Properties of Self-Compacting Engineered Cementitious Composites containing Nano Silica Subjected to Elevated Temperatures

by

Melvin Yeo Chin Yaw

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Universiti Teknologi PETRONAS
Bandar Seri Iskandar
31750 Tronoh
Perak Darul Ridzuan

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A project dissertation submitted to the
Civil Engineering Programme
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Approved by,

_____
ASSOCIATE PROF. DR. BASHAR S. MOHAMED

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

MAY 2014

CERTIFICATION OF ORIGINALITY

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ABSTRACT

In this study, the properties of Engineered Cementitious Composites with the addition of Nano Silica is investigated when exposed to temperatures of up to 400°C. 25 different mix proportions with PVA Fiber up to 2% and Nano Silica up to 4% is use to be molded into cubes of 50mmX50mmx50mm. These concrete cubes are water cured for 28 days before being heated up to temperatures of 100°C, 200°C, 300°C and 400°C. Then the residual strength, microstructure and pore characteristics will beinvestigated. Based on the compressive strength results of the concrete , the strength increases as the percentages of Nano Silica increases in the mix. The same also applies as the percentages of PVA Fiber increases. When subjected to elevated temperatures, the samples will show an increase of compressive strength to the temperature of 300°C and then reduces at 400°C. The morphology and microstructure of the samples are examined .All the ITZ between cement matrix and fiber are determined. The ITZ will decrease as the percentage of Nano Silica increases up to 3%. At 4%, the ITZ will increase marginally due to entrapped air in the fresh mixture thus causing more porosity. The MIP for the samples are done to determine the pore characteristics. The increase of Nano Silica will reduce the accessible porosity of the sample up to 3% addition of Nano Silica. Above that, the porosity of the composite will increase.

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CHAPTER 1: INTRODUCTION

1.1 BACKGROUND

Every year, the use of concrete for construction projects globally exceeds 12 billion tons (Li, Lepech, Wang,Weimann&Keoleian, 2007).This makes concrete to be the second most consumed substance onEarth, after water.

Conventional concrete used today have drawbacks. They tend to crack easily when under loads, be it environmental or mechanical load. It shows that the conventional concrete is brittle. This brittleness makes concrete less durable. Therefore, there has been lots of research in countering this problem. One of the solutions to the brittleness in conventional concrete is the Engineered Cementitious Composites(ECC). The ECC is a part of the high performance fiber reinforced cementitious composites (HPFRCC). ECC strain hardens after the first cracking which makes it very ductile. ECC will have a strain capacity of 300 to 500 times more than normal concrete.

Another solution to the problem would be by incorporating suitable cementitious materials with cement to react with the excess Calcium Hydroxide to produce additional C-S-H. This will replace the porous Calcium Hydroxide and refine the pore structures which will reduce the permeability of the concrete. Therefore, various methods such as the pozzolanic reaction by means of chemical reaction of Calcium Hydroxide to get additional C-S-H materials or by pore filling is used to tackle concrete cracking up to a certain extend. Cementitiuos materials like the fly ash are used not only as a pozzolan but also as a filler. Fly ash is largely incorporated in Engineered Cementitious Composite (ECC).

In addition, new research has come up with Nano technology to be incorporated into concrete to further improve on the durability of concrete. Nano material like the Nano Silica enhances the reaction with calcium hydroxide to develop more strength carrying structure of cement, the C-S-H and also as a pore filling effect in the concrete.

Concrete cracking in conventional concrete can be much resolved by using ECC material which incorporates the use of fiber as a bridge in the composite and also conventional pozzolans like fly ash and new age Nano technology pozzolans like the Nano Silica to create more C-S-H gels in the matrix and to be a filler. Therefore, in this research, the composite will be comprised of these few combinations which is the incorporation of Nano Silica with ECC in order to achieve a composite that surpasses the properties of conventional ECC.

This research will also involve testing the behavior of ECC when exposed to high temperature. There have been studies being done regarding the properties of ECC at elevated temperatures. However, there has been no known research to the author's knowledge regarding the properties of ECC at elevated temperatures with the addition of Nano Silica.

1.2 PROBLEM STATEMENT

All concrete will experience material degradation when exposed to high temperatures for a long period of time. The extent of the degradation will be very much influenced by the constituents of materials used for the concrete.

In order to further improve the properties of Engineered Cementitious Composites when exposed to elevated temperatures, it will require different additions/alterations of the materials to the composite.

However, there are countless of possibilities to alter the mixture or addition to the concrete mix. A lot of manpower, material and time would be used to find the suitable material in order to improve the properties of the composite.

Therefore in this research we are incorporating Nano Silica into the mix. We chose Nano Silica to be used in this research due to its ability to be a pozzolan and filler at the Nano scale. Therefore, this research is based on the composite with varying percentages of Nano Silica and PVA Fiber .

1.3 OBJECTIVE

The objectives of this study are:

1. To determine the residual compressive strength of the ECC with addition of Nano Silica specimen when subjected to elevated temperatures.
2. To determine the morphology and microstructure of Engineered Cementitious Composites with the addition of Nano Silica.
3. To determine the pore structure characteristics of the ECC with addition of Nano Silica.

1.4 SCOPE OF STUDY

This study will focus on the properties of the ECC specimen that has been subjected to elevated temperatures. Among the properties that will be tested is the compressive strength, porosity of the concrete and pore characteristics of the concrete.

The study will also include the usage of Nano Silica and PVA fiberof different percentages in the ECC mix.This is tostudythe relation between the amount of Nano Silica and PVA fiber with the properties of the concrete when exposed to elevated temperatures.

The limiting factor of this research will be the temperature that the concrete will be exposed to in the furnace. We are limiting the temperature to be 400°C due to risk of explosive spalling. Precautions will be taken to minimize risk like drying sample in oven 48 hours before exposing it to high temperatures but in case of accident, it will risk setbacks to the research.

Besides that, not all the samples can be sent for Mercury Intrusion Porosimetry (MIP) and Field Emission Scanning Electron Microscopy (FESEM) test as the lab procedure for these test are very lengthy and takes up much time. Besides, the lab for these test are always full and booking in advance will have to be done. So, only a few samples can be done which will not reflect the entire result of the experiment.

1.5 RELEVANCY AND FEASIBILITY

Industrialization is a very real thing happening in all parts of the world today and one of the major material that is needed in all developing countries would be concrete .In line with development, buildings are becoming more advanced and complicated thus requiring an advancement in concrete technology. This research focuses on finding the properties of ECC with addition of Nano Silica subjected to different temperatures which will enlarge our understanding in using this specific composite to be used in specific industrial/commercial utilization depending on suitability.

The project is feasible within the scope, time frame and budget given. All materials are easily available from the concrete lab and/or from nearby vicinities. All testing equipments are available from the Civil Department of UTP and/or from other departments. All literatures are easily available from various websites to improve understanding regarding the project. The scope of this study and the main objective has been clearly defined. All lab works of concrete mixing and testing can be completed within the time frame.

CHAPTER 2: LITERATURE REVIEW

2.1 ENGINEERED CEMENTITIOUS COMPOSITES

As stated by Soe, Zhang and Zhang (2013), engineered cementitious composite contains a mix of cement, fly ash, sand, water, chemical adhesives reinforced with fibres. Engineered Cementitious Composite (ECC) is a special class ofthe new generation HPFRCC featuring high ductility with relativelylow fiber content of volume no more than 2 %. ECC is very ductile under tensile loading and is very much more durable due to its crack width of less than 100 Nanometres.

Engineered cementitious composites are used in multiple fields, including in shear elements that are subjected to cyclic loading, beam and column and for general structural repairs. ECC is also used as shielding layer to increase the resistance of corrosion for structures. ECC is also used in underground structures, highway pavements and bridge decks.

According to Huang, Ni & Li (2013), "In ECC, aggregates influence the material's tensile performance mainly through alterations in matrix fracture toughness and fiber dispersion".The matrix fracture toughness has to be limited for attaining strain hardening behavior of the composite. Therefore, coarse aggregate is not used in the mixture.

According to Sahmaran, Ozbay, Yucel, Lachemi & Li (2012), "The use of concrete containing high volumes of fly ash (FA) has recently gained popularity as a resource efficient, durable, and sustainable option for a variety of concrete applications".Tensile strain capacity at a range of 3–5% has been demonstrated in ECC materials using polyvinyl alcohol (PVA) fibers with fiber volume fraction no greater than 2%. ECC will demonstrate 300-500 times more tensile strain capacity comparing to normal concrete. (Sahmaran, Bilici, Ozbay, Erdem, Yucel, Lachemi, 2012)."ECC forms numerous micro-cracks which allow the material to undergo large tensile inelastic straining".(Sahmaran, Li ,2008) .However, based on Huang, Ranade, Zhang, Ni & Li

(2013), the amount of Fly ash use in the composite will greatly influence the tensile performance of the composites. By increasing the FA/C ratio from 2.2 to 4.4, the first cracking strength and ultimate tensile strength will decrease by 20% and 30% respectively. The tensile strain capacity will increase by 25% with the same increase in FA/C ratio. This difference is because as the FA increases, the frictional bond at the interface between PVA fibers and matrix will increase. Also, there will be a decrease in interfacial chemical bond and matrix fracture toughness. Based on Yang & Li (2012), " by increasing the amount of FA, it will modify the interfacial transition zone between the fiber and the bulk matrix and lower the amount of hydration products that chemically binds the fiber to the matrix".

