

**EFFECT OF PALM OIL FUEL ASH (POFA) TO  
STEEL FIBRE CONCRETE TOWARDS DRYING  
SHRINKAGE AND HEAT OF HYDRATION**

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**B. ENG (HONS.) CIVIL ENGINEERING**

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TOWARDS DRYING SHRINKAGE AND HEAT OF HYDRATION

RASYIQAH AFIF BINTI MD ISHAK

Thesis submitted in fulfillment of the requirements  
for the award of the  
Bachelor Degree in Civil Engineering

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JUNE 2017

## **DEDICATION**

*Praise be to Allah S.W.T the Lord of the World*

*Who says (interpretation of the meaning)*

*“If you are grateful, I will give you more”*

*[Quran, Ibrahim 14:7]*

*I dedicated this research to my family especially*

*Mr. Md Ishak Bin Said and Mrs. Abadaa Binti Azmi,*

*Rusyda Elya, Rifaie Aliyah, Izzah Radiah and Muhammad Ikhwan*

*for giving me endless support and love.*

*And to my supervisor, Mr. Fadzil Mat Yahaya and all friends*

*for their endless help, patience and encouragement.*

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In the name of Allah S.W.T the most loving and merciful.

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## ABSTRAK

Bahan Pozzolan adalah bahan yang terdiri daripada silika atau alumina. Pozzolan dengan kehadiran air boleh bertindak balas dengan kalsium hidroksida ( $\text{Ca(OH)}_2$ ) untuk membentuk gel Kalsium-Silicate-Hydrate gel (CSH gel) yang bertindak terhadap kekuatan didalam konkrit. Bahan pozzolana dihasilkan oleh pembakaran sisa dari bahan semula jadi atau tiruan. Dalam kajian ini, abu kelapa sawit dikenali sebagai POFA digunakan sebagai bahan pozzolana di dalam konkrit. POFA adalah produk daripada kilang janakuasa dimana bahan buangan kelapa sawit seperti serat kelapa sawit dan tandan buah kosong dibakar untuk menjana kuasa elektrik. Saiz POFA yang digunakan adalah  $63\mu\text{m}$  dan peratus POFA bagi menggantikan sebahagian daripada simen didalam konkrit gentian keluli yang digunakan adalah 10%, 20% dan 30% POFA mengikut berat simen yang digunakan untuk memerhati tindakbalas POFA sebagai pengganti simen kepada konkrit gentian keluli dalam mengurangkan suhu semasa penghidratan dan pengecutan pengeringan disebabkan oleh peratus yang berlainan daripada sebahagian pengganti simen di dalam konkrit gentian keluli. Hasil daripada ujian penghidratan panas menunjukkan bahawa POFA sebagai sebahagian pengganti simen terhadap konkrit gentian keluli mengurangkan jumlah suhu dan melambatkan masa bagi suhu puncak berlaku iaitu pada 30% POFA yang terkandung di dalam konkrit gentian keluli sebagai pengganti simen. Selain itu, hasil ujian pengecutan pengeringan menunjukkan bahawa konkrit yang mengandungi 30% daripada POFA sebagai sebahagian daripada pengganti simen menunjukkan pengecutan yang lebih sedikit berbanding sampel lain. Kajian ini menunjukkan bahawa POFA adalah bermanfaat terutamanya dalam pembinaan konkrit gentian keluli sebagai sebahagian daripada pengganti simen.



## ABSTRACT

Pozzolan material is material that consist of siliceous or aluminous. Pozzolan in the presence of moisture can react with calcium hydroxide ( $\text{Ca(OH)}_2$ ) to form Calcium-Silicate-Hydrate gel (CSH gel) that responsible for the strength in cement based material. Pozzolanic material are produced by combustion of the waste from natural or artificial material. In this study, Palm Oil Fuel Ash (POFA) was utilized as pozzolanic material in concrete. POFA is by product from biomass product of power plant whereby oil palm residue such as fibre, shells and empty fruit bunches are burnt to generate electricity. The size of POFA is  $63\mu\text{m}$  and Portland cement type 1 was partial replace by 10%, 20% and 30% of POFA by weight of cement used in order to observe the behavior of POFA as a cement replacement to steel fibre concrete in reducing temperature during hydration and drying shrinkage due to different percentage of partial cement replacement. The result of heat of hydration tests revealed that POFA as a partial cement replacement towards steel fibre concrete reduces the amount of temperature and delay the time which the peak temperature occurs at 30% of POFA as a cement replacement. Other than that, the drying shrinkage test result showed that the steel fibre concrete containing 30% of POFA as a partial cement replacement was much less shrinkage compared to other sample. This study implies that POFA is a good and beneficial especially in the construction of steel fibre concrete as a cement replacement.

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## LIST OF SYMBOLS

%	Percentage
$\mu\text{m}$	Micrometre
$\text{mm}^2$	Millimetre Square
$\text{m}^3$	Meter Cube
g	Gram
$^{\circ}\text{C}$	Degree Celsius

## LIST OF ABBREVIATIONS

UMP	Universiti Malaysia Pahang
FKASA	Fakulti Kejuruteraan Awam dan Sumber Alam
ASTM	American Society for Testing and Materials
POFA	Palm Oil Fuel Ash
OPC	Ordinary Portland Cement
CO <sub>2</sub>	Carbon Dioxide
SiO <sub>2</sub>	Silicon Dioxide
Al <sub>2</sub> O <sub>3</sub>	Aluminium Oxide
CaO	Calcium Oxide
MgO	Magnesium Oxide
SO <sub>3</sub>	Sulphur Trioxide
Na <sub>2</sub> O	Sodium Oxide
K <sub>2</sub> O	Potassium Oxide
LOI	Loss on Ignition
CSH	Calcium-Silicate-Hydrate
MPa	Mega Pascal
Min	Minute



# CHAPTER 1

## INTRODUCTION

### 1.1 Background

Cement is an important material to produce a concrete. Figure 1.1 shows that Malaysia's cement production fluctuated since 1986 to 2016 but for overall trending, it increased from year 1986 to 2016. The growth of cement production generally because of the demand from the construction industry as it one of the important material in construction.

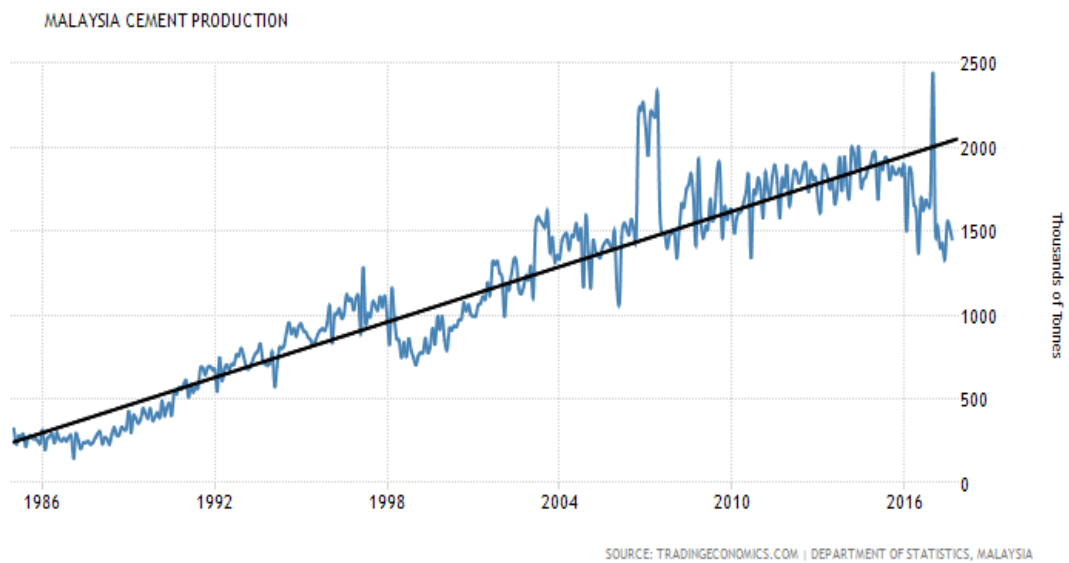


Figure 1.1 Malaysia Cement Production  
Source: ("Malaysia Cement Production," 2018)

Concrete refers to a mixture of cements, aggregates and sand. It defined as an important material in construction's world especially to construct a building, bridge and pavement. A good quality of concrete is a must in order to build a strong structure. Cement is important material in producing a good concrete. It act as binder or glue to bind all material which is aggregate and fines aggregate to become concrete. There were many types of cement but Ordinary Portland Cement (OPC) were commonly used in most every construction.

Steel Fibre Concrete increasingly been used in construction industry. Fibre were generally utilized in concrete to manage the plastic shrink cracking and drying shrink cracking. (Aggeliki K., 2011) They were many types of steel fibre such as hooked ends steel fibre, micro hooked ends steel fibre, and cooper coated steel fibre. Every type of steel fibre has different purposes such as micro hooked ends steel fibre were more suitable used for surface of high building and hooked end steel fibre widely used in construction field for concrete reinforcement as this type of steel fibre ensure the best status between steel fibre and concrete.

During the hardening of the concrete, hydration process occur inside the concrete. Hydration process were process to binding all the ingredient of concrete which are fine aggregate, course aggregate, cement and water. During this hydration process, cement reacted with water to produce one gel that act as a binding agent which is Calcium-Silicate-Hydrate gel (CSH gel). This gel will bind the ingredient of concrete and during hydration process, heat released. Based on Neville, 2011 heat that occur during hydration process, the quantity of heat of unhydrated cement that evolve upon complete hydration at a given temperature. The heat of hydration consists of the chemical heat of the reactions of hydration and the heat of adsorption of water on the surface of the gel formed by the processes of hydration.

From Figure 1.2, Malaysia known as one of the largest palm oil producer and exporter in the world after Indonesia and from Figure 1.3 it can be seen that the total 5.74 million hectare of palm oil plantation area in Malaysia with 2.68 million hectare at Peninsular Malaysia and 3.06 million hectare of palm oil plantation area at Sabah and Sarawak. To generate electricity, all the waste from palm oil residue such as fibre, shells and empty fruit bunches were burnt into the power plant and Palm Oil Fuel Ash (POFA) was biomass product of power plant (Jamo, Abdu, & Pahat, 2015). Then, POFA generally disposed to the landfill.

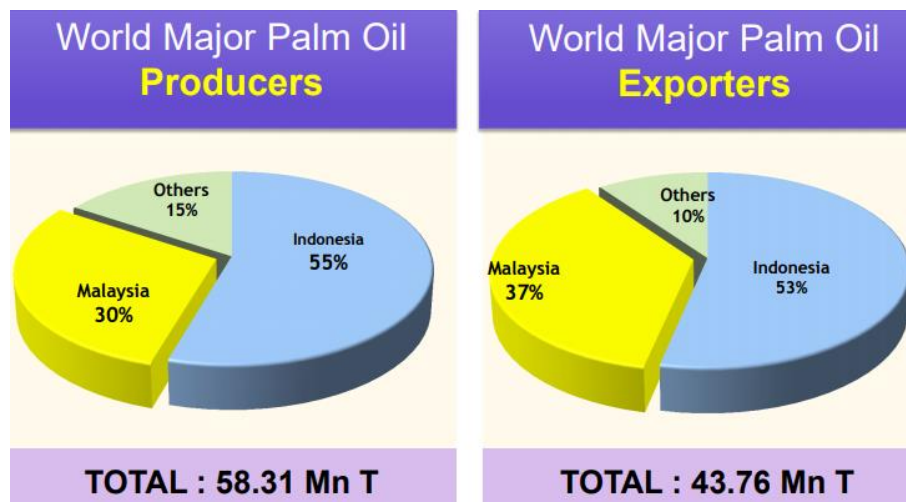


Figure 1.2 World Major Palm Oil Producers and Exporters

Source: (Din, 2017)

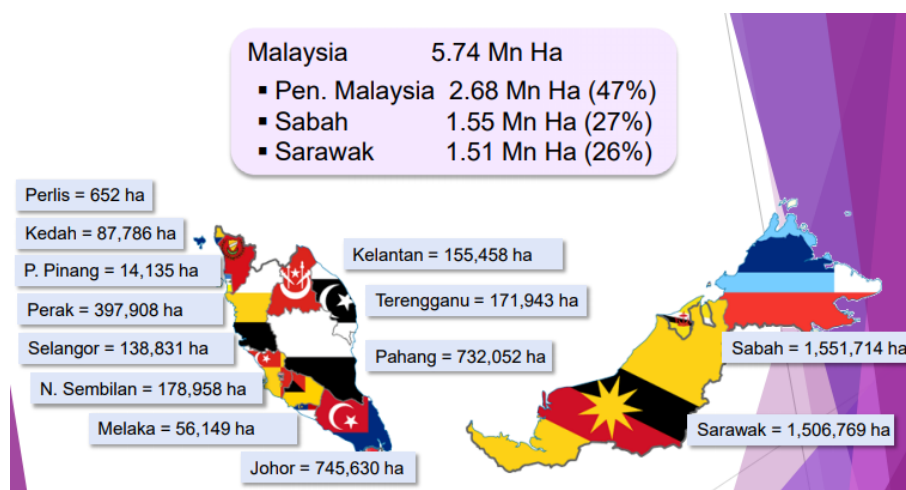


Figure 1.3 Oil Palm Planted Area in Malaysia

Source: (Din, 2017)

## 1.2 Problem Statement

The proposed study based on the problem that occur in Malaysia. In this era of globalization, Malaysia known as one of the biggest producers and exporter of palm oil and palm oil products. According to Malaysia Palm Oil Council (MPOC), Malaysia currently accounts for 39% of world palm oil production and 44% of world exports (Dr. Amin Idrees, 2014). Today, 4.49 hectares of land in Malaysia were all oil palm cultivation. Palm oil was extracted from oil plant tree and all the waste product was used as biomass energy. Palm oil fuel ash (POFA) was a by-product from biomass thermal power plants whereby oil palm residues were burnt to generate electricity (Jamo et al., 2015). But, increasing of POFA lead to land fill problem as POFA always increasing from time to time because not been used.

