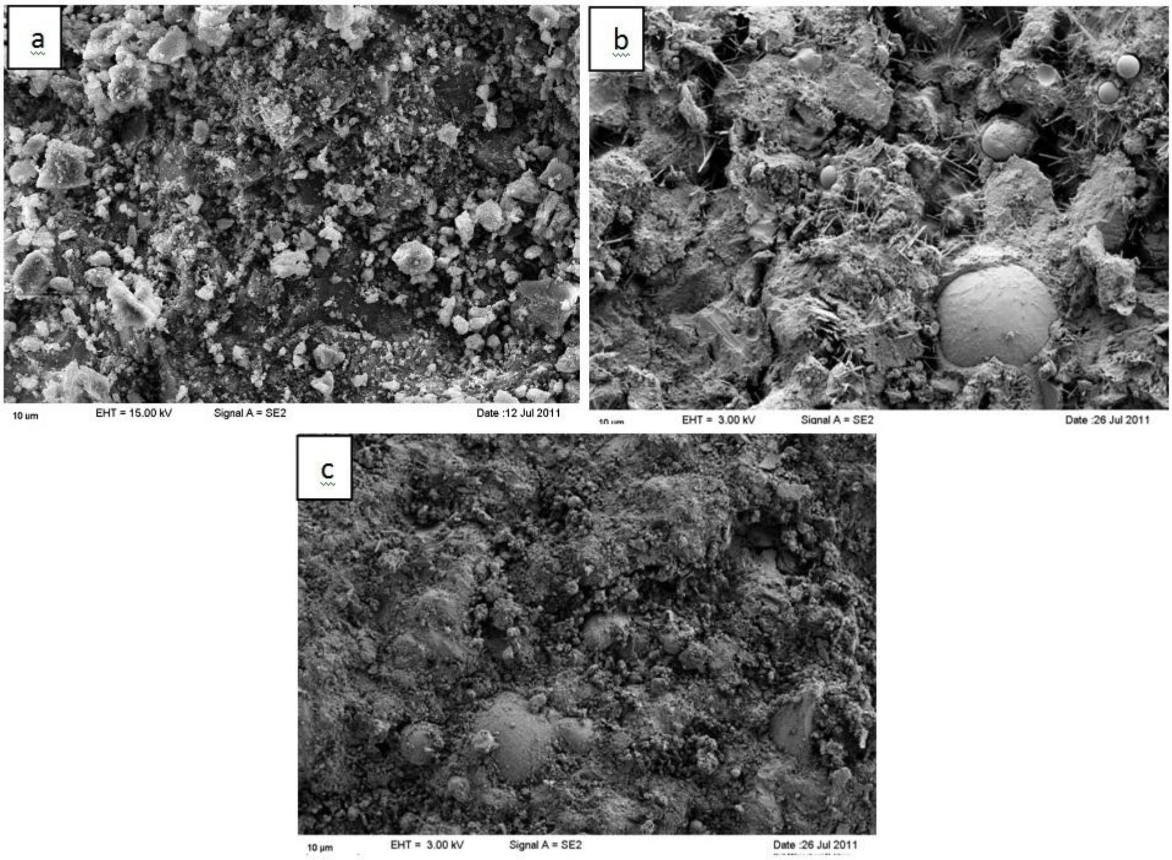

Fig. 3. SEM images for (a) controlled specimen P, (b) fly ash contained specimen F3 and (c) fly ash and nanosilica contained specimen

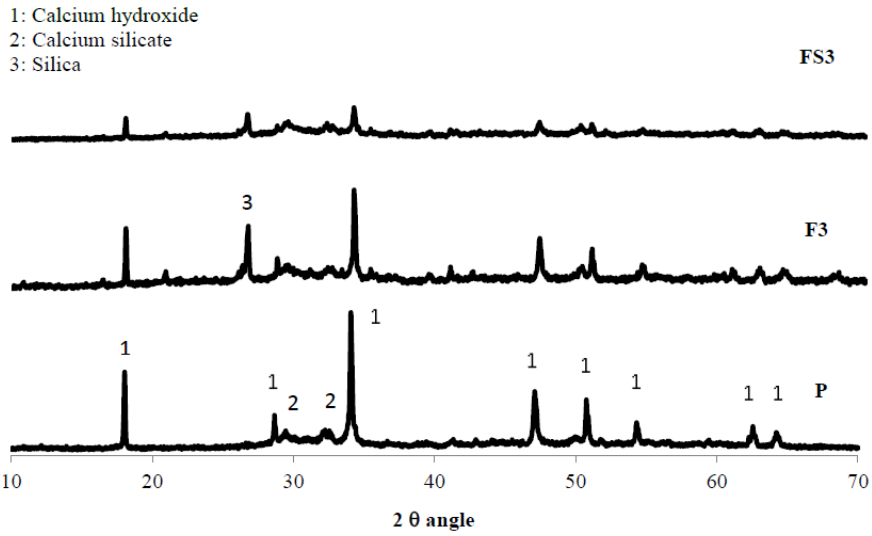

Fig. 4. XRD diagrams for controlled (P), fly ash (F3) and fly ash plus nanosilica(FS3) paste specimens

TGA: The thermal analysis for the hydrated cement paste in helium atmosphere showed that, through the temperature rise three peaks of weight loss can be detected (Fig. 5). The first weight loss located between 100 and 200 ºC was due to the evaporation of free water from the material. The second main weight loss was between 450 and 500ºC related to the dehydroxylation of calcium hydroxide. The third weight loss existed after 600ºC and corresponded to the dehydration of calcium silicate hydrate. After 400ºC, the hydration products started to disintegrate, and a complete disintegration occurred at 800ºC. Similar results with a narrow difference

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in the weight loss temperature peaks for hydrated cement paste containing different materials have been observed by other researchers [24, 30-32]. For fly ash plus nanosilica specimens less weight loss and disintegration were recorded along the temperature rise. This phenomenon shows that the samples containing fly ash plus nanosilica have denser and more stable hydration products than the cement paste samples. The contribution of nanosilica in enhancing the microstructure of hydrations products, particularly calcium silicate hydrate, led to the strength enhancement of specimens containing high volumes of fly ash.