Cavdar(2011) also came to the same result from using an increase of fly ash into the composite mix. The first cracking strength and flexural strength decreases but the deflection capacity will increase with increasing fly ash. Additionally, he did mention that the crack number will increase and crack width becoming tighter. This is an added advantage for the durability and self-healing of the composite. These effects will be beneficial to obtain high tensile ductility property. These properties of ECC make it very capable of absorbing high energy and are very resistant to impact shutter. The mechanical properties of ECC are highly dependent on the type, geometry and volume fraction of the constituents used in the mix. For example, the characteristics of the fibre will affect the mechanical properties significantly. (Soe et al., 2013)

ECC with increasing amount of fly ash will contribute to lower compressive strength of the composite. This is due to the reduction in strong primary hydration products due to the reduce amount of cement. (Huang et al.,2013). This is confirmed by Cavdar (2011) who also tested ECC with increasing fly ash and subjecting it to compressive strength test. Due to the reduced in cement, there is lesser hydration products. However, it was mentioned that fly ash is beneficial in long term strength development because of its pozzolanic property. At curing days of 28 days, the strength contribution of fly ash is not yet prevalent, but at later ages, fly ash will have a more pronounced strength contribution.

By using the absorption test as done by Cavdar (2011), it shows that the volume of penetrable pores reduces after the specimens are cured in water. This shows that the hydration continues to happen during water curing. It also shows that as the content of fly ash increase, the volume of penetrable pore will also increase.

When subjected to tensile load, ECC will form multiple cracks. This micro cracks will increase along with the increase of fly ash in the mix which is the sign of increasing tensile strain capacity. The formation of tight cracks in ECC is very much beneficial to the properties of the composite. For one, water permeability to the concrete decreases drastically by up to 7 orders with the normal micro cracks found in ECC which is less than 100 μm. This very much reduces the permeability of water, oxygen and chloride ion from reaching into the reinforcing steel and causing steel corrosion. According toZhang, Qian,Ma (2013), by increasing the amount of fly ash, it will increase the amount of water absorbed. This is due to the increased porosity bought by the increase of fly ash. Therefore, the volume of capillary pore may increase also, which will lead to larger water absorption.

2.2 EFFECT OF NANO SILICA IN CONCRETE

One of the newer technologies in concrete design is the use of pozzolanicNano Silica. The most commonly used NMs in cement products are Nano-SiO2 (NS), TiO2 (NT), Al2O3 (NA), Fe2O3 (NF), ZnO (NZ) and carbon Nano-tubes (CNTs). (Heikal,Aleem, & Morsi,2013). According to Kim, Heo & Lee (2014), "Nano Silica consists of particles between 1 nm and 500 nm, and is defined as a highly reactive siliceous colloidal material consisting of an amorphous SiO2 core with a hydroxylated surface". Nano Silica has a low cost of production, high reactivity and large specific surface area, which will result in a high degree of pozzolanic activity. According to Mukharjee & Barai (2014), "The use of Nano particles in cement based products was increasing day by day as these particles are effective in filling the voids of the C–H–S,

enhancing the rate of hydrations by acting as nucleation centers and reducing the size of Ca(OH)2 crystal".

According toSaid, Zeidan,Bassuoni and Tian(2012), with the addition of Nano Silica into the concrete, improvement of strength of the concrete can be attributed to the following four mechanisms.

I. Pozzolanic reaction. Cement water reaction will produce large amount of calcium hydroxide crystals. These crystals exist in the area between the aggregate and cement paste matrix which is bad for the strength and durability of the concrete. By the addition of Nano Silica which is very reactive, will produce C-S-H condensed gel by reacting with Ca(OH)2. This will reduce the amount of Ca(OH)2 and increase the amount of C-S-H gels in the concrete due to the pozzolanic reaction. The following shows the reaction of Nano Silica with the Ca(OH)2 to produce C-S-H gels.

$2C_3S + 11H_2O \rightarrow C\text{-}S\text{-}H + 3Ca(OH)_2$

$2C_3S + 9H_2O \rightarrow C\text{-}S\text{-}H + Ca(OH)_2$

$2SiO_2 + 3Ca(OH)_2 \rightarrow 3CaO.\ 2SiO_2\ .\ H_2O$

II. Nano filling property.

Nano particles which have the ability to be fillers can fill the porosity in the C-S-H gels to make the cement paste denser. According to Behfarnia & Salemi (2013), "Nano-scale SiO2 can act as a Nano-filler, filling the spaces between particles of gel of C‑S‑H".

III. Acting as a nucleus

Nano particles can act as a nucleus with an extremely strong bond with the C-S-H gel particles. This bond will increase the durability and mechanical properties of the concrete.

IV. Crystal making control

Formation process of crystals like $Ca(OH)_2$ will decrease if the amount of Nanoparticles and spacing is suitable. Up to 4% Nano Silica, compressive strength increases. But anything more than that will decrease the strength due to the Nanoparticles sticking together through a physical reaction and forming unstable balls.

Similarly, Jo et al., (2006); Zhang, Islam & Peethamparan (2012) also did their mix with varying amount of Silica fume and Nano Silica. Even with lesser amount of Nano Silica compared to Silica fume in terms of mass added into the mix, the compressive strength of mix containing Nano Silica will show better strength characteristics at both 7 days and 28 days. This confirms that pozzolanic reaction happens with the addition of Nano Silica and it performs much better and more effectively than Silica fume.

The addition of Nano Silica will change the microstructure of the hardened paste. As can be seen in SEM examination done byJo et al., (2006), the cement paste without Nano Silica will show the C-S-H gels appearing in isolation with many needle-hydrates surrounding the gel. However, the cement paste with the addition of Nano Silica shows the mixture with a much denser and compact formation of hydration products. The micrographs also show lesser number of Ca(OH)2 crystals. In a separate test done by Said et al., (2012) who uses Mercury Intrusion Porosimetry (MIP) to check the pore characteristics of the concrete confirms the above. This is because with the addition of Nano Silica, the total porosity decreases. With the mixture containing 3% Nano Silica and 6 percentNano Silica, the threshold pore diameters decreases by 36% and 48% respectively compared to the 0% Nano Silica mix. This can be contributed by the pozzolanic effect and filler effect of the Nano Silica but the contribution of each effect concerning the pore structure cannot be determined by the MIP.

Based on Said et al., (2012), mixes that are incorporated with Nano Silica will have an increase in temperature then followed with the deceleration period until a relatively constant temperature was recorded. However, this increase in temperature will peak higher with mixes containing Nano Silica as compared to mixes without Nano Silica.

The peak temperature will be about 20% higher and is reached in a shorter time period of approximately 4 hours. This increase should not be associated with the pozzolanic properties of Nano Silica because the pozzolanic reaction will only take place later with adequate formation of portlandite. Therefore, the reason attributing to this could be due to very high surface area of the Nano Silica particles which acts as a nucleation site for the hydration reactions. This means that the addition of Nano Silica will not only act as a pozzolanic or filler but also to sped up the kinetics of hydration due to its Nano size nature.

However,one thing to note is that with the addition of higher percentages of Nano Silica, the mix must be accompanied with higher usage of water and superplastisizer so that the specimens do not undergo dessication and cracking.(Jo et al., 2006).

According to Salemi, Behfarnia (2013), "In addition, becauseNanoparticles are more difficult to uniformly disperse, when the content are large, the weak zone in concrete increases which results in the decrease of the strength of the concrete".

It is also found that, fluidity; spreading and hardening time of the cement paste and mortar were reduced as the NS content was increased. (Heikal,Aleem, & Morsi,2013). This is attributed by the accelerated initial hydration of C3S due to the highly reactive surface of Nano Silica thus affecting the microstructure to be more close and compact.

However, it is known that the level of investigation of the micro effects with the addition of Nano Silica was minimal. The knowledge regarding the mechanisms that is affected by the addition of Nano Silica is not largely known which includes the flow properties, setting times, consistency, workability, rheological, micro structural, mechanical properties, etc.

2.3 EFFECT OF TEMPERATURE ON CONCRETE/CEMENTITIOUS COMPOSITES

When cement based composite is exposed to high temperature, there will be material degradation in terms of strength decrease, cracking, and spalling (Erdem ,2013)

. However, Cavdar(2011)mentioned that the effect of concrete by exposure to extreme heat source will be changes in the pore structures (cracking and spalling), destruction of bond between cement paste and aggregates , and the deterioration of the hardened cement paste.

When the concrete exposed to fire, the first thing would be the evaporation of the water from the concrete. Then with the increase of temperature, the hydrates will disintegrate and the loss of chemically bonded water will take place. Next, the decomposition of calcium hydroxide will occur at 350 degree centigrade. The partial volatilization of calcium Silicate hydrate gel will happen at 500 degree centigrade. By then the pore size and porosity of the hydrate matrix will increase and the mechanical properties decrease. At 573 degree centigrade, the crystal structure of quartz will transform to a high temperature phase which will increase the volume by 1 percent. This will accelerate the disintegration process of the hydrates. (Cavdar, 2011).

Studies done by Kalifa,Chene, Galle, (2001) have shown that by incorporating polypropylene fibers to the mix, the thermal stability will be improved. At 160 degrees centigrade where the fibers melt, there will be expansion channels. This additional porosity will either decrease the residual strength or improve it depending on factors like the cure condition, experimental conditions and also heating rate as recorded by Cavdar (2011).

Erdem (2014) confirms this with the ECC composites heated up to 200 degree centrigrade incorporating PVA fiber. The melting of the fiber will cause additional porosity and small channels which will lower the internal vapor pressure and allow for the escape of moisture which will eliminate the possibility of explosive spalling. At 2% fiber, the volume is high enough to ensure that the fibers will constitute a connected network. Explosive spalling under high temperatures is a concern which may be caused by internal cracking, pore pressure, rapid heating of concrete specimens, moisture content and strength grade of concrete. As recorded in Cavdar (2011), samples were exploded at temperature of 400 degrees centigrade. In order to overcome this problem, the samples were taken out from the curing medium 24 hours before testing and were

put in the oven for 24 hours to dry. This is to remove the water from the matrix and eventually eliminating the pressure build up.