Furthermore, production of cement will be increase year by year due to the demand from construction industry. This will be the problem to the environment as production of cement generates Carbon Dioxide ( $\text{CO}_2$ ).  $\text{CO}_2$  known as a result of a chemical conversion process used in the production of clinker, a component of cement which is limestone ( $\text{CaCO}_3$ ) converted to lime ( $\text{CaO}$ ).

Besides that, thermal crack always occurs during construction of mass concrete. Thermal crack occur during hydration process of concrete that can affect the integrity of the concrete. Thermal crack occur when the internal temperature of the concrete higher that the surface of concrete and make internal stress that lead to crack of concrete.

Therefore, a research about used of POFA as a partial replacement of cement to identify the benefit and useful of POFA into construction industry. Besides that, this effort can be reduced the land fill problem that occurred by POFA and thermal crack that often occur during construction of mass concrete. This research focused on the utilization of POFA in the steel fibre concrete production.

### **1.3 Research Objective**

The objectives of this study are:

- i. To measure the heat of hydration of the steel fibre concrete by using POFA as a partial cement replacement.
- ii. To measure the drying shrinkage of the steel fibre concrete by using POFA as a partial cement replacement.

### **1.4 Scope of Study**

The main purpose of this study to determine the heat of hydration and drying shrinkage of steel fibre concrete focusing with varies percentage amount of POFA which were 10%, 20% and 30% cement replacement. The test conducted by using 63µm size of POFA which the constant size for the entire specimens. Concrete mix prepared for steel fibre concrete specimen and also steel fibre concrete added with POFA for each 10%, 20% and 30% POFA as a partial replacement. The concrete then poured into 300×300×300mm mould for heat of hydration test. The heat of hydration test conducted by using thermocouple and data logger.

Small amount of the concrete then poured into 25×25×285mm steel mould to conduct drying shrinkage test. Four set of steel fibre concrete specimen and also four set of steel fibre concrete added with POFA for each percentage (10%, 20% and 30%). The drying shrinkage of each specimen assessed on 7, 28 and 90 days after curing. Curing method that involved in this test was air curing method. The test involves in this study had been conducted at Fakulti Kejuruteraan Awam dan Sumber Alam (FKASA) structure laboratory, Universiti Malaysia Pahang.

## 1.5 Research Significant

This research important to determine whether POFA as pozzolanic material suitable to be a partial cement replacement to steel fibre concrete or not. Drying shrinkage test and heat of hydration test conducted to measured steel fibre concrete with POFA as partial cement replacement with normal steel fibre concrete. If the results of this two test for steel fibre concrete with POFA same or much better than the normal steel fibre concrete, it shows that steel fibre concrete with POFA as a good solution for problem in this research.

Besides that, using POFA as a cement replacement can reduced used of cement in construction industry. Reduced used of cement means reducing carbon dioxide. It because of during cement production, carbon dioxide released during the production of clinker which calcium carbonate ( $\text{CaCO}_3$ ) heated in a rotary kiln at temperature 600-900°C to induce a series of complex chemical reactions. (Guidance, n.d.) The simplified stoichiometric relationship shown as  $\text{CaCO}_3 + \text{heat} \rightarrow \text{CaO} + \text{CO}_2$ .

Last but not least, this research important to determine whether POFA as a cement replacement can reduce temperature of concrete during hydration process. Reduced temperature during hydration process means reducing risk of thermal crack that can occur to the steel fibre concrete that can intrude the integrity of the concrete. Indirectly reduced the drying shrinkage that can occur to the steel fibre concrete.

## **CHAPTER 2**

### **LITERATURE REVIEW**

#### **2.1 Introduction**

Literature review is about various studies that discussed the relevant material which related to this project which was Palm Oil Fuel Ash (POFA), concrete, steel fibre, cement, aggregate and water. The characteristics were discussed based on previous studies.

## **2.2 Palm Oil Fuel Ash (POFA)**

Combustion of palm oil industry waste for the generation of electricity lead to Palm Oil Fuel Ash (POFA) produced from the power plants. Then, POFA generally disposed to the landfill and this cause traffic hazard besides potential of health hazard. Due to its high pozzolanic characteristic, POFA suitable to be the cement replacement for a concrete to decrease the landfill problem (Thomas, Kumar, and Arel, 2017).

Concrete with POFA as a partial replacement can reduced the temperature rise in the concrete and delayed the time to reach peak temperature (Abdul Awal & Warid Hussin, 2011). POFA as a cement replacement also a good material that can control heat of hydration that occur in the concrete during hydration process (Awal & Shehu, 2013).

### **2.2.1 Properties of Palm Oil Fuel Ash (POFA)**

Properties of POFA divide into physical properties and chemical composition. The physical properties of POFA were given in Table 2.1 by referring to three resources. POFA normally grey colour in bulk that becomes dark with increased proportion of unburned carbon (Awal and Shehu, 2013). POFA needs to be process before being used in a concrete mix due to the impurities accompanying by POFA production. POFA needs to be sieved in order to obtain the ash particle without any unburned fibres and to increased pozzolanic reactivity (Al-Mulali et al., 2015). Figure 2.1 to Figure 2.3 shows different Scanning Electron Microscopy (SEM) image of different particle size of POFA.

Table 2.2 shows chemical composition of POFA used by several researchers. Notable variations in the physical and chemical composition of the different POFA used by several researchers may cause by different factor such as burning temperature, particle fineness, part of the palm oil tree burned, etc. (Thomas et al., 2017).



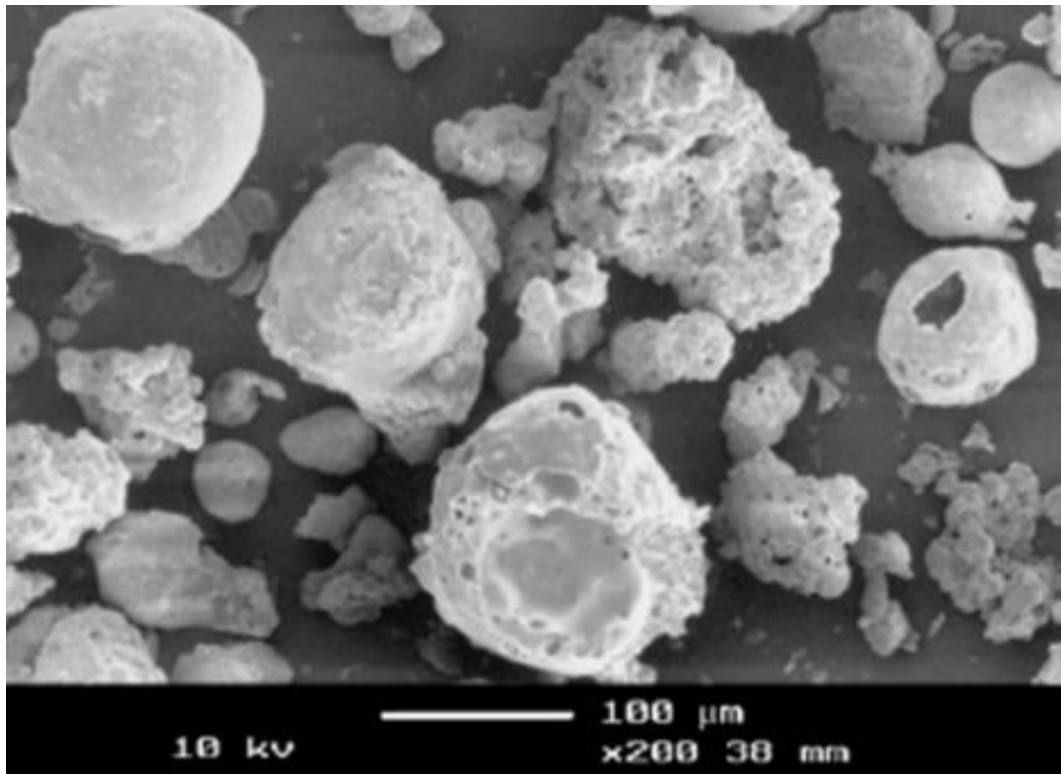


Figure 2.1 Original Size of POFA particles

Sources: (Al-Mulali et al., 2015)

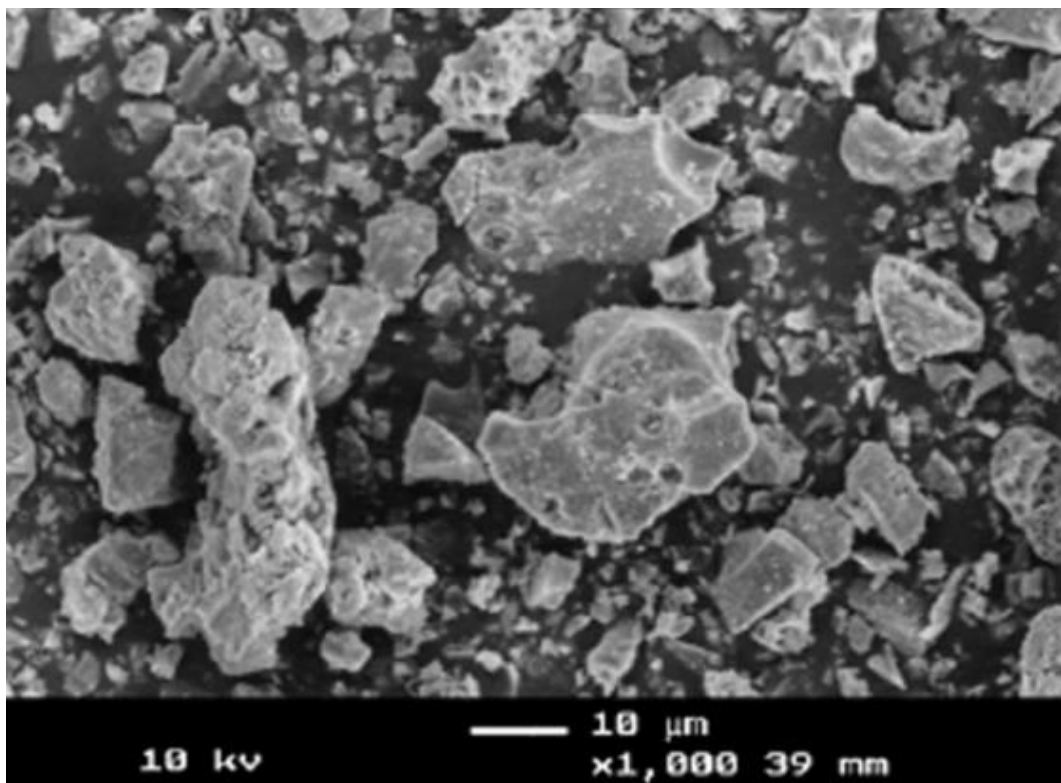


Figure 2.2 Medium Size of POFA particles

Sources: (Al-Mulali et al., 2015)

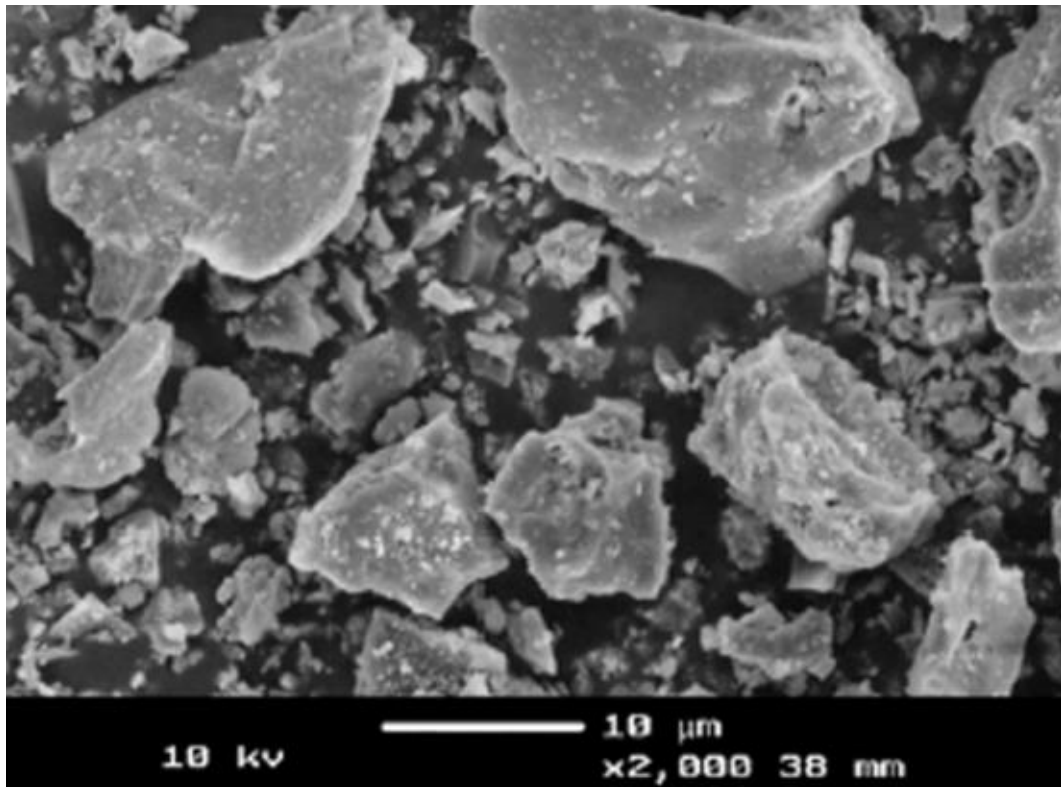


Figure 2.3 Small Size of POFA particles

Sources: (Al-Mulali et al., 2015)

Table 2.1 Physical Properties of Palm Oil Fuel Ash (POFA)

