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STUDY ON THE PROPERTIES OF PALM OIL FIBER

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Praise be to Allah, the Lord of the Worlds

Who says (interpretation of the meaning):

“Give thanks to Me and your parents. Unto Me is the final destination”

[Quraan, Luqman 31: 14]

All glory and honor to Him

ABSTRACT

This study investigated the strength and durability of Palm Oil Fiber Reinforced Concrete (POFRC). Two POFRC mixes with two different content and length of palm oil fiber (POF) were used. Specimen P_{F1} contained 0.50% POF of 3 cm length while specimen P_{F2} contained 0.25% POF of 5 cm length. The strength and durability of both specimens were compared with a control mix, P_0.

The concrete specimens were cured in water for 28 days, before immersing them in 1% hydrochloric acid solution for 1800 hours. The durability of POFRC against acid attack was evaluated by the loss of weight and compressive strength. In this paper, the attack of chloride ions on specimens immersed in 3% sodium chloride solution using the colorimetric method was studied. After 7, 28 and 90 days curing, the progressive deterioration was evaluated through spraying of 0.2N silver nitrate solution on the split cylinders and by visual inspection. Microanalyses were conducted upon test termination to elucidate the damage mechanisms using FESEM and EDX analysis. In most cases, $P_{F1,}$ which had the highest fiber content (0.5%) did not give good results while P_{F2} showed good performance against deteriorations compared to the control mix, P_0.

ABSTRAK

Kajian yang dijalankan adalah menyelidik tentang kekuatan dan ketahanlasakan konkrit diperkuat serat kelapa sawit (POFRC). 2 jenis spesimen konkrit yang berbeza kandungan dan panjang serat digunakan. Spesimen P_{F1} mengandungi 0.50% serat kelapa sawit dengan 3 cm panjang, manakala spesimen P_{F2} mengandungi 0.25% serat kelapa sawit dengan 5 cm panjang. Kekuatan dan ketahanlasakan ini dibandingkan dengan konkrit kawalan, P_0.

Kesemua spesimen konkrit direndamkan di dalam air selama 28 hari sebelum rendamanan lain dilakukan. Spesimen untuk ujian ketahanlasakan terhadap asid direndamkan di dalam 1% asid hidroklorik selama 1800 jam. Ketahanlasakan konkrit POFRC terhadap serangan asid dinilai berdasarkan kehilangan berat dan kehilangan kekuatan mampatan pada 1800 jam. Untuk ujian ketahanlasakan terhadap ion klorida, spesimen direndam ke dalam larutan 3% natrium klorida selama 7, 28 dan 90 hari. Dengan menggunakan kaedah analisis kolorimetri, spesimen silinder yang terdedah disembur dengan larutan 0.2 N nitrat perak dan pemeriksaan tampakan dilakukan. Analisis mikro dijalankan terhadap spesimen selepas tamat ujian untuk menerangkan mekanisme kerosakan dengan menggunakan FESEM dan analisis EDX. Secara seluruhannya, P_{F1} yang mempunyai kandungan serat yang tinggi (0.50%) tidak memberikan keputusan yang baik manakala, specimen P_{F2} pula menunjukkan kemajuan yang baik terhadap serangan asid dan klorida berbanding konkrit kawalan P_0.

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LIST OF ABBREVIATIONS

ACI	-	American Concrete Institute
$AgNO_3$	-	Silver Nitrate
ASTM	-	American Society of Testing of Materials
BS	-	British Standards
CPO	-	Crude palm oil
C-S-H	-	Calcium Silicate Hydrate
CH	-	Calcium Hydrate
FESEM	-	Field Emersion Scanning Electron Microscopic
FRC	-	Fiber Reinforced Concrete
HCl	-	Hydrochloric Acid
MOPB	-	Malaysian Oil Palm Board
NaCl	-	Sodium Chloride
OPC	-	Ordinary Portland Cement
POFA	-	Palm Oil Fuel Ash
POF	-	Palm Oil Fiber
PORRC	-	Palm Oil Fiber Reinforced Concrete
XRD	-	X-ray diffraction

CHAPTER I

INTRODUCTION

1.1 General

Concrete is the most common material that has been used in the construction sector of the world. Its usage is around 10 billion tons per year, which is equivalent to 1 ton per every living person. Even though this material is being used as a modern material, concrete has been in use for hundreds of years. Concrete is a tremendously popular structural material due to its low cost and easy of fabrication of construction.

The word concrete comes from the Latin *concretus*, which means mixed together or compounded. Concrete consist of sand or stone, known as aggregate, combined with cement paste to bind it. Aggregate can be found in various sizes and be categorized as fine (sand) and coarse (crushed stone or gravel). The aggregate in concrete can be in greater proportion of concrete which is bulky and cheaper than the cement.

As the constituents of concrete come from stone, people have always thought that concrete has the same quality and will last forever. However, concrete must be

thought of as a distinct material to stone. It has its own characteristics in terms of durability, weathering and repair.

Concrete is a relatively durable and tough building material, but it can be severely weakened by poor manufacture or a very aggressive environment. A number of historic concrete structures exhibit problems that are related to their date of origin. These problems can be solved by application of polymer in concrete construction. (Lee, 2007)

1.2 Background of Fiber Reinforced Concrete

Fiber reinforced concrete (FRC) is concrete mixed with discontinuous discrete fibers. The short discrete fibers contain in it are uniformly distributed and oriented. Fibers include steel fibers, glass fibers, synthetic fibers and natural fibers. Within these different fibers that character of fiber reinforced concrete changes with varying concretes, fiber materials, geometries, distribution, orientation and densities (Somayaji, 2001).