2.4 SELF COMPACTING CONCRETE

Self-compacting concrete is also known as self-consolidating concrete and it is a highly flowable concrete that spreads into the form without the need of mechanical vibration. Self-compacting concrete is significant because it maintains all concrete's durability and characteristics. Self-compacting concrete has characteristics of such that it does not segregate, has high deformability and excellent stability. Self-compacting concrete (SCC) is a type of high fluidity and strength concrete developed in Japan in 1988 to increase productivity and durability in concrete construction. SCC mixtures contain super plasticizer, admixtures, limited amounts of aggregate and low water-powder ratio. (Sua-iam,Makul, 2013).

Self-compacting concrete has many benefits over regular concrete. Among those are:

a) Improved constructability.
b) Labor reduction.
c) Bond to reinforcing steel.
d) Improved structural Integrity.
e) Accelerates project schedules.
f) Reduces skilled labor.
g) Flows into complex forms.
h) Reduced equipment wear.
i) Minimizes voids on highly reinforced areas.
j) Produces superior surface finishes.
k) Superior strength and durability.
l) Allows for easier pumping procedure.
m) Fast placement without vibration or mechanical consolidation.
n) Lowering noise levels produced by mechanical vibrators.
o) Produces a uniform surface.

p) Allows for innovative architectural features.

q) Produces a wider variety of placement techniques.

CHAPTER 3: RESEARCH METHODOLOGY& PROJECT ACTIVITIES

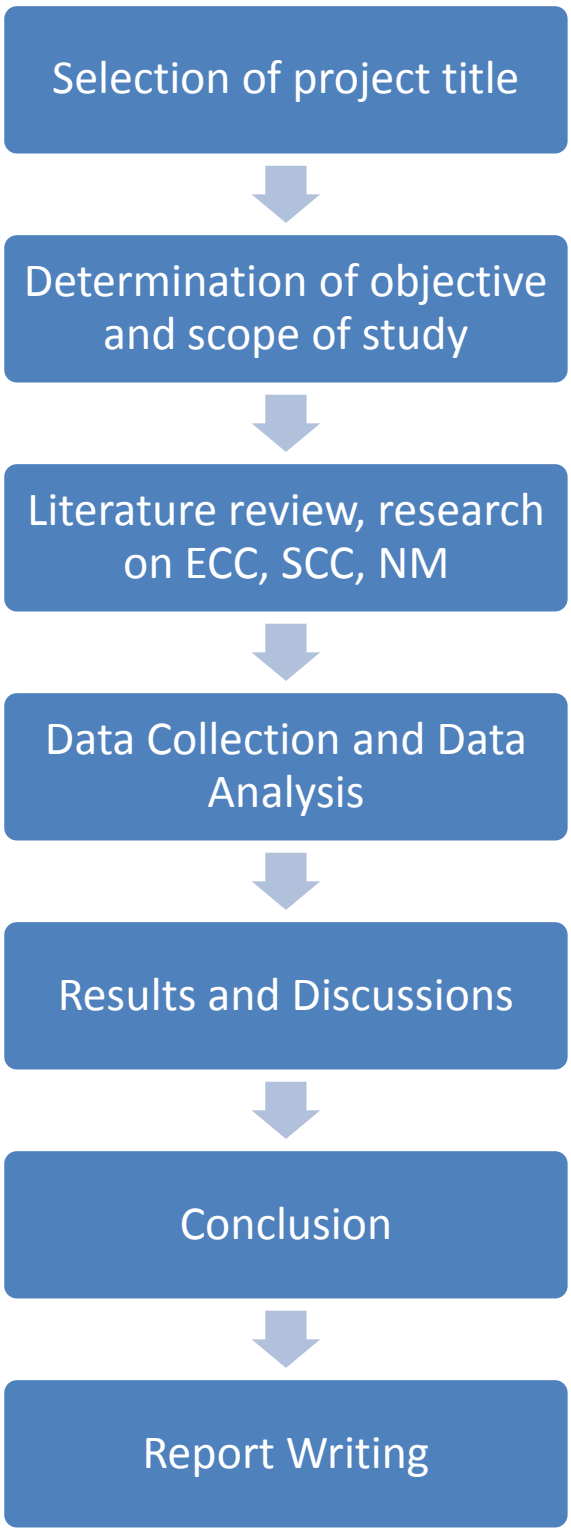

Figure 1: RESEARCH METHODOLOGY

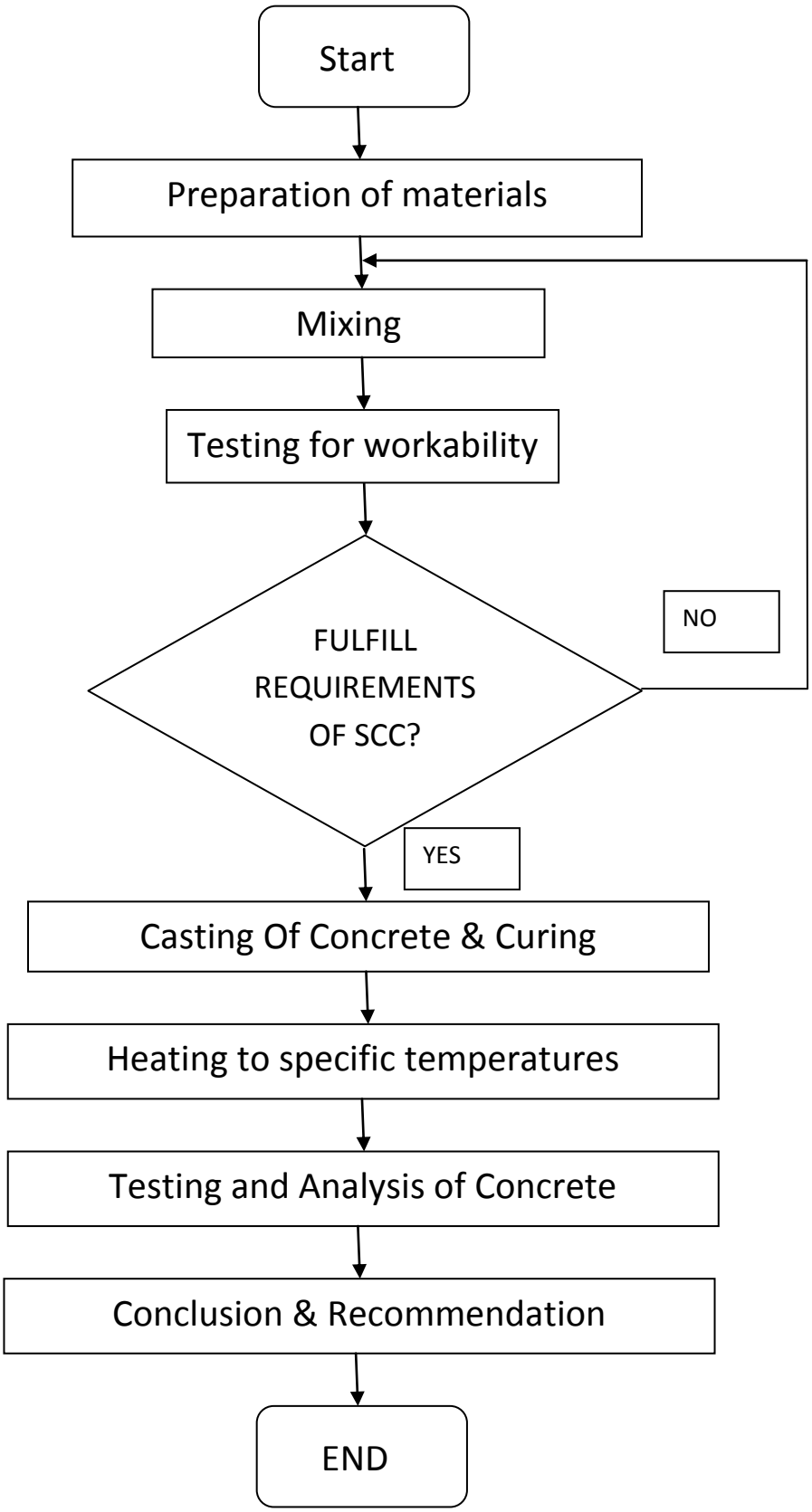

Figure 2: LAB METHODOLOGY

3.1 MATERIALS

The materials used in this projectare:

a) Ordinary Portland cement

Figure 3: Type 1 Cement

b) Fly Ash

Figure 4: Type F Fly Ash

c) River Sand

Figure 5: Coarse River Sand

d) PVAFiber

Figure 6: RFS 400 18mm PVA Fiber

e) Super-plasticizer

Figure 7: Sika ViscoCrete-2044

f) Nano Silica

Figure 8: 99.5% Pure Nano Silica Powder

g) Clean mixing water

3.2 MIX DESIGN

Table 1:Mix Design

Mix	Cement (kg/m^3)	Sand (kg/m^3)	Fly Ash (kg/m^3)	Water (kg/m^3)	w/c	Super-plastisizer (kg/m^3)	PVA Fiber(%)	Nano Silica(%)
1a	583	467	700	187	0.32	4.5	0	0
1b	583	467	700	187	0.32	4.5	0	1
1c	583	467	700	187	0.32	4.5	0	2
1d	583	467	700	187	0.32	4.5	0	3
1e	583	467	700	187	0.32	4.5	0	4
2a	583	467	700	187	0.32	4.5	0.5	0
2b	583	467	700	187	0.32	4.5	0.5	1
2c	583	467	700	187	0.32	4.5	0.5	2
2d	583	467	700	187	0.32	4.5	0.5	3
2e	583	467	700	187	0.32	4.5	0.5	4
3a	583	467	700	187	0.32	4.5	1	0
3b	583	467	700	187	0.32	4.5	1	1
3c	583	467	700	187	0.32	4.5	1	2
3d	583	467	700	187	0.32	4.5	1	3
3e	583	467	700	187	0.32	4.5	1	4
4a	583	467	700	187	0.32	4.5	1.5	0
4b	583	467	700	187	0.32	4.5	1.5	1
4c	583	467	700	187	0.32	4.5	1.5	2
4d	583	467	700	187	0.32	4.5	1.5	3
4e	583	467	700	187	0.32	4.5	1.5	4
5a	583	467	700	187	0.32	4.5	2	0
5b	583	467	700	187	0.32	4.5	2	1
5c	583	467	700	187	0.32	4.5	2	2
5d	583	467	700	187	0.32	4.5	2	3