Physical Properties	(Awal and Shehu, 2013)	(Abdul Awal & Warid Hussin, 2011)	(Lim et al., 2015)
Specific Gravity	2.42	2.22	2.42
Blaine Fineness (cm <sup>2</sup> /g)	4930	5200	4935
Amount retained on 45 µm sieve (%)	1.05	–	4.98

Sources: (Awal and Shehu, 2013; Abdul Awal & Warid Hussin, 2011; Lim et al., 2015)

Table 2.2 Chemical Composition of Palm Oil Fuel Ash (POFA)

Chemical Composition	(Awal and Shehu, 2013)	(Abdul Awal & Warid Hussin, 2011)	(Lim et al., 2015)
Silica (SiO <sub>2</sub> )	59.62	43.60	53.5
Aluminium Oxide (Al <sub>2</sub> O <sub>3</sub> )	2.54	11.40	1.9
Iron (III) Oxide (Fe <sub>2</sub> O <sub>3</sub> )	5.02	4.70	1.1
Calcium Oxide (CaO)	4.92	8.40	8.3
Magnesium Oxide (MgO)	4.52	4.80	4.1
Potassium Oxide (K <sub>2</sub> O)	7.52	3.5	6.5
Sodium Oxide (Na <sub>2</sub> O)	0.76	0.39	–
Phosphorus Pentoxide (P <sub>2</sub> O <sub>5</sub> )	3.58	–	–
Sulphur Trioxide (SO <sub>3</sub> )	1.28	2.8	2.36
LOI	8.25	18.0	20.9
SiO <sub>2</sub> +Al <sub>2</sub> O <sub>3</sub> +Fe <sub>2</sub> O <sub>3</sub>	67.18	59.70	56.5

Sources: (Awal and Shehu, 2013; Abdul Awal & Warid Hussin, 2011; Lim et al., 2015)

## **2.3 CONCRETE**

Concrete is the most widely used in construction industry due to its durability, versatility, sustainability and economy. Concrete made from two components which were aggregate and paste. Aggregates were generally made up from two groups which were fine and course aggregate that occupy 60 to 80 percent of volume of concrete. Besides that, paste composed from cement and water and ordinarily constitutes 20 to 40 percent of total volume of concrete.

### **2.3.1 Properties of Concrete**

The concrete has its own properties during fresh concrete and hardened concrete. During fresh concrete stage, concrete can be moulded into any shape and it is in the plastic state. The following were the important properties of the concrete:

1. Setting of Concrete.

Setting of concrete means the hardening of concrete before it gains its strength. Setting of concrete have many factors that affecting the setting of concrete which were water cement ratio, temperature, cement content, type of cement, admixture, and amount and type of aggregate.

2. Workability.

Workability of concrete referred as the ease of concrete to be mould, placed and transported. To measure the workability of the concrete, slump concrete test conducted according to Malaysian Standard Testing Of Concrete - Part 1: Fresh Concrete - Section 2: Slump Test (MS26-1-2:2009, 2010). There were 3 type of slump result which were true slump, shear slump and collapse slump as shown as in Figure 2.4. There were many factors that can affect concrete workability which were water cement ratio, type and amount of aggregate, type and amount of cement, and chemical admixture.

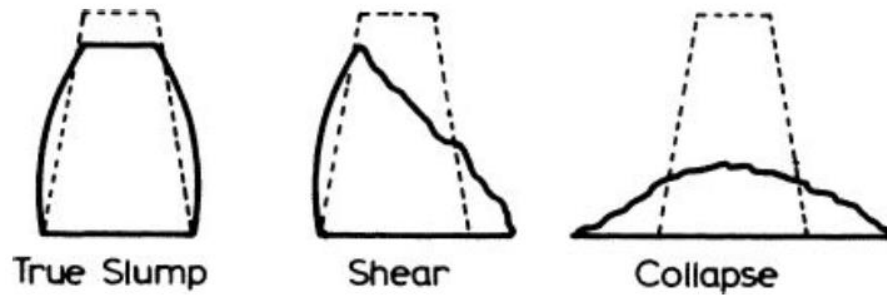


Figure 2.4 True slump, Shear slump and Collapse slump

Source: (Neville, 2011)

### 3. Bleeding and Segregation.

Bleeding of concrete referred as water gain in the concrete. Concrete bleeding occurred when there was water from the concrete comes out to the surface of concrete. Bleeding occurred when there was highly wet mix of concrete. Besides that, segregation defined as the separation of the constituent materials of concrete.

### 4. Hydration of Concrete.

Hydration of concrete is the process where the strength of concrete developed. Hydration process of concrete continuing for long time as long as there is cement and water reacts together to form Calcium-Silicate-Hydrate (CSH) gel that combine all the ingredient of the concrete and make the concrete denser. As denser the concrete, the strength of the concrete also increases.

### 5. Air Entrainment.

Air entrainment of concrete reduced the density of the concrete and reduce the strength of the concrete.

## **2.4 Cement**

Cement is the important and basic material that used in construction industry to produce a concrete as it act as a binder. There were so many types of cement that based on their own advantages and purposes. In Malaysia, Portland Cement (PC), Sulphate Resisting Portland Cement (SRPC) and Rapid Hardening Portland Cement (RHPC) were the most popular and usually used in construction industry. Generally, cement produced from calcareous material which is limestone together with clay and sand.

### **2.4.1 Ordinary Portland Cement (OPC)**

The most common cement used in almost every construction in Malaysia and also around the world is Ordinary Portland Cement (OPC). It is the common type of powdery cementitious building material made from finely pulverized alumina, iron oxide, lime, magnesia, and silica burnt in a kiln. OPC known as basic ingredients of concrete, mortar and plaster in construction. Normally, OPC used for reinforced concrete buildings, pavements and bridges where the soil conditions were normal. The name of Portland cement given by British engineer, Joseph Aspdin in 1825 due to similarity in colour and quality when it hardens like Portland stone.

## 2.4.2 Type of Portland Cement

In Malaysia, the Portland Cement that used must fulfil ASTM C150 Standard Specification for Portland Cement (ASTM International, 2011). ASTM C150 have five types of Portland Cement and the table below showed the general characteristics type of Portland Cement based on ASTM C150.

Table 2.3 Type of Portland Cement

Type	Classification	Characteristics	Application
<b>I</b>	General purpose	Fairly high C <sub>3</sub> S content for good early strength development	General construction (most buildings, bridge, pavements, precast units and etc.)
<b>II</b>	Moderate sulphate resistance	Low C <sub>3</sub> S content (<8%)	Structure exposed to soil or water containing sulphate ions.
<b>III</b>	High early strength	Ground more finely, may have slightly more C <sub>3</sub> S	Rapid construction, cold weather concreting.
<b>IV</b>	Low heat of hydration (slow reaction)	Low C <sub>3</sub> S content (<50%) and C <sub>3</sub> A	Massive structure such as dams, now rare.
<b>V</b>	High Sulphate Resistance	Very low C <sub>3</sub> A content (<5%)	Structure exposed to high level of sulphate ions.
<b>White</b>	White color	No C <sub>4</sub> AF, low MgO	Decorative (otherwise has properties similar Type I)

Sources: (“Types of Portland Cement,” 2017)

### 2.4.3 Physical Properties of Ordinary Portland Cement

Table 2.4 Physical Properties of Ordinary Portland Cement (OPC)

Physical Properties	OPC
Specific Gravity	3.28
Blaine Fineness (cm <sup>2</sup> /g)	3990
Amount retained on 45 µm sieve (%)	–

Sources: (Awal & Shehu, 2013)

### 2.4.4 Chemical Composition of Ordinary Portland Cement (OPC)

Table 2.5 Chemical Composition for Ordinary Portland Cement (OPC)

Chemical Composition	OPC
SiO <sub>2</sub>	20.40
Al <sub>2</sub> O <sub>3</sub>	5.20
Fe <sub>2</sub> O <sub>3</sub>	4.19
CO	62.39
MgO	1.55
K <sub>2</sub> O	0.005
Na <sub>2</sub> O	0.75
P <sub>2</sub> O <sub>5</sub>	0.28
SO <sub>3</sub>	2.11
LOI	2.36

Sources: (Awal and Shehu, 2013)



## 2.5 Steel Fibre

Steel fibre is a composite material and can led to improvement of concrete mechanical properties. Furthermore, the brittle behaviour and the energy absorption capacity can be improved with the addition of steel fibre concrete into the concrete (Abbass, Khan, & Mourad, 2018). There were many types of steel fibre concrete that used in construction industry that have their own specialty and importance such as hooked end steel fibre, micro hooked end steel fibre, glued steel fibre, etc. Figure 2.5 shows hooked end steel fibre, micro hooked end steel fibre and glued steel fibre.



Figure 2.5 Hooked End, Micro Hooked End and Glued Steel Fibre

Source: (“Henan Zhancheng Steel Fibre Co.,” 2018)

### 2.5.1 Steel Fibre Concrete

As it commonly known, concrete weak in tension (Nawy, 2008). Additions of steel fibre into the mix of concrete lead to improve the durability of concrete to increase strength of the concrete in tension load. Composite material that mixed cementitious matrix and discontinuous reinforcement was what that describe steel fibre concrete (Marcos Meson, 2016). There was previous study that study about mechanical properties of steel fibre concrete vary respect to temperature changes and the results shows that beam toughness values with high temperature after three points bending significantly withstand lowest load before failure occurs compared to the ambient temperature for 24 hours testing (Richardson & Ovington, 2017). Figure 2.6 shows the result from Richardson & Ovington, 2017 studies. From the results, Richardson & Ovington, 2017 concluded that as decreasing temperature, increasing the toughness of fibre reinforced concrete.

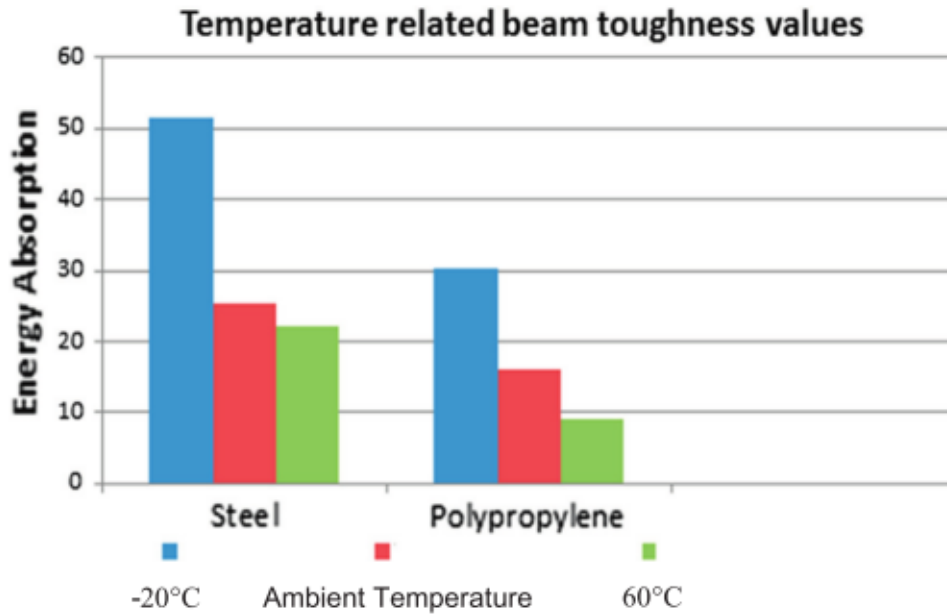


Figure 2.6 Results Average Total Energy Absorption After Three Point Bending  
Sources: (Richardson & Ovington, 2017)

## 2.6 Water

Water is an important constituent in concrete as it reacted with cement, form a paste and bind all the aggregate together. The water causes the hardening of concrete through a process called hydration. The quantity of water in the mix plays a vital role on the strength of concrete. Low water cement ratio lead to high strength but low in workability and high water cement ratio lead to decrease the strength of concrete but good in workability. The importance of water in concrete work were:

- a) To develop adhesion between the paste of cement and all the ingredient of concrete.
- b) To impart workability of concrete to facilitate placing the concrete in the desired position and shape.
- c) Water needed when hydration process occur. Hydration of cementing materials to set and harden during the period of curing.

## **2.7 Aggregate**

Aggregate is one of the ingredients of the concrete. Aggregate in concrete act as structure filler and occupies most of the volume of concrete at least three-quarters of the volume of the concrete. Aggregate originally viewed as an inert material dispersed throughout the cement paste largely for economic reasons (Neville, 2011). Aggregate generally contains pores.

## **2.8 Shrinkage**

Shrinkage known as autogenous volume change and it occur in the interior of concrete mass. The contraction of the cement paste restrained by the rigid skeleton of the concrete that already hydrate and also by the aggregate particle (Neville, 2011). Shrinkage can be divided into two types which were plastic shrinkage and drying shrinkage. Plastic shrinkage often occurs during the fresh stage of concrete and drying shrinkage can be seen after the concrete hardened and it occurs in a long term as long as there was water in the concrete.

### **2.8.1 Drying Shrinkage**

Withdrawal of water from concrete to the atmosphere causes drying shrinkage and this movement irreversible. The concrete tends to have change in volume when drying shrinkage occur (Neville, 2011). Drying shrinkage known as complicated phenomenon that involving several different mechanism and these mechanism were dominant at different range of internal pore humidity (Xiao Jian et al., 2007). Based on previous study, the increasing of pozzolana material to the concrete as a partial cement replacement can reduce the drying shrinkage due to the pore refinement of the fine ash particle (Thomas et al., 2017). When the pore of the concrete becomes smaller, it can control the water from losing to the atmosphere and indirectly reduce the drying shrinkage.

## **2.9 Heat of Hydration in Concrete**

Heat of hydration is heat that given off during the chemical reaction as the cement hydrates and become hardened. Concrete achieves its strength through a chemical process called hydration (Hodgson, 2000). The hydration of cement compounds known as exothermic. According to Abdul Awal & Warid Hussin, 2011 heat generated within the concrete matrix during hydration when cement hydrated the compounds reacted with water to acquire stable low energy states and the process accompanied by the release of energy in the form of heat. Quantity of heat developed during complete hydration of a certain amount of unhydrated cement at a given temperature defined as heat of hydration (Neville, 2011).