BET Porosity: To study the changes in the pore structure at the nano-scale range, the BET porosity test was performed on the specimens. The pore size distribution curves for the specimens are shown in Figure 6. Replacing cement by fly ash resulted in an appreciable increase in nano-pores [33] that are smaller than 25 angstroms (Å), which are considered gel pores [26], while the incorporation of nanosilica into the system caused a significant decrease in the nano-pores in the specimens. For the larger pores, there was not an obvious difference between the specimens. The key action of nanosilica in enhancing the strength is its ability to reduce the pores that are smaller than 25 Å.

This phenomenon is due to the ability of nanosilicain producing more compacted silicate products and hence making the nano structure for the system denser.

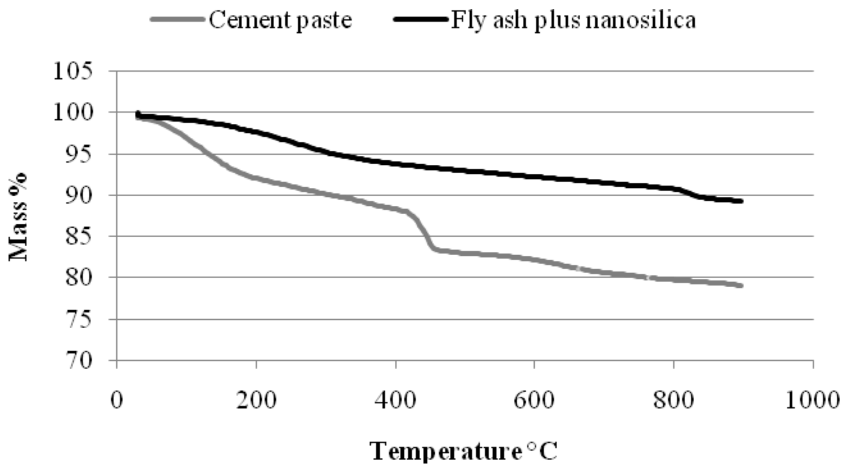

Fig. 5. TGA of control cement paste and cement paste containing fly ash plus nanosilica

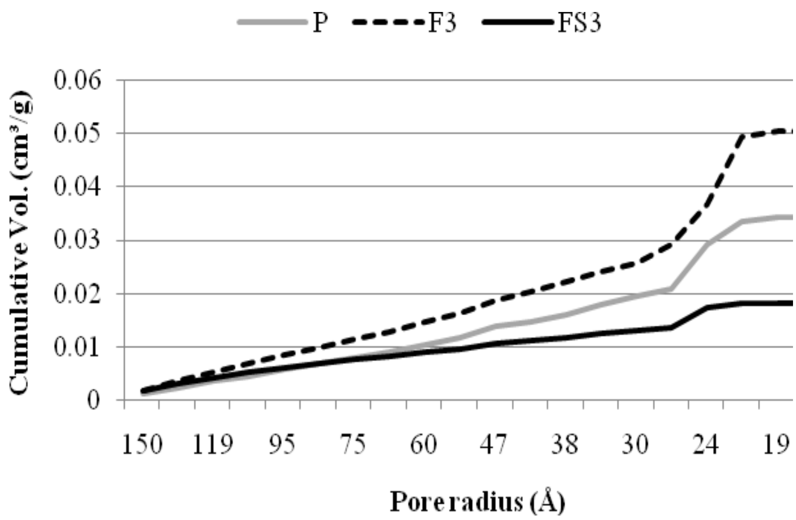

Fig. 6. Pore size distribution for controlled cement paste (P), fly ash (F3) and fly ash plus nanosilica (FS3) paste specimens.

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IV. CONCLUSIONS

The conclusions based on the strength results and the micro structural investigations were summarized below:

- Replacing up to 35% cement by fly ash can enhance the strength of mortars after just 28 days, while further replacements reduce the strength.
- The reduction in strength for 45% fly ash as a cement replacement is more pronounced at earlier ages than the later ages.
- Nanosilica not only compensates the strength loss caused by fly ash at earlier ages but also increases it appreciably at later ages.
- It is possible to produce high strength mortars with compressive strengths up to 90 MPa by adding nanosilica to high volume fly ash mortars, containing 45% less cement.
- Through the SEM images, it can be concluded that Nanosilica enhances the microstructure of mortars and concrete by acting as nucleus in producing more compacted structure of C-S-H.
- XRD tests showed that nanosilica have a high pozzolanic activity in converting calcium hydroxide to calcium silicate hydrate the material that is responsible for the strength of concrete.
- It can be concluded from the TGA tests that the specimens containing fly ash plus nanosilica have denser and more stable hydration products of calcium silicate hydrate than the control specimens
- The key action of nanosilica in enhancing the strength is its ability to reduce the gel pores that are smaller than 25 Å, the phenomenon that makes the C-S-H structure denser.

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