5e	583	467	700	187	0.32	4.5	2	4

3.3 LABORATORY WORK: CONCRETE MIXING & CURING

Based on the mix design, the materials which are Ordinary Portland Cement (OPC), River Sand, Fly Ash(FA) , PVA Fiber ,Superplastisizer(SP), Water and Nano Silica are measured using the weighing machine.

Figure 9: Weighing of the materials

Figure 10: All materials weighed

The OPC, River sand, Fly Ash , PVA Fiber and Nano Silica are placed in the cement mixer . The superplastisizer is mixed together with the water and the container holding the SP is mixed thoroughly with the mixture to avoid loss of SA to be stuck on the wall of the container. Half of the SP+Water mixture is added into the mixture. The mixer is turned on for 3 minutes or until the mix is thoroughly distributed. The remaining SP+water are added and the mixing continues for an additional 3 minutes. Further usage of super-plastisizer is added 2 ml at a time until slump of fresh concrete is achieved.

Figure 11: Materials inside the mixer

9 moulds of 50mmx50mmx50mm (27cubes) is casted for each of the 25 mixes. The concrete cubes are removed from the moulds after 24 hours. Each cube are labeled and put into the curing tank for 28 days before testing.

Figure 12:The moulds prepared for the mix

Figure 13: The concrete cubes in curing tank

3.3.1WORKABILITY TEST

Workability is defined as the amount of internal work needed to compact fresh concrete . Concrete that is highly workable requires very little compactive effort yet does not segregate. Therefore, meeting the requirement of self-compacting concrete. All fresh concrete are tested for workability via the following tests.

Table 2: Workability Test

Test	Accordance	Range of values	
		Min	Max
Slump-flow by Abrams Cone	EN 12350-8:2010	650	800
T50cm slump flow	EN 12350-8:2010	2 seconds	5 seconds
V funnel	EN 12350-9:2010	6 seconds	12 seconds
L box	EN 12350-10:2010	0.8 (h2/h1)	1 (h2/h1)

Figure 14: T-50 slump flow

Figure 15: Slump Flow

Figure 16: Slump Flow measurement

3.3.2 HEATING OF CONCRETE

Before the concrete are being subjected to the furnace, all the concrete cubes are put in the oven to dry for 24 hours at 60˚C. This is to evaporate all existing moisture on the concrete cube.

Then, the concrete cubes are heated to the following temperatures before being subjected to the FESEM, MIP and Compressive Test. At each time, 5 concrete cubes will be put into the furnace to be subjected to the preset temperature. Then after the samples are cooled down, the samples are taken out and another set of 5 concrete cubes are put in and subjected to another preset temperature. The following table shows the summary of temperatures that will be subjected to the cubes.

Table 3: Concrete Heating

Temperature	**No. of concrete cubes**	**Heating rate**	**Target temperature heating Duration**	**Cooling time**
100◦C	5	5◦C/min	1 hour	≈1 hour
200◦C	5	5◦C/min	1 hour	≈2 hours
300◦C	5	5◦C/min	1 hour	≈3 hours
400◦C	5	5◦C/min	1 hour	≈4 hours

Figure 17: Protherm Furnace for heating of samples

3.3.3 LABORATORY WORK: CONCRETE TESTING

The following tests are done to find out the properties of this composite:

I. Compressive Strength Test
II. Mercury Intrusion Porosimetry (MIP)
III. Field Emission Scanning Electron Microscopy (FESEM)

3.3.3.1FIELD EMISSION SCANNING ELECTRON MICROSCOPY (FESEM)

The FESEM is used for ultra-high resolution electron imaging of the concrete after being subjected to the different temperatures.

Before the samples are subjected to FESEM, the samples have to be cut to a much smaller and thinner section. This is done by using the diamond cutter at the mechanical engineering laboratory of Block 15, UTP. Then, the samples are polished to ensure smoothness at the surface. The following is an example of the FESEM capturing the ECC microstructure and the FESEM facility at Block J, UTP.

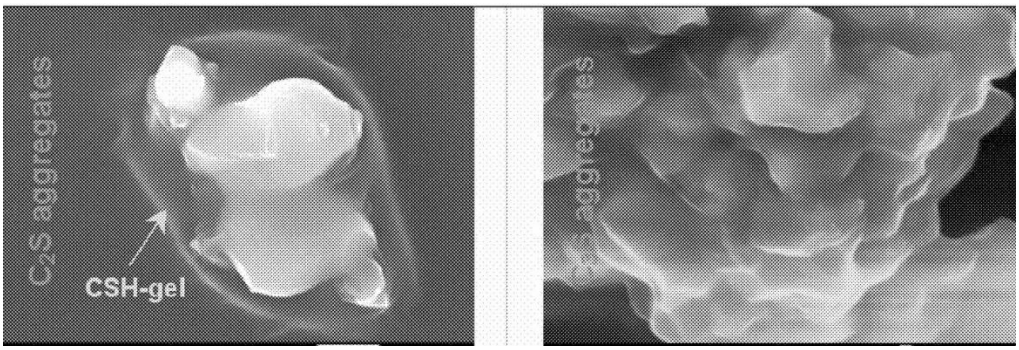

Figure 18 : FESEM of Concrete

Figure 19: FESEM at Block J, UTP

Figure 20: The samples prepared for FESEM

3.3.3.2MERCURY INTRUSION POROSIMETRY

Mercury Intrusion Porosimetry is used to determine various quantifiable aspects of a material's porous nature, such as pore diameter, total pore volume, surface area, and bulk and absolute densities. Similar to FESEM, the samples have to be prepared

before hand. It has to be cut using the diamond cutter to size similar to that of the FESEM sample. Only then the sample can fit into the vial for the MIP test.

3.3.3.3COMPRESSIVE STRENGTH TEST

A compression test is used to determine the behavior of the concrete cubes under crushing loads. Three cubes are used at each test to ensure uniformity in the result and the averagedvalue is taken.

Figure 21: Compression Machine

3.4KEY MILESTONES

The planned schedules for Final Year Project II are as follows:

Table 4: Key Milestones

Preparing and testing of samples (Compressive Strength Test)	Week 1- Week 7
Submission of progress report	Week 7
Prepare samples to undergo FESEM and MIT	Week 8-Week 10
Pre-sedex	Week 10
Viva presentation	Week 13
Submission of Dissertation	Week 14

3.5GANTT CHART

The Gantt chart for FYP 1 is as follow:

No	Detail / Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Selection of Project Title														
2	Identify Problem Statement, Objectives and Scope of Study														
3	Collection of Literature Review														
4	Submission of Extended Proposal														
5	Research on ECC,SCC, and Nano Silica														
6	Proposal Defense														
7	Submission of Draft Report														
8	Submission of Final Report														

Figure 22: Gantt Chart FYP1

The Gantt Chart for FYP 2 is as follow:

No	Detail / Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14	
1	Mixing Concrete															
2	Curing of concrete															
3	Submission of progress report															
4	Testing of concrete															
5	Analysing of result and discussion															
6	Pre-Sedex															
7	Submission of final draft report															
8	Submission of dissertation															
9	Submission of technical paper															
10	Viva															
11	Submission of Project Final Dissertation															

Figure 23:Gantt Chart FYP 2

CHAPTER 4: RESULTS AND DISCUSSIONS

The results are showing the correlations of the amount of Nano Silica, PVA Fiber, temperatures subjecting the samples and the affected compressive strength. There are a total of 5 different Nano Silica percentages(0%,1%,2%,3%,4%) and PVA Fiber (0%,0.5%,1%,1.5%,2%).This accounts for 25 different mixes . Each of the mix is subjected to elevated temperatures of 100˚C,200˚C, 300˚C and 400˚C. All the samples are cured for 28 days in the curing tank. Before being subjected to the elevated temperatures, the samples are taken out from the curing tank to air dry, then put in a oven at 60 for 48 hours to completely dry the samples to prevent spalling for occurring.

4.1 SURFACE CHARACTERISTICS

When the specimens are exposed to elevated temperatures, there are no colour changes observed. However, cracks are observed on the specimens exposed to temperatures of above 300˚C.

4.2 COMPRESSIVE STRENGTH

The pictures below shows the concrete cubes after being subjected to the compression machine. The cubes show different mode of failure. Most of the cube shows almost equal cracks at all four sides which is a satisfactory failure as stated in BS EN 12390-3:2009.

Figure 24: Sample Failure

Figure 25: Sample Failure

Figure 26: Sample Failure

Figure 27: Sample Failure

The following graphs shows the relationship between the percentage of Compressive Strength versus temperature with regards to the increase of Nano Silica .