## **CHAPTER 3**

### **METHODOLOGY**

#### **3.1 Introduction**

This chapter clearly defines the research methods used to conduct the study. In this chapter, the explanation of the material used and test method followed in conducting various experiment investigation. Laboratory experiments had been conducted to identify the effect of using POFA as a partial cement replacement material to the steel fibre concrete.

Several experiments had been conducted to study the effect of POFA as the cement replacement to steel fibre concrete towards drying shrinkage and heat of hydration. All the testing conducted according to the ASTM standard.

### 3.2 Flow Chart of Research

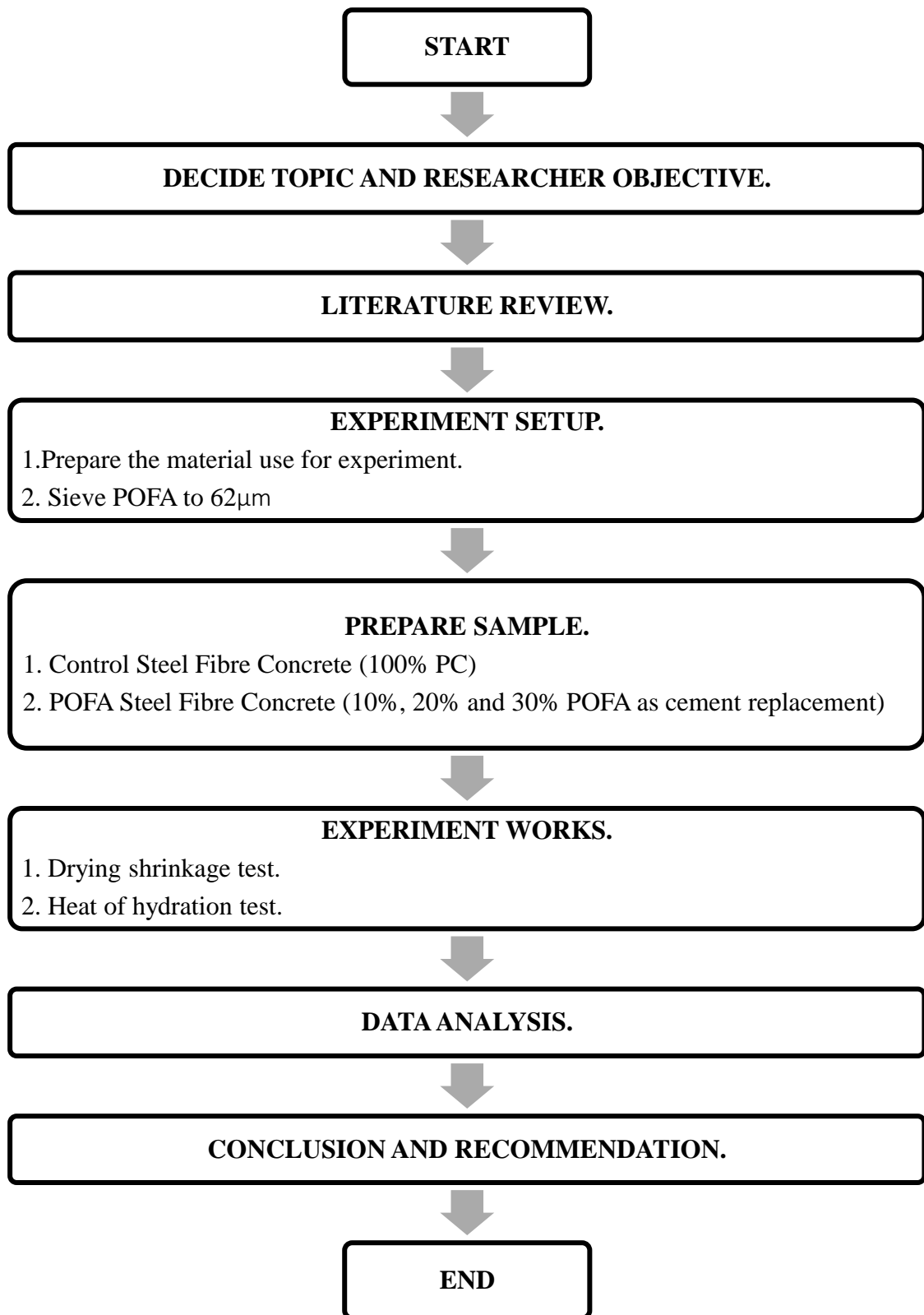


Figure 3.1 Research Methodology Flow Chart

### **3.3 Preparation Materials**

The materials which were Ordinary Portland Cement (OPC), steel fibre, water, aggregate and POFA were used in the preparation of the specimen in this study. All the materials list below.

#### **3.3.1 Ordinary Portland Cement (OPC)**

Ordinary Portland Cement (OPC) that used was OPC with type 1 Portland cement as in ASTM C 150-07.

#### **3.3.2 Steel Fibre**

Steel fibre used was hooked end steel fibre type with length 60mm and diameter of 0.75mm. The picture of steel fibre used shown in Figure 3.3.2.



Figure 3.2 Hooked End Steel Fibre

### 3.3.3 Water

Water that used during mixing all the ingredients of concrete should have no impurities that may affect the chemical reaction between the compound of cement and water. In this study, the tap water was used.

### 3.3.4 Aggregate

In this study, two types of aggregate were used which were coarse aggregate and fine aggregate. The coarse aggregate particle distribution shown in Figure 3.3 and the maximum size of coarse aggregate was 20mm shown in Figure 3.5. Next, fine aggregate that passing 600 $\mu$ m sieve for particle distribution shown in Figure 3.4 that sieve with sieve shaker as shown in Figure 3.6.

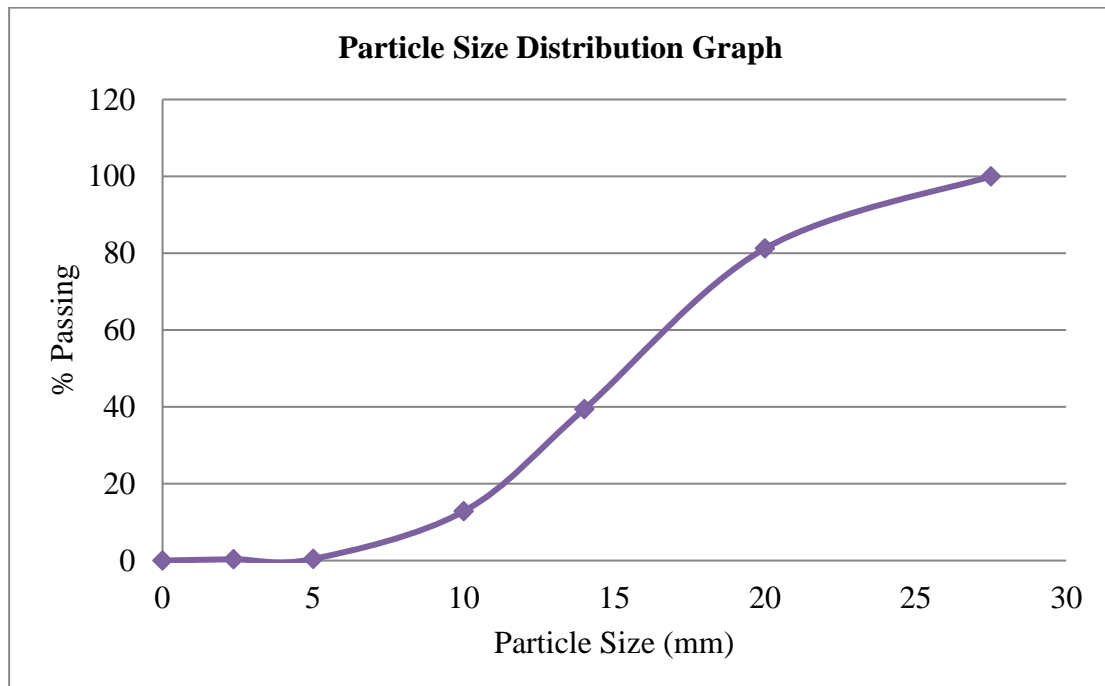


Figure 3.3 Particle Size Distribution of Coarse Aggregate



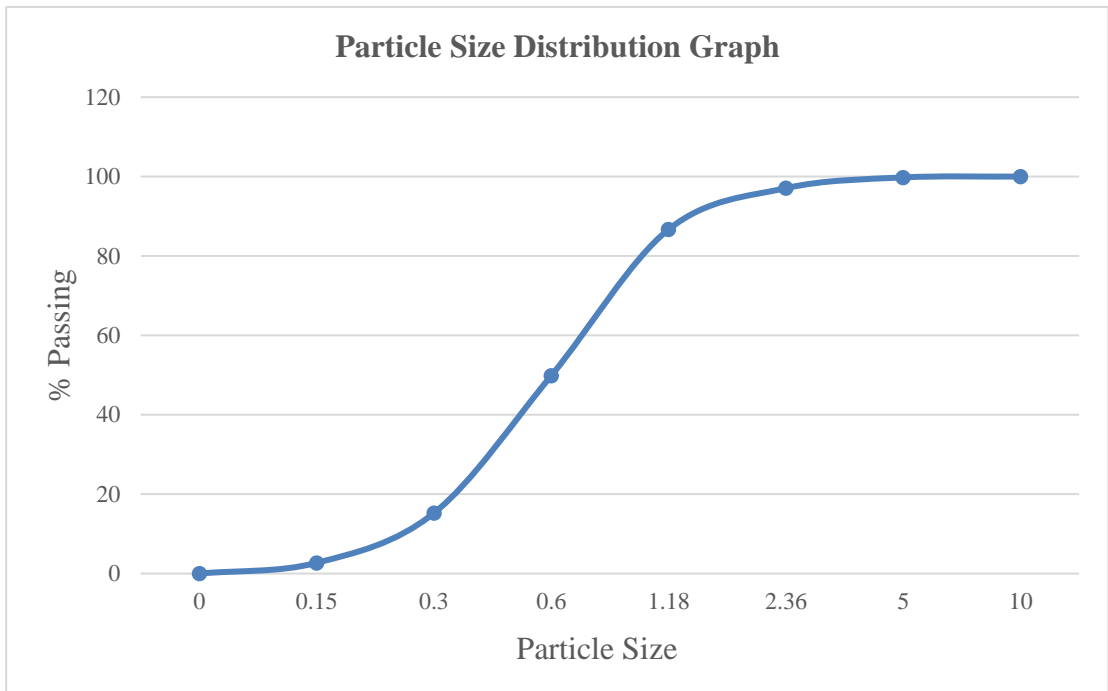


Figure 3.4 Particle Size Distribution of Fine Aggregate



Figure 3.5 Course Aggregate with maximum size of 20mm



Figure 3.6 Fine Aggregate Sieve with Sieve Shaker

### 3.3.5 Palm Oil Fuel Ash (POFA)

In this study, POFA was obtained from the nearest factory of palm oil which at Lepar Hilir Palm Oil Mill, Gambang, Kuantan.

#### 3.3.5.1 Preparation of POFA

The POFA obtained were dried into the oven within 24 hours and sieve with sieve size  $63\mu\text{m}$ . POFA that passing  $63\mu\text{m}$  sieve was stored into the container. Figure 3.5 shown POFA after drying and sieves with  $63\mu\text{m}$ .

The percentage of POFA used were 10%, 20% and 30% from the amount of cement weight. The criteria of POFA according to Pozzolanic Material ASTM C618-12a (ASTM, 2010). Table 3.1 shows the chemical composition that used in this study. The chemical composition shows in Table 3.1 shows that summation of silicon dioxide, aluminium oxide and iron oxide was more than 52%. Based on ASTM C618-12a, this POFA was in class C Pozzolanic Material and can be used in this study.

Table 3.1 Chemical Composition of POFA

Chemical Composition	Percentage (%)	
	POFA	OPC
Silicon Dioxide (SiO <sub>2</sub> )	42.24	23.00
Aluminium Oxide (Al <sub>2</sub> O <sub>3</sub> )	4.48	4.00
Iron Oxide (Fe <sub>2</sub> O <sub>3</sub> )	6.09	3.50
Potassium Oxide (K <sub>2</sub> O)	7.76	0.51
Magnesium Oxide (MgO)	4.02	1.23
Calcium Oxide (CaO)	5.63	64.0
Sodium Oxide (Na <sub>2</sub> O)	0.10	0.25

### 3.4 Preparation of Mould

The purpose of the present investigation to study the effect of POFA as a partial cement replacement. Two type of mould were involved in this study which were wood mould and steel mould. The mould used for heat of hydration test was wood mould with size 300×300×300mm and for drying shrinkage test was steel mould with size 25×25×285mm. Wood mould should be cover with silicone gel to avoid fresh concrete can pass through the space has in the wood. Figure 3.7 shows the work to cover the wood mould with silicone gel. The mould for heat of hydration test and drying shrinkage test were shown in Figure 3.8 and Figure 3.9 respectively.



Figure 3.7 Work to Cover the Wood Mould with Silicone Gel



Figure 3.8 Wood Mould for Heat of Hydration Test



Figure 3.9 Steel Mould for Drying Shrinkage Test

### 3.5 Mix Proportion of Concrete

For this study, the design mix that used was 30 N/mm<sup>2</sup> for specified characteristic strength at 28 days. The free water ratio was 0.54 used for this study and the mixing of POFA based on a simple approach of direct replacement was adopted. Table 3.2 below shows the mix proportion of 1 m<sup>3</sup> quantity of the material used in the mix concrete based on the amount of the specimen and 20% for wastage usage.