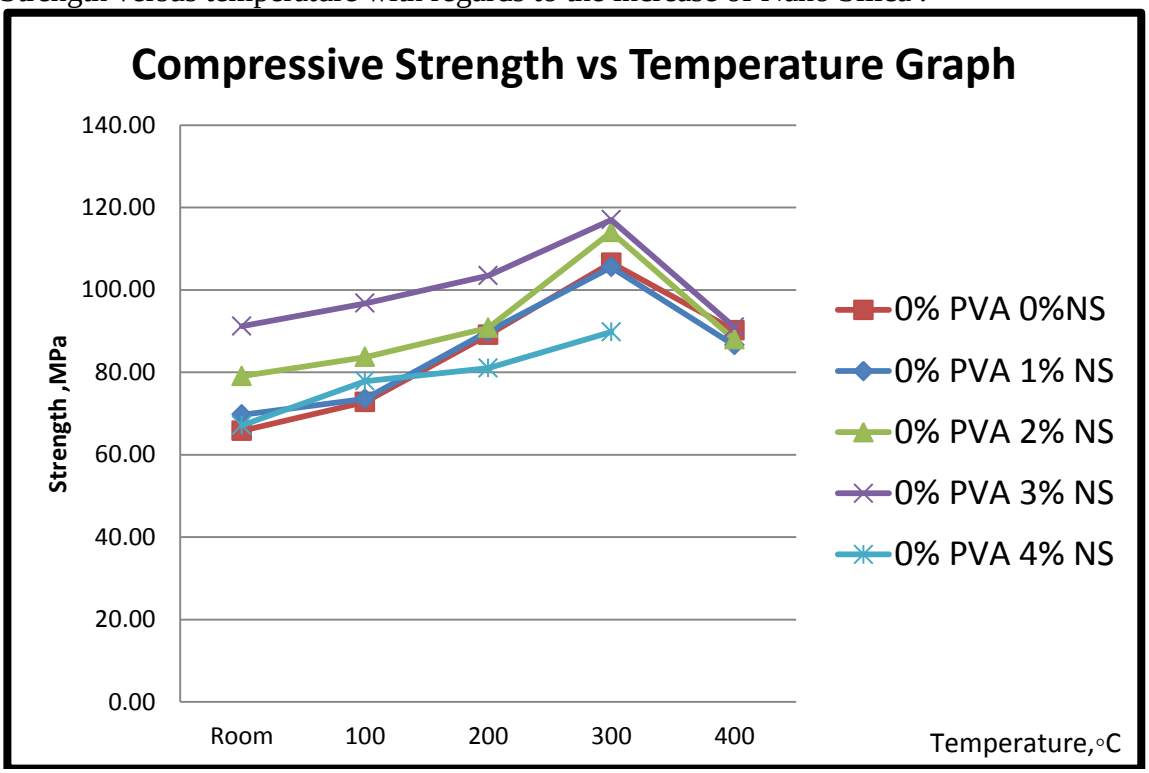

Figure 28:Compressive Strength of 0% PVA versus Temperature

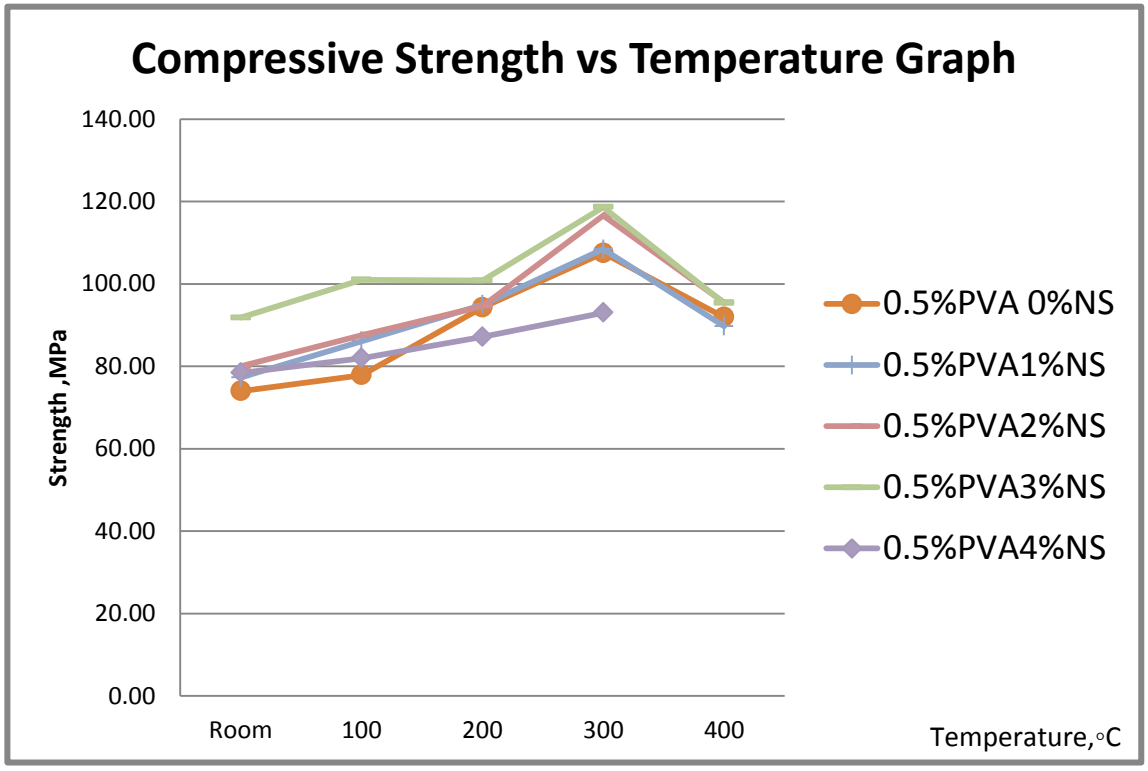

Figure 29:Compressive Strength of 0.5% PVA versus Temperature

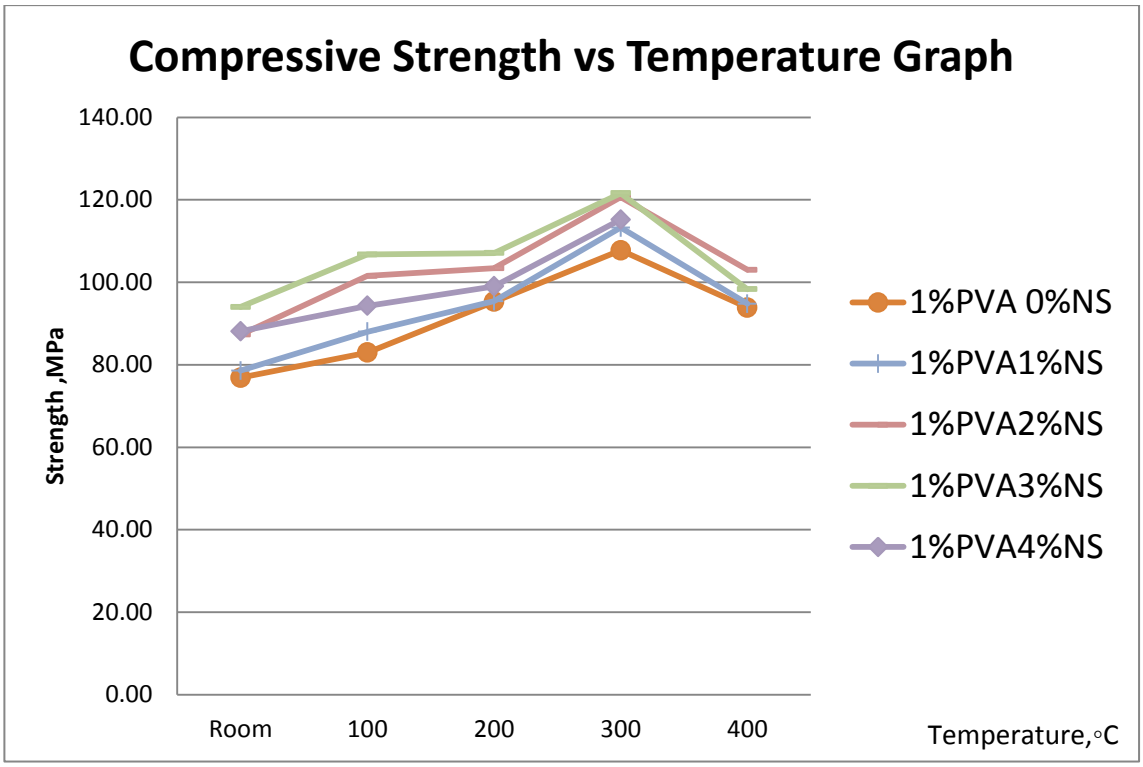

Figure 30:Compressive Strength of 1% PVA versus Temperature

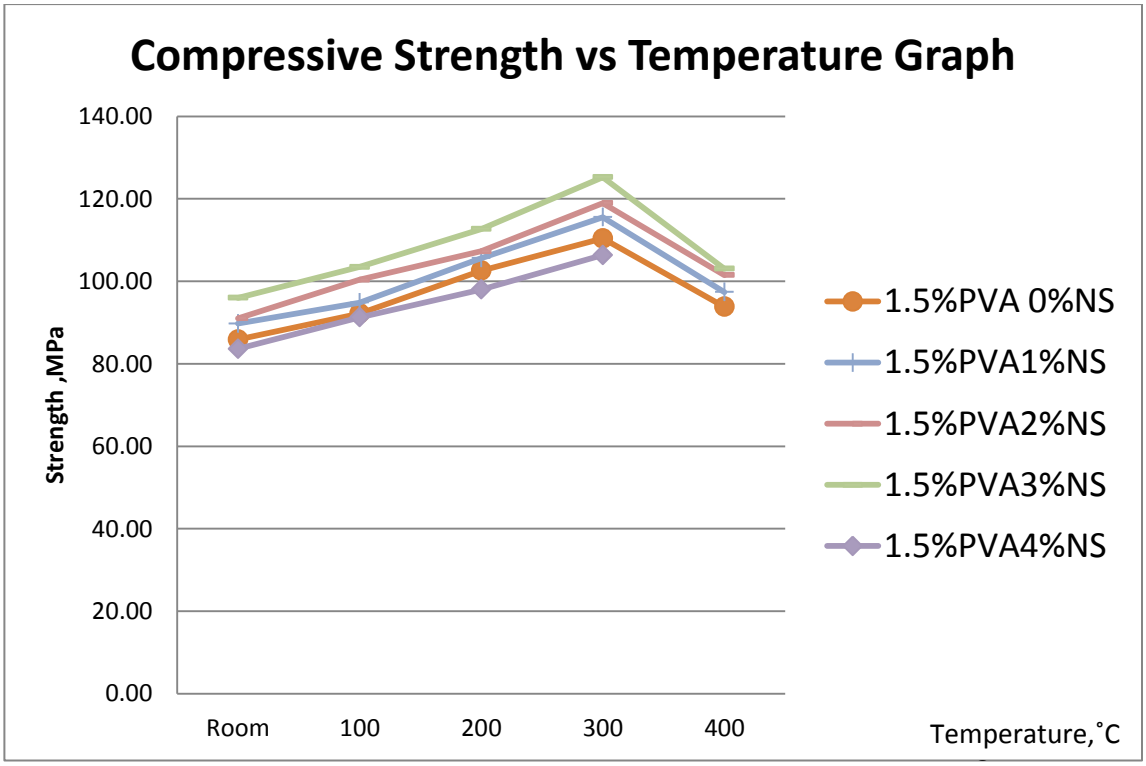

Figure 31: Compressive Strength of 1.5% PVA versus Temperature

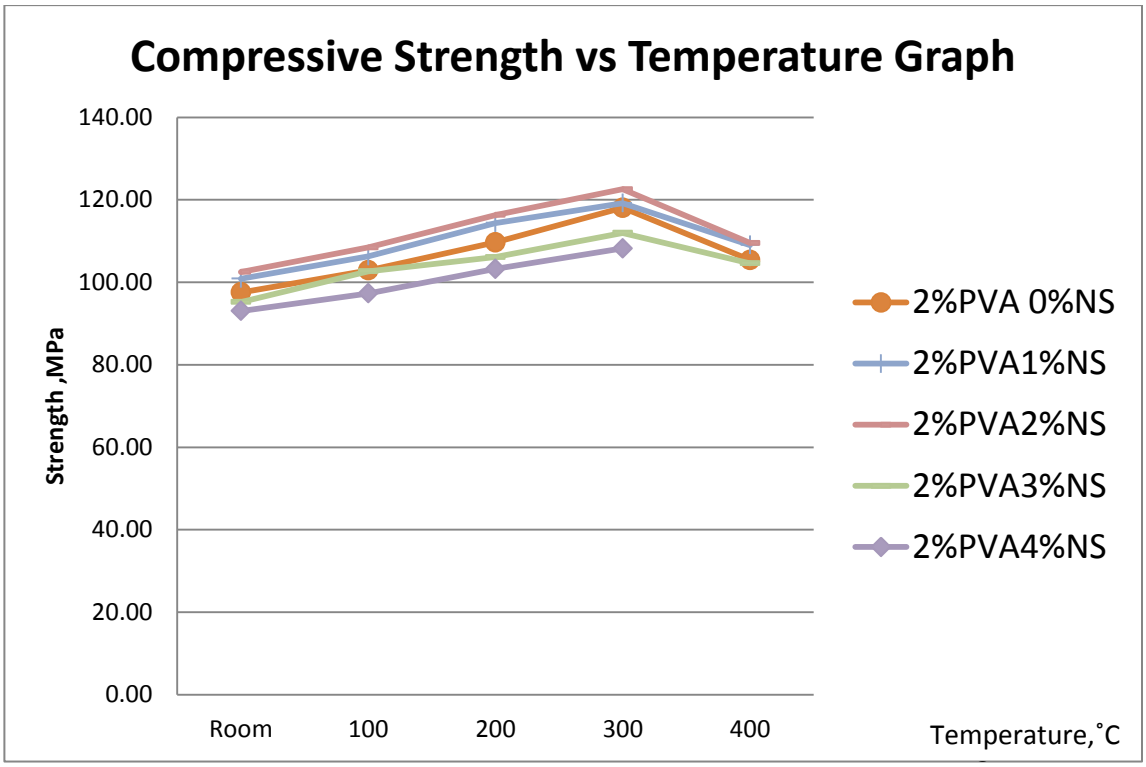

Figure 32: Compressive Strength of 2% PVA versus Temperature

The compressive strength results are taken from the average of three specimen .The control samples have an average strength of 65.77 MPA at room temperature. When the samples are added with Nano Silica, the trend shows an increase in compressive strength as the amount of Nano Silica added is increased from 0% to 1% and to 2%. This confirmsprevious literatures that the addition of Nano Silica will improve the compressive strength of the composite. This could be due to the Nano Silica acting as a pozzolan, filler or as both like comfirmed by Said, Zeidan,Bassuoni and Tian(2012),.

However, regardless of the percentage of PVA Fiber added to the composite, addition of Nano Silica percentage above 3% will cause a decrease in compressive strength. The decrease is in the range of 15% -26% .This decrease could be attributed to the excessive usage of superplastisizer which as noted in some literatures may cause decrease in strength (Alsadey S., 2012).The amount of super-plastisizer use in our mixes is to get the proper slump and workability of raw concrete.

The percentage of super plasticizer added increases as the percentage of Nano Silicaincrease. In order to obtain the same workability, the amount of super-plastisizer will have to be increased. This increase is more significant as the amount of PVA Fiber is also increasing in the mix of the composite.

According to Yu, Spiesz and Brouwers(2014), when adding super-plastisizer into the mix, it will retard the dormant period of cement hydration. But, this effect can be reversed with the addition of Nano Silica which promotes the hydration of cement. Therefore, it is important to find the right amount of super-plastisizer and Nano Silica to be in the mix.

The compressive strength of the ECC increases as the samples are subjected to elevated temperatures of 100℃, 200℃ and300℃ for all mixes of varying percentages of Nano Silica and PVA fiber. This increase could be due to the acceleration of the hydration process cause by the evaporation of free water content in the concrete. (H. A. M. Bishr, 2008).

At temperature above 300˚C, the ECC mix demonstrates a decrease in strength for all mixes regardless of percentages of PVA Fiber and Nano Silica. This reduction is due to the dehydration of concrete of the free water, interlayer water and disintegration of the chemically bound water.

At 400˚C, the concrete undergoes explosive spalling. However, this is only observed with mixes of 4% Nano Silica for all percentages of PVA Fiber. This spalling may occur due to the denseness of the concrete matrix which doesn’t allow for the internal pressure to be released.

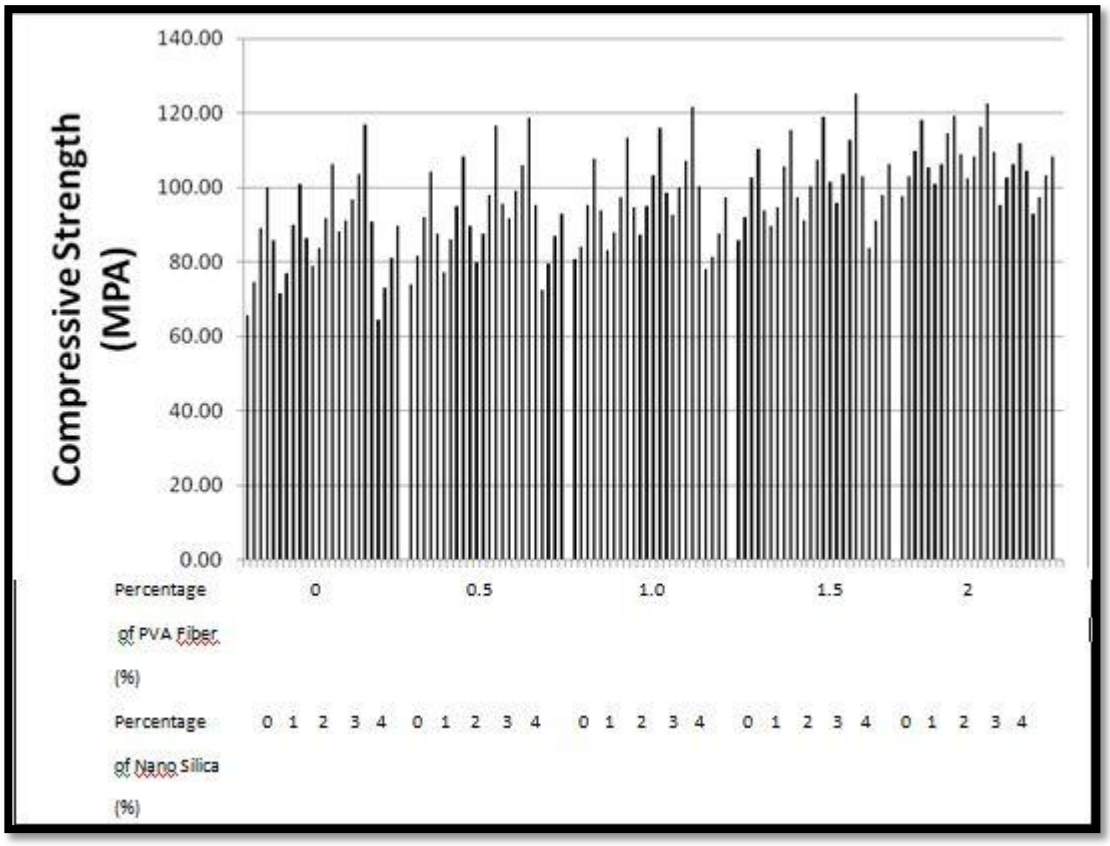

Figure 33: Overall Compressive Strength

4.3 FESEM

The FESEM is used in this project to obtain the morphological characteristics and microstructure of the concrete. The FESEM shows that overall, the mix has a very dense structure and good ITZ. The following are the FESEM for 5 samples. It shows that as the percentage of Nano Silica increases, the ITZ for the sample will decrease. Thus, it confirms that the addition of Nano Silica will cause a refinement in the microstructure. It can be observed that the dense structure has only very few air pores which could be attributed to the formation of C-S-H gels due to reaction of cement with water. This could also be due to its filler properties in reducing pore sizes.