Table 3.2 Mix Proportion of Concrete for 1m<sup>3</sup>

Materials	0% POFA	10% POFA	20% POFA	30% POFA
OPC (kg)	328	295	262	229
POFA (kg)	-	33	66	99
Fine Aggregate (kg)	785	785	785	785
Course Aggregate (kg)	1177	1177	1177	1177
Water (kg)	190	190	190	190
Steel Fibre (kg)	7	7	7	7

### 3.6 Laboratory Testing

The concrete was mixed by using the vertical shaft concrete mixer that provided at FKASA laboratory, UMP. All the material was weighed by using digital balance in kilogram. The test involved in this study followed the ASTM standard for drying shrinkage and for heat of hydration test, followed paper from Awal & Shehu, 2013.

### 3.6.1 Heat of Hydration Test

Heat of hydration test was conducted by using thermocouple and data logger. The specimen used in this test with dimension of 300×300×300 mm as explained in preparation of mould.

After mixed the ingredient of steel fibre concrete, the fresh steel fibre concrete poured into the mould and heat of hydration tests begin. Figure 3.10 shows the heat of hydration test arrangement. The heat of hydration test conducted after the cement poured into the formwork and the data collected by the data logger within 24 hours of the test.



Figure 3.10 Heat of Hydration Test Arrangement

### 3.6.2 Drying Shrinkage Test

For drying shrinkage test, a small amount of concrete were used. The fresh steel fibre concrete then poured into the steel mould with dimension 25×25×285 mm as explained in preparation of mould. The curing method used for drying shrinkage test was air curing method and the test conducted at the age of the steel fibre concrete at 7 days, 28 days and 60 days. Figure 3.11 shows the mould of the drying shrinkage with steel fibre concrete.



Figure 3.11 Steel Fibre Concrete in Steel Mould

The initial length of the steel mould was 285mm and all the measurement measured by Vernier Calliper. This test followed the standard of ASTM C157 (ASTM C157, 2010).

## **CHAPTER 4**

### **RESULTS AND DISCUSSION**

#### **4.1 Introduction**

This chapter focused on the results that were obtained from heat of hydration test and drying shrinkage test. All tests were using different percentage of POFA as a partial cement replacement. In this study, the POFA used as a partial cement replacement material in steel fibre concrete to determine the external and internal temperature and the difference length that can occur because of the shrink of the specimen. The experiment data shown in the table and graph for better resolution for analysis and comparison.



## 4.2 Heat of Hydration Test

The heat of hydration test were run after the concrete mixed. Result of internal temperature for each percentage of POFA (Appendix A1, A2, A3 and Appendix A4). The internal temperature of concrete was measured from the thermocouples that locate at the centre inside the concrete. Figure 4.1 shows the internal temperature of the cement concrete that consist of 0% POFA.

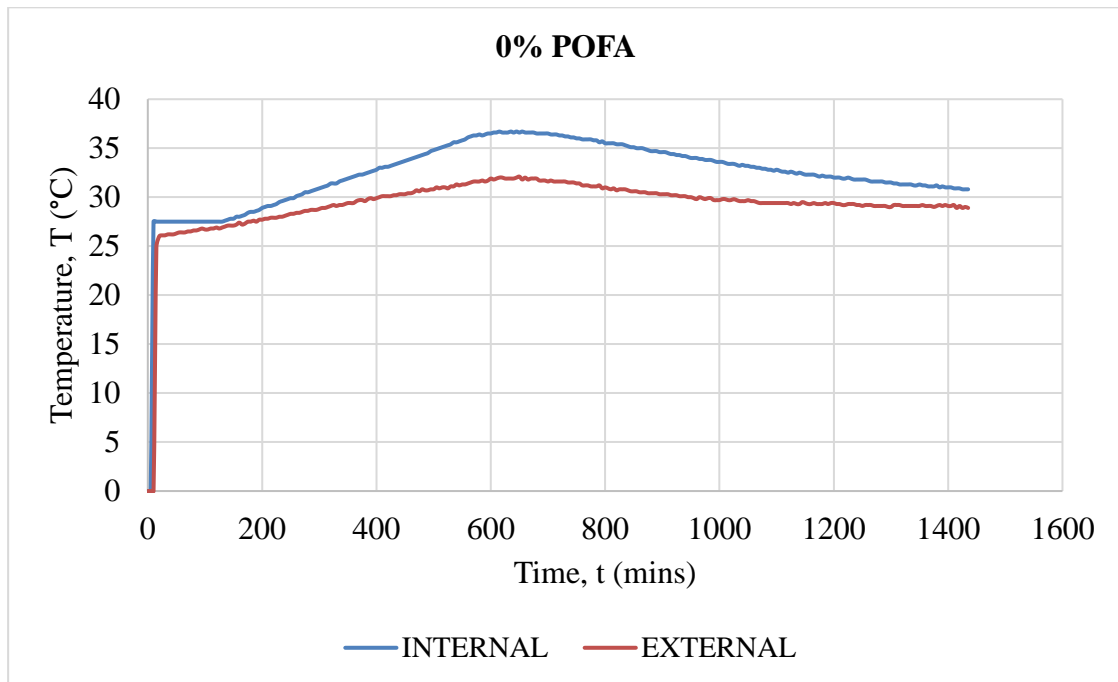


Figure 4.1 Internal and External Temperature of 0% POFA

The external temperature recorded was lower than the internal temperature as shown in Figure 4.1 which POFA not used as a cement replacement. The peak temperature for internal was 36.7°C at 615 minutes and for the external peak temperature was 32.1°C at 650 minutes. This result shows that the internal temperature was higher than the external temperature. In this specimen, the reaction that involved only hydration process.

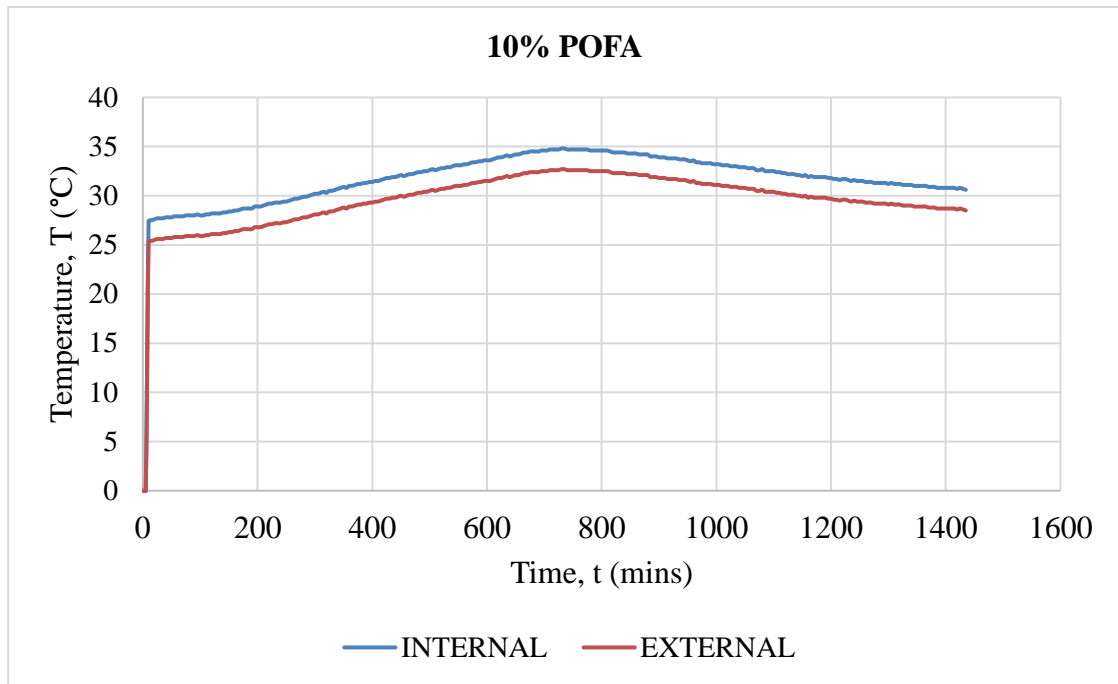


Figure 4.2 Internal and External Temperature of 10% POFA

Based on Figure 4.2 above, this figure also share the same pattern with Figure 4.2 which external temperature was lower than the internal temperature. But for this specimen which was steel fibre concrete that consist of 10% of POFA as a partial cement replacement, the peak temperature occur at the internal of concrete was 34.8°C at 730 minutes and for the external temperature, 32.7°C also at 730 minutes. From the results, it shows that the temperature was decreasing from Figure 4.1 because of the reaction involve in this specimen consist of hydration process and pozzolanic reaction.

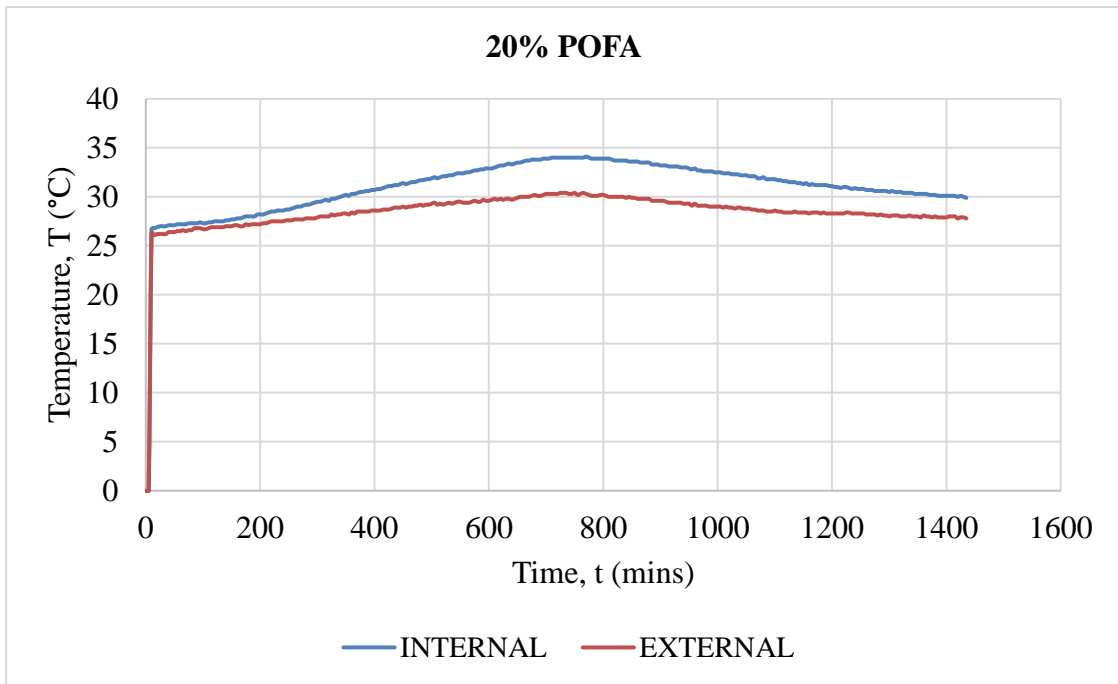


Figure 4.3 Internal and External Temperature of 20% POFA

Based on Figure 4.3 above, this figure shows external temperature was lower than the internal temperature of the specimen which consists of 20% of POFA as a partial cement replacement. The peak temperature for this specimen was 34.1°C at 770 minutes for internal temperature of the concrete. At the external peak temperature was 30.4°C at 725 minutes. It can be seen from the results that it had the same pattern as Figure 4.1 and Figure 4.2 which external temperature was lower than the internal temperature. The temperature of internal and external temperature was decreased to compare with Figure 4.1 and Figure 4.2 because of the reaction involved in this specimen consist of hydration process and pozzolanic reaction.

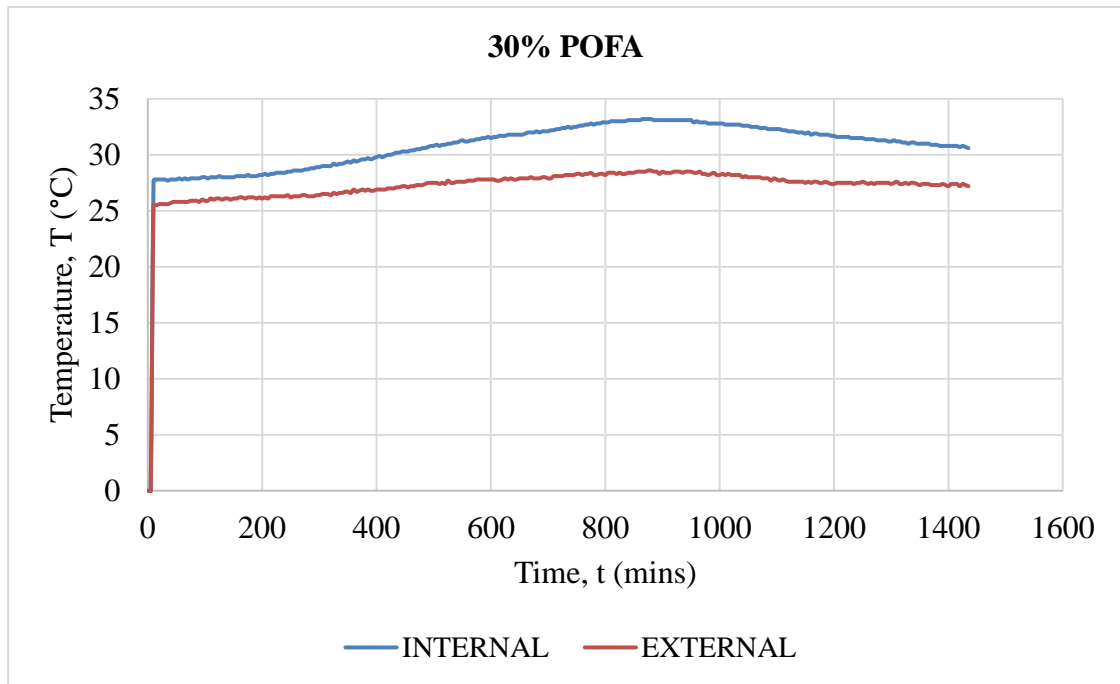


Figure 4.4 Internal and External Temperature of 30% POFA

Based on the Figure 4.4 above showed that internal temperature specimen of 30% POFA as a partial cement replacement were higher than the external temperature. The peak temperature were 33.2°C at 865 minutes for internal temperature and 28.6°C at 875 minutes for external temperature.