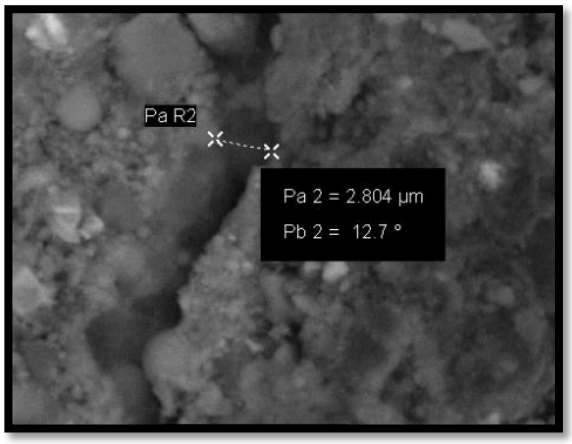

Figure 34: 0% Nano Silica

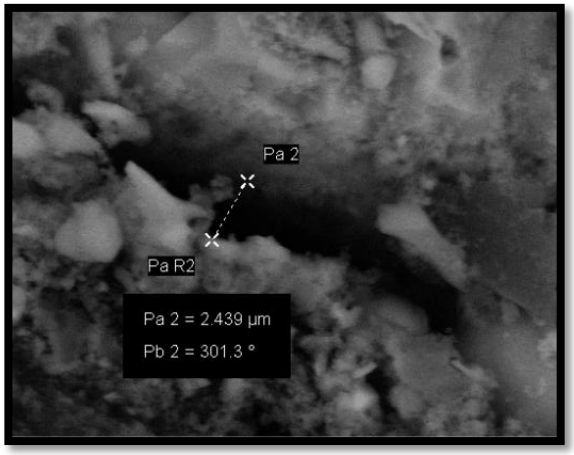

Figure 35:1% Nano Silica

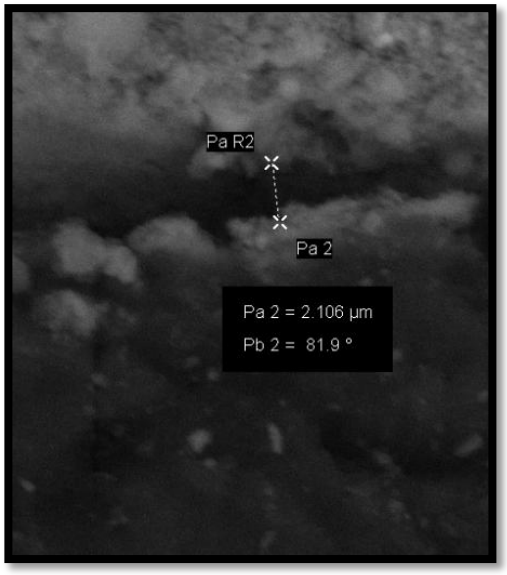

Figure 36:2% Nano Silica

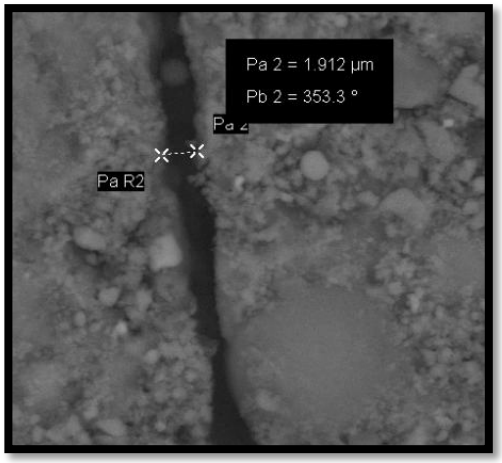

Figure 37:3% Nano Silica

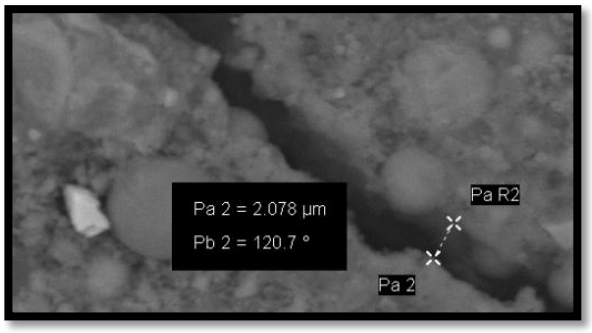

Figure 38:4% Nano Silica

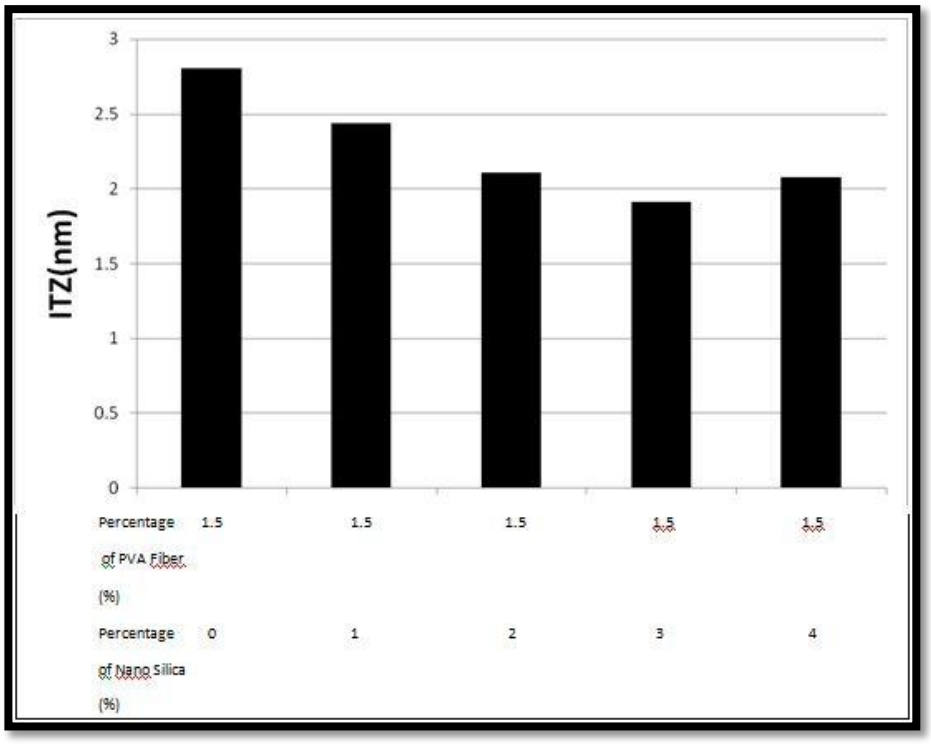

Figure 39: ITZ Graph for PVA Fiber 1.5%

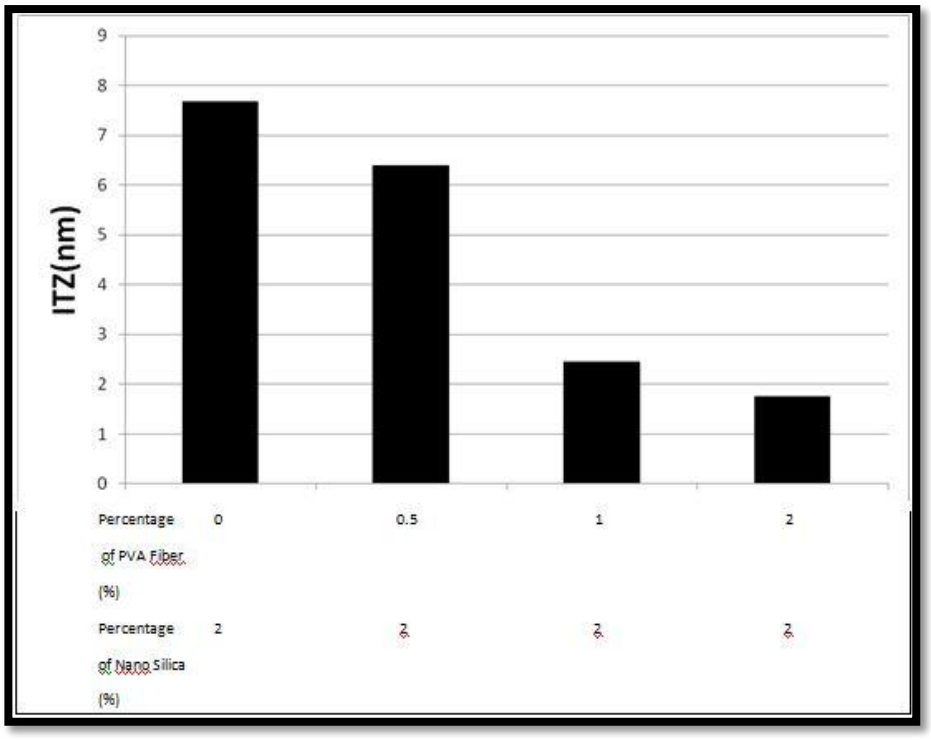

Figure 40: ITZ graph for Nano Silica 2%

The microstructure with specimen of 4% Nano Silica has quite a number of pores that can be observed. A bigger ITZ value for the specimen is also obtained compared to the ITZ of 3% addition of Nano Silica. Additionally, extra cracks can be observed running parallel to the cracks that usually appear next to the fiber. This additional crack will contribute to additional porosity to the cement matrix. This reflects the result that is obtained from the compressive strength test which shows a decrease in all 4% of addition of Nano Silica composites. Yu, Spiesz and Brouwers, (2014) states that addition of Nano Silica will increase the viscosity of the mixture. Therefore, this causes a lot of air to the trapped in the mix which increases the porosity of the concrete. This explains the reason of increasing porosity and decreasing compressive strength of the 4% Nano Silica mix.