Based on Figure 4.1, Figure 4.2, Figure 4.3, and Figure 4.4 that all of this heat of hydration test result shared the same pattern which internal temperature was higher than the external temperature. When internal temperature too high than the external temperature, it can cause internal stress and lead to thermal crack that can gives negative impact towards the integrity of the concrete. But, Figure 4.4 which were steel fibre concrete that consist of 30% of POFA as a partial cement replacement had the lowest internal temperature of specimen and also lowest external temperature of the specimen. This lower temperature can reduced the risk of internal stress that occurred during hydration process and also reduced risk to the steel fibre concrete to crack.

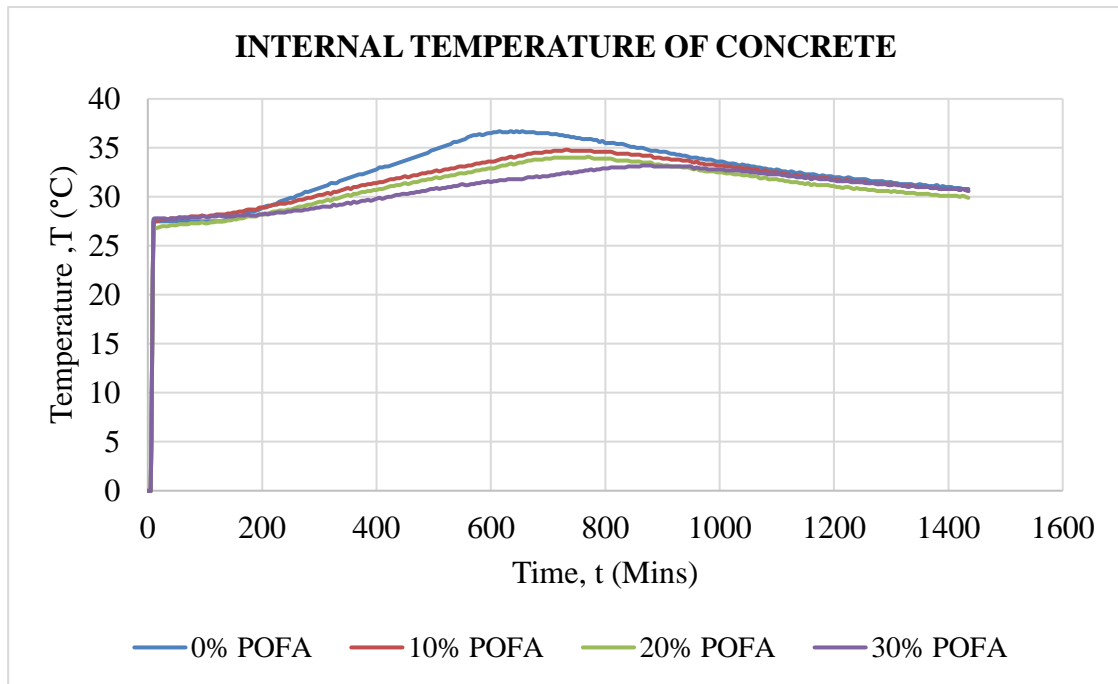


Figure 4.5 Internal Temperature of Steel Fibre Concrete

Table 4.1 The Results of Internal Temperature of Steel Fibre Concrete

<b>PROPERTIES</b>	<b>0% POFA</b>	<b>10% POFA</b>	<b>20% POFA</b>	<b>30% POFA</b>
Peak temperature (°c)	36.7	34.8	34.1	33.2
Time since mixing to peak temperature (minutes)	615	730	770	865

Based on Figure 4.5 and Table 4.1, as increased the percentage of POFA in steel fibre concrete as a partial cement replacement, the internal temperature of concrete was decreased. This result concludes that 30% of POFA as a partial cement replacement in steel fibre concrete had the lowest temperature during 24 hours of hydration process which the temperature was 33.2°C at 865 minutes compared to other specimens. This result show that 30% of POFA as a partial cement replacement was the best as it have the lowest internal temperature. This result occurred because of instead of 100% of OPC that involve in hydration process in control specimen, only 70% of them involve in hydration process and another 30% were involve in pozzolanic reaction which occur after hydration process finish.

The results shows for the heat of hydration test shows that, POFA as a partial cement replacement reduced the temperature of the steel fibre concrete and this results also supported by the previous research which POFA has good potentials in controlling heat of hydration of concrete. Particularly for mass concrete where thermal cracking due to excessive heat rise was great concern (Awal & Shehu, 2013).

Other than that, the results shows that as the increased of percentage of POFA as a cement replacement, the time to reach the peak temperature of the specimen were delayed from 0% POFA, 10% POFA, 20% POFA and 30% POFA. 0% POFA with 615 minutes for times to reach peak temperature, 10% POFA with 730 minutes, 20% POFA with 770 minutes and 30% with 865 minutes for times to reach peak temperature. The similar trend had been reported by Abdul Awal & Warid Hussin, 2011 that the use of POFA as a partial cement replacement, can reduced the total temperature rise as well as delayed the time at which the peak temperature occurred.

### 4.3 Drying Shrinkage Test

The drying shrinkage test was conducted at 7, 28 and 60 days and initial length of the mould was 28.5 cm. The curing method for this test was air curing method. Table 4.2 shows the difference length of the concrete for each percentage of POFA used and the difference length of the concrete was taken by using the Vernier Calliper. Figure 4.2 shows the graph of the drying shrinkage test based on Table 4.1 results.

Table 4.1 The Results of Drying Shrinkage Test

PERCENTAGE OF POFA (%)	CONCRETE AGE (DAY)			
	0	7	28	60
<b>0</b>	0	1.60	1.72	1.76
<b>10</b>	0	1.56	1.68	1.72
<b>20</b>	0	1.54	1.64	1.70
<b>30</b>	0	1.38	1.62	1.68

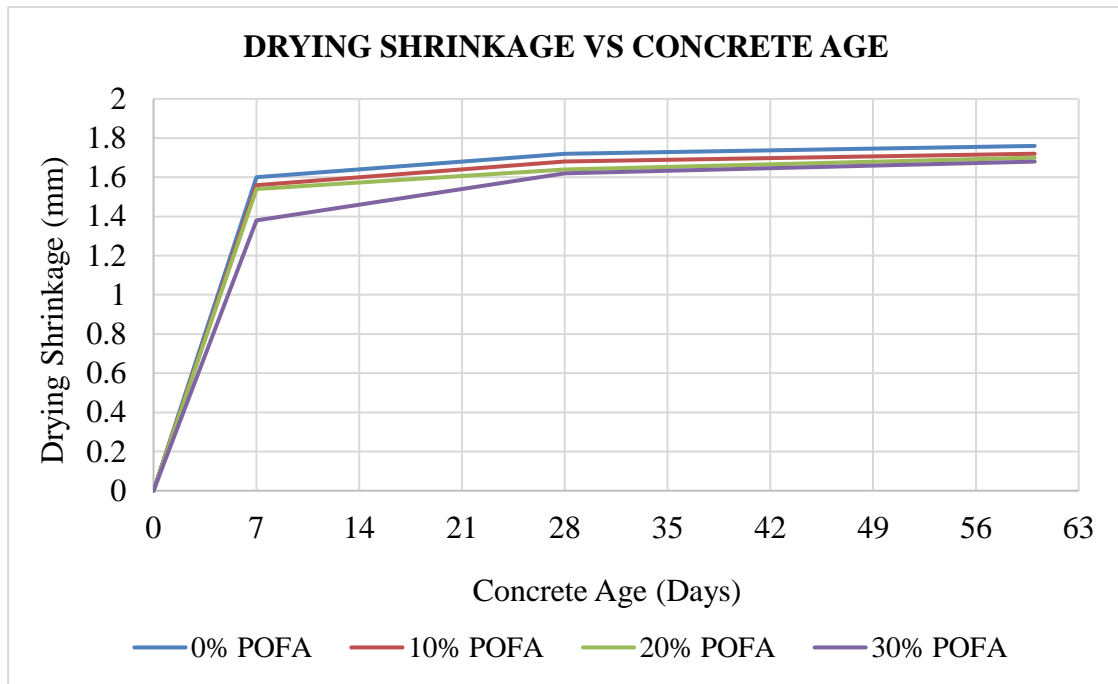


Figure 4.4 Drying Shrinkage Test Results

Based on Figure 4.6, the 30% of POFA as a partial cement replacement had the lowest loss of length compared with the other specimen. From the overall graph, as increased percentage of POFA as a partial cement replacement to the specimen, the drying shrinkage measurement of the specimen was also decreased. This result supported with the previous study which addition of POFA in the concrete have impact on reducing the dimensional change of specimens (Raut & Gomez, 2016). This result occurred because of the addition of POFA effect the pore refinement caused by pozzolanic reaction. Hydration process produce Calcium-Silicate-Hydrate Gel (CSH gel) and pozzolanic reaction produce more secondary CSH gel in the concrete. This secondary CSH gel act as a filling agent that closed the pores that available in the concrete and this results pore refinement. Reduction of pores size in the concrete reduced the water loss from the concrete and make the drying shrinkage reduced.

## **CHAPTER 5**

### **CONCLUSION AND RECOMMENDATION**

#### **5.1 Introduction**

In this research, the main objective to determine the effect of POFA as a partial cement replacement to steel fibre concrete towards heat of hydration and drying shrinkage. The data collected presented that the POFA can be partially replaced in steel fibre concrete. This chapter highlight on several conclusions drawn from the research that had been conducted and recommendation for better concrete mix and future study also discussed in this chapter.



## 5.2 Conclusion

As a conclusion, the results from heat of hydration test and drying shrinkage test shows that the POFA suitable to be a partial cement replacement to the steel fibre concrete towards heat generation and drying shrinkage. The main point of this study were outlined below:

1. Based on the results obtained from heat of hydration test, the internal temperature of steel fibre concrete containing POFA as a partial cement replacement can be reduced. Indirectly, the time of internal temperature to achieve peak temperature also delayed compared to the control specimen which not contained any POFA in the specimen. It because of heat released during hydration process when cement meet with water. When the cement reduced, hydration process reduced and heat release during hydration process reduced.
2. Steel fibre concrete containing POFA as a partial cement replacement also have better shrinkage than the control specimen. For the control specimen, there was the highest drying shrinkage length than steel fibre concrete that contained 30% of POFA as a partial cement replacement. The result was taken for 7, 28 and 60 days. It because of the addition of POFA effected the pore refinement caused by pozzolanic reaction. Reduction of size of pores controls the loss of water in the concrete and makes the drying shrinkage reduced. The amount of CSH gel was higher in the steel fibre concrete that contained POFA because it had two reaction that produce CSH gel which were hydration reaction and pozzolanic reaction. Pozzolanic reaction occured after hydration process finished as it consume  $\text{Ca(OH)}_2$  that produced from hydration process. CSH gel that produced from pozzolanic reaction act as a filling agent that filled the pores in the steel fibre concrete.

### 5.3 Recommendation

This research mainly focus on the performance of steel fibre concrete containing POFA as a partial cement replacement in terms of heat on steel fibre concrete during hydration process and drying shrinkage of the steel fibre concrete. There are some recommendation for future research suggested from the result obtained from research:

- i. The study on effect of POFA as a partial cement replacement towards durability of steel fibre concrete such as acid attack resistance should be carried out. It is because durability is one of the aspect that crucial. If the steel fibre concrete durability is low, the concrete cannot last long in certain environment. So, the further study about durability of to know that steel fibre concrete containing POFA as a partial cement replacement last longer in many environment.
- ii. The study on effect of POFA as a partial cement replacement with fine size of POFA towards mechanical properties of steel fibre concrete should be carried out. It is because different size of POFA can give different results towards the properties of steel fibre concrete.

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Appendix I

**APPENDIX I**

AVERAGE OF DRYING SHRINKAGE FOR EACH % OF POFA

<b>PERCENTAGE OF POFA (%)</b>	<b>CONCRETE AGE (DAY)</b>			
	<b>0</b>	<b>7</b>	<b>28</b>	<b>60</b>
0	0	1.60	1.72	1.76
10	0	1.56	1.68	1.72
20	0	1.54	1.64	1.70
30	0	1.38	1.62	1.68

## APPENDIX A1

HEAT TEST: Specimen 1: Control (0%)

Date Testing: 17/04/2018

Size Sample = 1' x 1' x 1'

Time: 4.40pm

Time (minutes)	Temperature ,T (°C)		Time (minutes)	Temperature ,T (°C)		Time (minutes)	Temperature ,T (°C)	
	Internal	External		Internal	External		Internal	External
0	0	0	225	29.3	27.9	450	33.7	30.3
5	0	0	230	29.5	28	455	33.8	30.4
10	27.5	0	235	29.6	28	460	33.9	30.4
15	27.5	24.9	240	29.7	28.1	465	34	30.5
20	27.5	26	245	29.8	28.2	470	34.1	30.7
25	27.5	26.1	250	29.9	28.3	475	34.2	30.6
30	27.5	26.1	255	29.9	28.3	480	34.3	30.8
35	27.5	26.2	260	30.1	28.4	485	34.4	30.7
40	27.5	26.2	265	30.2	28.4	490	34.5	30.7
45	27.5	26.2	270	30.3	28.5	495	34.7	30.8
50	27.5	26.3	275	30.5	28.6	500	34.8	30.8
55	27.5	26.4	280	30.5	28.6	505	34.9	31
60	27.5	26.4	285	30.6	28.7	510	35	30.9
65	27.5	26.4	290	30.7	28.7	515	35.1	31
70	27.5	26.5	295	30.8	28.7	520	35.2	31
75	27.5	26.5	300	30.9	28.8	525	35.3	30.9
80	27.5	26.6	305	31	28.9	530	35.4	31.1
85	27.5	26.6	310	31.1	28.9	535	35.6	31.1
90	27.5	26.7	315	31.2	29	540	35.6	31.3
95	27.5	26.8	320	31.4	29.1	545	35.7	31.2
100	27.5	26.7	325	31.4	29.2	550	35.8	31.3
105	27.5	26.7	330	31.4	29.1	555	35.9	31.3
110	27.5	26.8	335	31.6	29.2	560	36.1	31.4
115	27.5	26.8	340	31.7	29.2	565	36.2	31.5
120	27.5	26.9	345	31.8	29.3	570	36.3	31.6
125	27.5	26.8	350	31.9	29.4	575	36.3	31.6
130	27.5	26.9	355	32	29.4	580	36.4	31.6
135	27.6	27	360	32.1	29.4	585	36.3	31.7
140	27.7	27.1	365	32.2	29.6	590	36.4	31.6
145	27.8	27.1	370	32.3	29.7	595	36.5	31.7
150	27.8	27.1	375	32.3	29.7	600	36.5	31.9
155	28	27.2	380	32.4	29.6	605	36.6	31.8
160	28	27.4	385	32.5	29.8	610	36.6	31.9
165	28.1	27.2	390	32.6	29.9	615	36.7	32
170	28.2	27.3	395	32.7	29.8	620	36.6	32
175	28.4	27.5	400	32.8	29.9	625	36.6	31.9
180	28.5	27.5	405	33	30	630	36.6	31.9
185	28.5	27.6	410	33	30.1	635	36.7	31.9
190	28.6	27.5	415	33.1	30.1	640	36.6	31.9
195	28.7	27.7	420	33.1	30.1	645	36.7	32
200	28.9	27.7	425	33.2	30.1	650	36.6	32.1
205	29	27.8	430	33.3	30.2	655	36.7	31.8
210	29.1	27.8	435	33.4	30.2	660	36.6	32
215	29.1	27.9	440	33.5	30.3	665	36.6	31.8
220	29.2	27.9	445	33.6	30.3	670	36.6	31.9

## APPENDIX A1

HEAT TEST: Specimen 1: Control (0%)  
Size Sample = 1' x 1' x 1'

Date Testing: 17/04/2018  
Time: 4.40pm

Time (minutes)	Temperature ,T (°C)		Time (minutes)	Temperature ,T (°C)		Time (minutes)	Temperature ,T (°C)	
	Internal	External		Internal	External		Internal	External
675	36.6	31.9	900	34.6	30.3	1125	32.6	29.3
680	36.5	31.9	905	34.6	30.3	1130	32.5	29.4
685	36.5	31.7	910	34.5	30.3	1135	32.4	29.3
690	36.5	31.7	915	34.4	30.2	1140	32.5	29.3
695	36.5	31.7	920	34.4	30.2	1145	32.4	29.5
700	36.5	31.6	925	34.3	30.2	1150	32.3	29.4
705	36.4	31.7	930	34.3	30.1	1155	32.3	29.3
710	36.4	31.6	935	34.2	30.1	1160	32.3	29.3
715	36.4	31.6	940	34.2	30.1	1165	32.3	29.4
720	36.3	31.6	945	34.1	30	1170	32.2	29.3
725	36.3	31.6	950	34	30	1175	32.1	29.3
730	36.2	31.6	955	34	29.9	1180	32.2	29.3
735	36.2	31.5	960	34	29.8	1185	32.1	29.4
740	36.1	31.5	965	33.9	30	1190	32.1	29.3
745	36.1	31.5	970	33.9	29.9	1195	32.1	29.4
750	36	31.4	975	33.8	29.8	1200	32	29.4
755	36	31.4	980	33.8	29.9	1205	32	29.3
760	35.9	31.2	985	33.8	29.9	1210	32	29.3
765	35.9	31.2	990	33.7	29.7	1215	31.9	29.2
770	35.9	31.2	995	33.6	29.7	1220	31.9	29.2
775	35.9	31.1	1000	33.6	29.7	1225	32	29.3
780	35.8	31.1	1005	33.6	29.8	1230	31.9	29.3
785	35.8	31.1	1010	33.5	29.8	1235	31.8	29.3
790	35.6	31.2	1015	33.4	29.7	1240	31.8	29.2
795	35.7	30.9	1020	33.4	29.7	1245	31.8	29.2
800	35.5	31	1025	33.4	29.8	1250	31.8	29.1
805	35.5	30.9	1030	33.3	29.7	1255	31.8	29.2
810	35.5	30.8	1035	33.2	29.6	1260	31.7	29.2
815	35.5	30.9	1040	33.3	29.6	1265	31.6	29.2
820	35.4	30.7	1045	33.2	29.6	1270	31.6	29.1
825	35.4	30.8	1050	33.2	29.7	1275	31.6	29.1
830	35.4	30.8	1055	33.1	29.6	1280	31.6	29.1
835	35.3	30.8	1060	33.1	29.6	1285	31.6	29.1
840	35.2	30.7	1065	33	29.5	1290	31.5	29.1
845	35.1	30.6	1070	33	29.4	1295	31.5	29
850	35.1	30.6	1075	32.9	29.4	1300	31.5	29
855	35	30.6	1080	32.9	29.4	1305	31.4	29.2
860	35	30.5	1085	32.8	29.4	1310	31.4	29.2
865	35	30.5	1090	32.8	29.4	1315	31.3	29.2
870	34.9	30.5	1095	32.7	29.4	1320	31.3	29.1
875	34.8	30.4	1100	32.8	29.4	1325	31.3	29.1
880	34.7	30.4	1105	32.7	29.4	1330	31.3	29.1
885	34.7	30.4	1110	32.6	29.4	1335	31.3	29.1
890	34.7	30.4	1115	32.6	29.4	1340	31.2	29.1
895	34.6	30.3	1120	32.5	29.4	1345	31.2	29.1





## APPENDIX A2

HEAT TEST: Specimen 2: Control (10%)  
Size Sample = 1' x 1' x 1'

Date Testing: 19/04/2018  
Time: 4.40pm

Time (minutes)	Temperature ,T (°C)		Time (minutes)	Temperature ,T (°C)		Time (minutes)	Temperature ,T (°C)	
	Internal	External		Internal	External		Internal	External
0	0	0	225	29.2	27.1	450	32.1	30
5	0	0	230	29.3	27.2	455	32	29.9
10	27.4	25.3	235	29.3	27.2	460	32.1	30
15	27.5	25.4	240	29.3	27.2	465	32.2	30.1
20	27.6	25.5	245	29.4	27.3	470	32.2	30.1
25	27.7	25.6	250	29.4	27.3	475	32.3	30.2
30	27.7	25.6	255	29.5	27.4	480	32.4	30.3
35	27.7	25.6	260	29.6	27.5	485	32.4	30.3
40	27.8	25.7	265	29.7	27.6	490	32.5	30.4
45	27.8	25.7	270	29.7	27.6	495	32.5	30.4
50	27.8	25.7	275	29.8	27.7	500	32.6	30.5
55	27.9	25.8	280	29.9	27.8	505	32.7	30.6
60	27.9	25.8	285	29.9	27.8	510	32.6	30.5
65	27.9	25.8	290	30	27.9	515	32.7	30.6
70	27.9	25.8	295	30.1	28	520	32.8	30.7
75	28	25.9	300	30.2	28.1	525	32.8	30.7
80	28	25.9	305	30.2	28.1	530	32.9	30.8
85	28	25.9	310	30.3	28.2	535	32.9	30.8
90	28	25.9	315	30.4	28.3	540	33	30.9
95	28.1	26	320	30.3	28.2	545	33.1	31
100	28	25.9	325	30.5	28.4	550	33.1	31
105	28	25.9	330	30.5	28.4	555	33.1	31
110	28.1	26	335	30.6	28.5	560	33.2	31.1
115	28.1	26	340	30.7	28.6	565	33.2	31.1
120	28.2	26.1	345	30.8	28.7	570	33.3	31.2
125	28.2	26.1	350	30.9	28.8	575	33.4	31.3
130	28.2	26.1	355	30.8	28.7	580	33.4	31.3
135	28.2	26.1	360	31	28.9	585	33.5	31.4
140	28.3	26.2	365	31	28.9	590	33.5	31.4
145	28.3	26.2	370	31.1	29	595	33.6	31.5
150	28.4	26.3	375	31.2	29.1	600	33.6	31.5
155	28.4	26.3	380	31.2	29.1	605	33.6	31.5
160	28.5	26.4	385	31.3	29.2	610	33.7	31.6
165	28.5	26.4	390	31.3	29.2	615	33.8	31.7
170	28.6	26.5	395	31.4	29.3	620	33.9	31.8
175	28.7	26.6	400	31.4	29.3	625	33.9	31.8
180	28.7	26.6	405	31.5	29.4	630	34	31.9
185	28.7	26.6	410	31.5	29.4	635	34.1	32
190	28.7	26.6	415	31.6	29.5	640	34	31.9
195	28.9	26.8	420	31.7	29.6	645	34.1	32
200	28.9	26.8	425	31.8	29.7	650	34.2	32.1
205	28.9	26.8	430	31.8	29.7	655	34.2	32.1
210	29	26.9	435	31.9	29.8	660	34.3	32.2
215	29.1	27	440	31.9	29.8	665	34.4	32.3
220	29.2	27.1	445	32	29.9	670	34.4	32.3

## APPENDIX A2

HEAT TEST: Specimen 2: Control (10%)  
 Size Sample = 1' x 1' x 1'

Date Testing: 19/04/2018  
 Time: 4.40pm

Time (minutes)	Temperature ,T (°C)		Time (minutes)	Temperature ,T (°C)		Time (minutes)	Temperature ,T (°C)	
	Internal	External		Internal	External		Internal	External
675	34.5	32.4	900	33.9	31.8	1125	32.2	30.1
680	34.5	32.4	905	33.9	31.8	1130	32.2	30.1
685	34.5	32.4	910	33.9	31.8	1135	32.2	30.1
690	34.5	32.4	915	33.8	31.7	1140	32.1	30
695	34.6	32.5	920	33.8	31.7	1145	32.1	30
700	34.6	32.5	925	33.8	31.7	1150	32	29.9
705	34.6	32.5	930	33.8	31.7	1155	32.1	30
710	34.7	32.6	935	33.7	31.6	1160	31.9	29.8
715	34.7	32.6	940	33.7	31.6	1165	32	29.9
720	34.7	32.6	945	33.7	31.6	1170	31.9	29.8
725	34.7	32.6	950	33.6	31.5	1175	31.9	29.8
730	34.8	32.7	955	33.5	31.4	1180	31.9	29.8
735	34.8	32.7	960	33.6	31.5	1185	31.9	29.8
740	34.7	32.6	965	33.4	31.3	1190	31.9	29.8
745	34.7	32.6	970	33.4	31.3	1195	31.8	29.7
750	34.7	32.6	975	33.3	31.2	1200	31.8	29.7
755	34.7	32.6	980	33.3	31.2	1205	31.7	29.6
760	34.7	32.6	985	33.3	31.2	1210	31.7	29.6
765	34.7	32.6	990	33.3	31.2	1215	31.6	29.5
770	34.7	32.6	995	33.2	31.1	1220	31.7	29.6
775	34.7	32.6	1000	33.2	31.1	1225	31.7	29.6
780	34.6	32.5	1005	33.2	31.1	1230	31.6	29.5
785	34.6	32.5	1010	33.1	31	1235	31.5	29.4
790	34.6	32.5	1015	33.1	31	1240	31.6	29.5
795	34.6	32.5	1020	33.1	31	1245	31.5	29.4
800	34.6	32.5	1025	33	30.9	1250	31.5	29.4
805	34.6	32.5	1030	33	30.9	1255	31.5	29.4
810	34.6	32.5	1035	33	30.9	1260	31.4	29.3
815	34.5	32.4	1040	32.9	30.8	1265	31.4	29.3
820	34.4	32.3	1045	32.9	30.8	1270	31.4	29.3
825	34.4	32.3	1050	32.9	30.8	1275	31.3	29.2
830	34.4	32.3	1055	32.8	30.7	1280	31.3	29.2
835	34.4	32.3	1060	32.8	30.7	1285	31.3	29.2
840	34.4	32.3	1065	32.8	30.7	1290	31.3	29.2
845	34.3	32.2	1070	32.6	30.5	1295	31.3	29.2
850	34.3	32.2	1075	32.6	30.5	1300	31.2	29.1
855	34.3	32.2	1080	32.7	30.6	1305	31.3	29.2
860	34.3	32.2	1085	32.5	30.4	1310	31.2	29.1
865	34.2	32.1	1090	32.5	30.4	1315	31.2	29.1
870	34.2	32.1	1095	32.5	30.4	1320	31.2	29.1
875	34.2	32.1	1100	32.5	30.4	1325	31.1	29
880	34.2	32.1	1105	32.4	30.3	1330	31.1	29
885	34	31.9	1110	32.4	30.3	1335	31.1	29
890	34	31.9	1115	32.3	30.2	1340	31.1	29
895	34	31.9	1120	32.3	30.2	1345	31	28.9



### APPENDIX A3

HEAT TEST: Specimen 3: Control (20%)  
 Size Sample = 1' x 1' x 1'