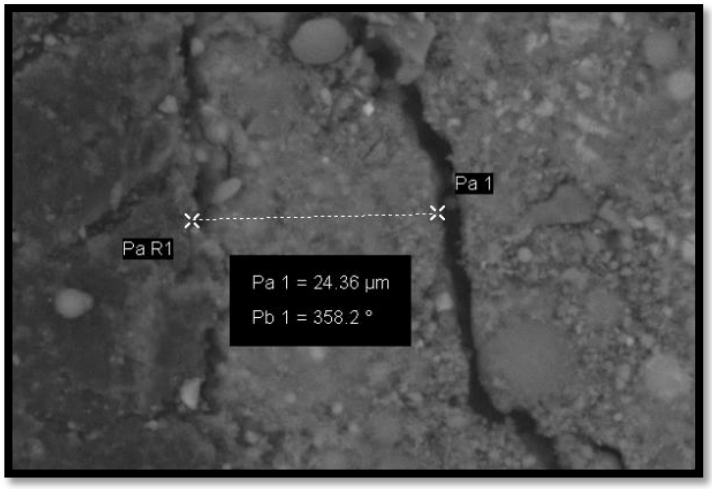

Figure 41: Additional hairline cracks at 4%

4.4 MERCURY INTRUSION POROSIMETRY (MIP)

Table 5: Porosity of Samples for 1.5% PVA Fiber

Sample	1.5% PVA, 0% NS	1.5% PVA, 1% NS	1.5% PVA, 2% NS	1.5% PVA, 3% NS	1.5% PVA, 4% NS
Accessible Porosity %	3.43	2.95	1.78	4.39	3.15

Table 6: Porosity of Samples for 2% N.S.

Sample	0% PVA, 2% NS	0.5% PVA, 2% NS	1.0% PVA, 2% NS	2% PVA, 2% NS
Accessible Porosity %	6.89	5.93	5.81	5.14

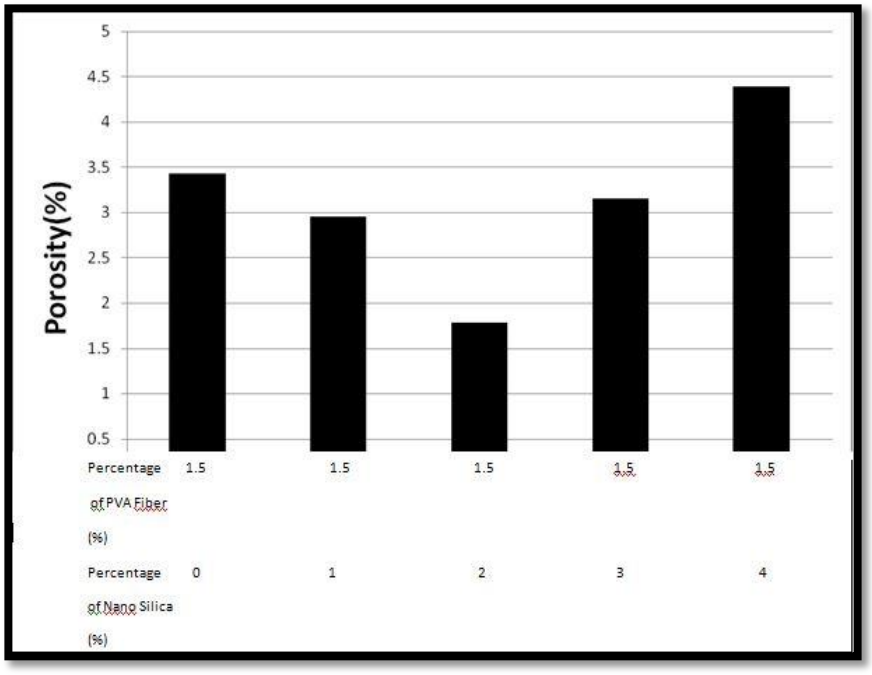

Figure 42: Porosity for 1.5% PVA Fiber

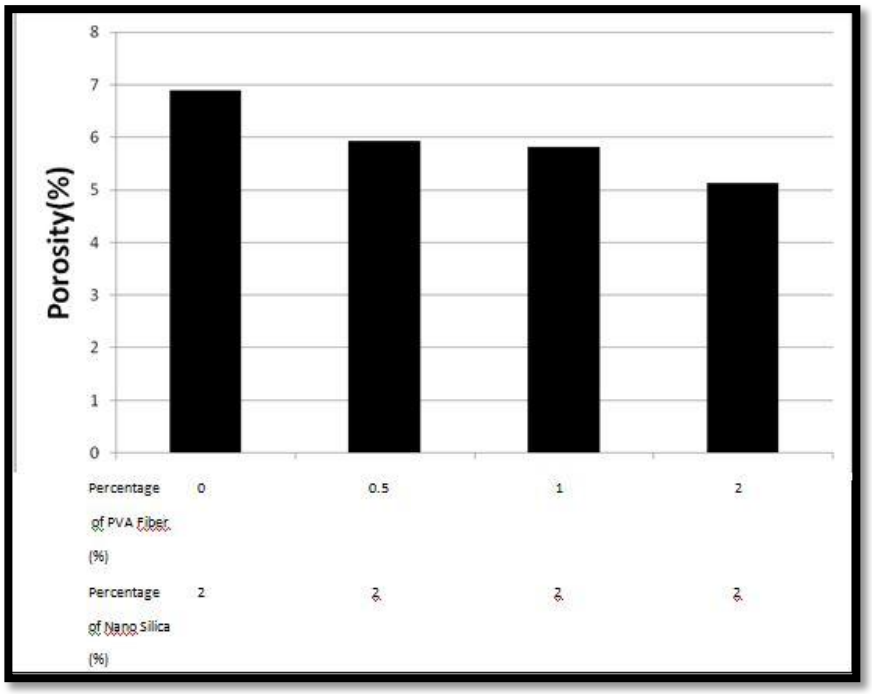

Figure 43: Porosity for 2% Nano Silica

The table above shows the accessible porosity of all 9 samples. It can be observed that as the percentage of Nano Silica increases, the porosity reduces. It shows that the pore structure is more refined and the concrete gets denser as percentage of Nano Silica increases. The concrete may also show denser properties due to the discontinuity of its capillary pore by formation of C-S-H gels. This test pretty much confirms the two previous results of compressive strength and FESEM test. The addition of Nano Silica will reduce the porosity of the cement matrix up to 3%. Up to this percentage, the ITZ of the cement matrix and fiber will reduce and compressive strength result will increase. At 4%, the porosity of the cement matrix will increase thus reducing compressive strength. Figure 43 also shows that as the percentage of PVA Fiber increases, the porosity of the cement will reduce which is in accordance to the increase in compressive strength of the concrete.

CHAPTER 5: CONCLUSION

Due to the increasing usage of ECC as replacement for OPC today, more research has to be done to fully develop the full potential of this concrete. One of the important aspects that must first be studied is the properties of the concrete when exposed to elevated temperatures. The properties of these concrete will be tested by using the Compressive Strength Test to find out strengths of concrete after being exposed to elevated temperatures, FESEM to find out the microstructure of concrete and MIP to look at pore characteristics .

All the compressive strength of the cubes are taken at 28 days of water curing. The compressive strength shows that as the percentage of Nano Silica increases, the compressive strength increases up to 3% addition. At 4% addition, the compressive strength will reduce. Samples that are heated will also show increase in compressive strength up to 300˚C. At 400˚C, the compressive strength will reduce. Therefore, the first objective is achieved.

The morphology and microstructure of the samples are taken using the FESEM . This test identifies the ITZ between the cement matrix and the fiber.

The pore characteristics of the samples is done using the MIP test. It shows the percentage of allowable porosity of each sample. All objectives are achieved.

RECOMMENDATION

As future works, more tests should be employed for the samples for example the flexural test, bending test to better identify the properties of this composite. Also, more samples should be sent for the MIP and FESEM test. Proper planning must be made prior to this as the booking for these lab can be quite difficult at times. This will ensure that a more conclusive result can be interpreted if more samples are tested for their morphology and pore characteristics. Besides that, furnaces should be available for Civil Engineering Department to facilitate with the preparing of samples to the respective temperature.

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