Date Testing: 24/04/2018  
 Time: 2:00 pm

Time (minutes)	Temperature ,T (°C)		Time (minutes)	Temperature ,T (°C)		Time (minutes)	Temperature ,T (°C)	
	Internal	External		Internal	External		Internal	External
0	0	0	225	28.5	27.5	450	31.4	29
5	0	0	230	28.6	27.5	455	31.3	28.9
10	26.7	26.3	235	28.6	27.5	460	31.4	29
15	26.8	26.1	240	28.6	27.5	465	31.5	29
20	26.9	26.2	245	28.7	27.6	470	31.5	29.1
25	27	26.2	250	28.7	27.6	475	31.6	29.1
30	27	26.2	255	28.8	27.6	480	31.7	29.2
35	27	26.2	260	28.9	27.7	485	31.7	29.1
40	27.1	26.4	265	29	27.7	490	31.8	29.2
45	27.1	26.4	270	29	27.7	495	31.8	29.2
50	27.1	26.4	275	29.1	27.7	500	31.9	29.3
55	27.2	26.5	280	29.2	27.8	505	32	29.4
60	27.2	26.5	285	29.2	27.8	510	31.9	29.2
65	27.2	26.6	290	29.3	27.8	515	32	29.3
70	27.2	26.5	295	29.4	27.8	520	32.1	29.2
75	27.3	26.6	300	29.5	27.9	525	32.1	29.3
80	27.3	26.6	305	29.5	28	530	32.2	29.4
85	27.3	26.8	310	29.6	28	535	32.2	29.3
90	27.3	26.8	315	29.7	28	540	32.3	29.4
95	27.4	26.8	320	29.6	28	545	32.4	29.5
100	27.3	26.7	325	29.8	28.2	550	32.4	29.5
105	27.3	26.7	330	29.8	28.1	555	32.4	29.4
110	27.4	26.8	335	29.9	28.2	560	32.5	29.4
115	27.4	26.9	340	30	28.2	565	32.5	29.4
120	27.5	26.9	345	30.1	28.3	570	32.6	29.5
125	27.5	26.9	350	30.2	28.3	575	32.7	29.6
130	27.5	26.9	355	30.1	28.2	580	32.7	29.5
135	27.5	26.9	360	30.3	28.4	585	32.8	29.5
140	27.6	27	365	30.3	28.3	590	32.8	29.7
145	27.6	27	370	30.4	28.5	595	32.9	29.6
150	27.7	27	375	30.5	28.5	600	32.9	29.6
155	27.7	27.1	380	30.5	28.5	605	32.9	29.7
160	27.8	27.1	385	30.6	28.5	610	33	29.8
165	27.8	27	390	30.6	28.5	615	33.1	29.7
170	27.9	27	395	30.7	28.6	620	33.2	29.8
175	28	27.2	400	30.7	28.6	625	33.2	29.7
180	28	27.1	405	30.8	28.6	630	33.3	29.8
185	28	27.2	410	30.8	28.6	635	33.4	29.8
190	28	27.2	415	30.9	28.7	640	33.3	29.7
195	28.2	27.2	420	31	28.7	645	33.4	29.8
200	28.2	27.2	425	31.1	28.7	650	33.5	29.9
205	28.2	27.3	430	31.1	28.8	655	33.5	30
210	28.3	27.3	435	31.2	28.9	660	33.6	30
215	28.4	27.4	440	31.2	28.9	665	33.7	30
220	28.5	27.5	445	31.3	28.9	670	33.7	30

### APPENDIX A3

HEAT TEST: Specimen 3: Control (20%)  
 Size Sample = 1' x 1' x 1'

Date Testing: 24/04/2018  
 Time: 2:00 pm

Time (minutes)	Temperature ,T (°C)		Time (minutes)	Temperature ,T (°C)		Time (minutes)	Temperature ,T (°C)	
	Internal	External		Internal	External		Internal	External
675	33.8	30.1	900	33.2	29.6	1125	31.5	28.4
680	33.8	30.2	905	33.2	29.6	1130	31.5	28.4
685	33.8	30.1	910	33.2	29.5	1135	31.5	28.5
690	33.8	30.1	915	33.1	29.4	1140	31.4	28.4
695	33.9	30.2	920	33.1	29.4	1145	31.4	28.4
700	33.9	30.3	925	33.1	29.4	1150	31.3	28.4
705	33.9	30.3	930	33.1	29.4	1155	31.4	28.4
710	34	30.3	935	33	29.4	1160	31.2	28.3
715	34	30.3	940	33	29.3	1165	31.3	28.4
720	34	30.3	945	33	29.3	1170	31.2	28.4
725	34	30.4	950	32.9	29.3	1175	31.2	28.4
730	34	30.4	955	32.8	29.1	1180	31.2	28.3
735	34	30.4	960	32.9	29.3	1185	31.2	28.4
740	34	30.3	965	32.7	29.1	1190	31.2	28.3
745	34	30.3	970	32.7	29.1	1195	31.1	28.3
750	34	30.4	975	32.6	29.1	1200	31.1	28.3
755	34	30.2	980	32.6	29.1	1205	31	28.3
760	34	30.3	985	32.6	29	1210	31	28.3
765	34	30.4	990	32.6	29	1215	30.9	28.3
770	34.1	30.3	995	32.5	29	1220	31	28.3
775	34	30.2	1000	32.5	29	1225	31	28.4
780	33.9	30.1	1005	32.5	29	1230	30.9	28.4
785	33.9	30.1	1010	32.4	28.9	1235	30.8	28.3
790	33.9	30.1	1015	32.4	29	1240	30.9	28.3
795	33.9	30.2	1020	32.4	28.9	1245	30.8	28.3
800	33.9	30.2	1025	32.3	28.9	1250	30.8	28.3
805	33.9	30.1	1030	32.3	28.8	1255	30.8	28.3
810	33.9	30	1035	32.3	28.9	1260	30.7	28.2
815	33.8	30	1040	32.2	28.9	1265	30.7	28.2
820	33.7	30	1045	32.2	28.8	1270	30.7	28.2
825	33.7	30	1050	32.2	28.8	1275	30.6	28.2
830	33.7	30	1055	32.1	28.8	1280	30.6	28.1
835	33.7	30	1060	32.1	28.7	1285	30.6	28.2
840	33.7	29.9	1065	32.1	28.7	1290	30.6	28.1
845	33.6	30	1070	31.9	28.6	1295	30.6	28.1
850	33.6	29.9	1075	31.9	28.6	1300	30.5	28
855	33.6	29.9	1080	32	28.6	1305	30.6	28.1
860	33.6	29.8	1085	31.8	28.5	1310	30.5	28.1
865	33.5	29.8	1090	31.8	28.5	1315	30.5	28.1
870	33.5	29.8	1095	31.8	28.5	1320	30.5	28
875	33.5	29.8	1100	31.8	28.6	1325	30.4	28
880	33.5	29.8	1105	31.7	28.5	1330	30.4	28
885	33.3	29.6	1110	31.7	28.5	1335	30.4	28.1
890	33.3	29.6	1115	31.6	28.4	1340	30.4	28
895	33.3	29.6	1120	31.6	28.4	1345	30.3	28



## APPENDIX A4

HEAT TEST: Specimen 4: Control (30%)  
Size Sample = 1' x 1' x 1'

Date Testing: 26/04/2018  
Time: 2:00 pm

Time (minutes)	Temperature ,T (°C)		Time (minutes)	Temperature ,T (°C)		Time (minutes)	Temperature ,T (°C)	
	Internal	External		Internal	External		Internal	External
0	0	0	225	28.4	26.3	450	30.3	27.2
5	0	0	230	28.4	26.3	455	30.3	27.1
10	27.7	25.5	235	28.4	26.3	460	30.4	27.2
15	27.8	25.5	240	28.4	26.3	465	30.4	27.2
20	27.8	25.6	245	28.5	26.4	470	30.5	27.3
25	27.8	25.6	250	28.5	26.2	475	30.5	27.3
30	27.8	25.6	255	28.6	26.3	480	30.6	27.3
35	27.7	25.6	260	28.6	26.3	485	30.6	27.4
40	27.8	25.7	265	28.6	26.3	490	30.7	27.5
45	27.8	25.8	270	28.6	26.4	495	30.8	27.5
50	27.8	25.8	275	28.7	26.4	500	30.8	27.5
55	27.9	25.8	280	28.7	26.3	505	30.9	27.5
60	27.8	25.8	285	28.8	26.3	510	30.8	27.4
65	27.9	25.8	290	28.8	26.3	515	30.9	27.5
70	27.8	25.8	295	28.9	26.4	520	30.9	27.4
75	27.9	25.9	300	28.9	26.4	525	31	27.7
80	27.9	25.9	305	29	26.5	530	31	27.5
85	27.9	25.9	310	29	26.5	535	31.1	27.5
90	27.9	25.8	315	29	26.5	540	31.1	27.6
95	28	26	320	29	26.4	545	31.2	27.6
100	28	25.9	325	29.2	26.6	550	31.3	27.6
105	27.9	25.9	330	29.1	26.5	555	31.2	27.6
110	28	26.1	335	29.2	26.6	560	31.2	27.7
115	28	26	340	29.2	26.6	565	31.3	27.7
120	28	26.1	345	29.3	26.7	570	31.3	27.7
125	28.1	26.1	350	29.4	26.7	575	31.4	27.8
130	28	26	355	29.3	26.6	580	31.4	27.8
135	28	26.1	360	29.5	26.9	585	31.5	27.8
140	28	26.1	365	29.4	26.7	590	31.5	27.8
145	28	26	370	29.5	26.8	595	31.6	27.8
150	28	26.1	375	29.6	26.9	600	31.5	27.8
155	28.1	26.1	380	29.6	26.8	605	31.6	27.8
160	28.1	26.2	385	29.7	26.9	610	31.6	27.7
165	28.1	26.2	390	29.6	26.8	615	31.7	27.7
170	28.1	26.1	395	29.7	26.8	620	31.7	27.8
175	28.2	26.2	400	29.8	26.9	625	31.7	27.9
180	28.1	26.2	405	29.9	26.9	630	31.8	27.8
185	28.1	26.2	410	29.8	26.9	635	31.8	27.9
190	28.1	26.1	415	29.9	26.9	640	31.8	27.8
195	28.2	26.2	420	30	27	645	31.8	27.8
200	28.2	26.1	425	30	27	650	31.8	27.9
205	28.3	26.2	430	30.1	27	655	31.8	27.9
210	28.2	26.1	435	30.2	27.1	660	31.9	27.9
215	28.3	26.3	440	30.2	27.1	665	32	27.9
220	28.3	26.3	445	30.3	27.2	670	32	28



## APPENDIX A4

HEAT TEST: Specimen 4: Control (30%)  
Size Sample = 1' x 1' x 1'

Date Testing: 26/04/2018  
Time: 2:00 pm

Time (minutes)	Temperature ,T (°C)		Time (minutes)	Temperature ,T (°C)		Time (minutes)	Temperature ,T (°C)	
	Internal	External		Internal	External		Internal	External
675	32	27.9	900	33.1	28.5	1125	32.1	27.6
680	32.1	27.9	905	33.1	28.4	1130	32.1	27.6
685	32	27.9	910	33.1	28.4	1135	32	27.6
690	32.1	28	915	33.1	28.4	1140	32	27.6
695	32.1	28	920	33.1	28.5	1145	32	27.6
700	32.1	27.9	925	33.1	28.5	1150	31.9	27.5
705	32.2	27.9	930	33.1	28.5	1155	32	27.6
710	32.2	28.1	935	33.1	28.4	1160	31.8	27.5
715	32.3	28.1	940	33.1	28.5	1165	31.9	27.6
720	32.3	28.1	945	33.1	28.5	1170	31.9	27.6
725	32.4	28.1	950	33.1	28.5	1175	31.8	27.5
730	32.4	28.2	955	32.9	28.4	1180	31.8	27.5
735	32.5	28.2	960	33	28.4	1185	31.8	27.6
740	32.4	28.2	965	32.9	28.5	1190	31.8	27.5
745	32.5	28.2	970	32.9	28.3	1195	31.7	27.4
750	32.5	28.3	975	32.9	28.3	1200	31.7	27.4
755	32.6	28.3	980	32.8	28.2	1205	31.6	27.5
760	32.6	28.2	985	32.8	28.3	1210	31.6	27.5
765	32.7	28.3	990	32.8	28.4	1215	31.6	27.5
770	32.7	28.3	995	32.8	28.2	1220	31.6	27.5
775	32.8	28.4	1000	32.8	28.2	1225	31.6	27.5
780	32.7	28.2	1005	32.8	28.3	1230	31.5	27.5
785	32.8	28.2	1010	32.7	28.2	1235	31.5	27.4
790	32.8	28.3	1015	32.7	28.3	1240	31.5	27.5
795	32.9	28.3	1020	32.7	28.2	1245	31.5	27.5
800	32.9	28.2	1025	32.7	28.2	1250	31.5	27.6
805	32.9	28.4	1030	32.7	28.2	1255	31.4	27.5
810	33	28.4	1035	32.7	28.2	1260	31.4	27.4
815	33	28.4	1040	32.6	28	1265	31.4	27.5
820	33	28.3	1045	32.6	28.1	1270	31.4	27.4
825	33	28.4	1050	32.6	28	1275	31.3	27.5
830	33	28.3	1055	32.5	28	1280	31.3	27.5
835	33	28.3	1060	32.5	28	1285	31.3	27.5
840	33.1	28.4	1065	32.5	28	1290	31.3	27.5
845	33.1	28.4	1070	32.4	28	1295	31.2	27.5
850	33.1	28.4	1075	32.4	27.9	1300	31.2	27.4
855	33.1	28.5	1080	32.4	27.9	1305	31.3	27.5
860	33.1	28.5	1085	32.3	27.9	1310	31.2	27.6
865	33.2	28.5	1090	32.3	27.7	1315	31.2	27.4
870	33.2	28.5	1095	32.3	27.9	1320	31.1	27.4
875	33.2	28.6	1100	32.3	27.8	1325	31.1	27.5
880	33.2	28.6	1105	32.3	27.7	1330	31	27.4
885	33.1	28.5	1110	32.2	27.8	1335	31.1	27.5
890	33.1	28.5	1115	32.2	27.7	1340	31.1	27.5
895	33.1	28.3	1120	32.1	27.6	1345	31	27.4