Fibers have an excellent potential to improve the mechanical properties of rapid-setting materials, and could be used effectively to improve the performance of repairs. The investigations indicate that the behavior of fiber-reinforced rapid-setting materials is similar to that of normal Portland cement fiber-reinforced concrete, (Balaguru, 1992). Fiber reinforce concrete improves flexural toughness, an indicator of ductility, crack resistance and also increased splitting tensile strength (Rabalais, 1992).

Fibers are usually used in concrete to control plastic shrinkage cracking and drying shrinkage cracking. They also lower the permeability of concrete and thus reduce bleeding of water. Some types of fibers produce greater impact, abrasion and shatter resistance in concrete.

The tensile strength, on the other hand, increased as the fiber volume fraction increased. It is also noted that the casting method, the fiber placing process, and the specimen configuration significantly affect the strength and tensile properties of these fiber reinforced concrete composite (Wecharatana and Lin, 1992)

1.3 Problem Statement

In Malaysia, there are about 3.1 million hectares of oil palm trees that produce over 9 million tonnes of crude palm oil (CPO) annually. The oil production represents only 10% and the remaining 90% consists of lignocellulosic material of the total biomass produced by the industry, (Amar et al., 2005). Oil palm empty fruit bunch (OPEFB) fiber is one of the readily available, non woody natural fiber in Malaysia. OPEFB fiber is a byproduct from the oil palm industry.

Therefore, it is really useful to find the application for these materials, which will surely lessen environmental problems related to the disposal of oil palm wastes and produce materials that could offer a favorable balance of quality, performance and cost.

Since now, there are not many researchers that have done study on the oil palm fiber concrete for the strength properties including tensile and flexural. Also, there is insufficient data on the durability performance aspect of Palm Oil Fiber Reinforced Concrete (POFRC). Thus, it is necessary to check this properties of the material before the material can be broadly used as a building material.

1.4 Objectives

The objectives of this study are:-

i. To find the compressive strength of POFRC Concrete;
ii. To find the durability performance aspect of POFRC;
iii. To study the damage mechanism upon test termination using microanalysis FESEM and EDX analysis.

1.5 Scope of Research

It is noticeable that various studies in Malaysia focus on the oil palm fiber as well as all over the world. The utilization of Palm Oil Fiber (POF) as addictive material of ordinary Portland cement in research programs have been started at the Faculty of Civil Engineering of the University to study the various strength properties of concrete (Megandran, 2007; Huzaifa 2008)

Two POFRC mixes with two different content and length of palm oil fiber (POF) were used. Specimen P_{F1} contained 0.50% POF of 3 cm length while specimen P_{F2} contained 0.25% POF of 5 cm length. The strength and durability of both specimens were compared with a control mix, P_0. This research is mainly about the strength and durability of palm oil fiber in concrete. The durability aspects are including penetration of chloride and resistance of acid attack.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Many studies on the effect of fiber's application in concrete material have been reported (Torrijos *et al*., 2007; Aruntas *et al.*, 2008; Haddad *et al*, 2007; Mohammadi *et al*., 2006; Brandt, 2008; Ibell, 2008; Alnahhal and Aref 2007; Hsie *et al*., 2008; Megandran, 2007; Huzaifa, 2008) and conducted recently. However, there is insufficient data on strength and durability aspects of palm oil fiber as addictive to concrete. Therefore, further investigation should be conducted concerning the application of this material. Either properties of the palm oil fiber or the effect of palm oil fibers after incorporating with concrete is required.

2.2 Palm Oil Fiber

Palm oil fiber is a lignocellulosic material which mainly consists of cellulose, lignin and hemicelluloses, has achieved demand thrust in the recent years. As such, lignocelluloses fibers are available plentiful, they have been fully exploited even with insufficient technology development.

Oil palm fiber is a non-hazardous biodegradable material extracted from oil palm's empty fruit bunch (OPEFB) through decortation process. The fibers are clean, non-carcinogenic, free from pesticides and soft parenchyma cells. Figure 2.1 show the fibers that have been used for this study.

There has been a growing interest in utilizing natural fibers as reinforcement in construction industry for making low cost construction material. Several studies on application of the fiber in concrete structure have been conducted by researchers but not many on fiber characteristic. The study on characteristic of this biomass would influence in generating the utilization and application in all field.

Figure 2.1: Palm Oil Fiber

2.3 OPEFB Fiber Characteristic

2.3.1 Fiber Morphology

The structure and properties of Palm Oil Fiber have been investigated by several researchers (Abdul Khalil *et al*.,2006; Law *et al*., 2007). The understanding of morphology and characteristic will not only help open up a new avenue for this fiber, but also emphasize the importance of this agriculture material.

Analysis of palm oil fiber was carried out by using scanning electron microscope (FESEM, Material Lab, Faculty of Mechanical, UTM). Figures 2.2 to 2.7 show the image of scanning electron micrographs of palm oil fiber respectively. Figure 2.2 shows the cross section the fiber. As can be seen in Figure 2.2, the cross section of POF is oval and fairly uniform in dimension. It contains various sizes of vascular bundle. The vascular bundles are widely imbedded in the thin-walled parenchymatous ground tissue. Each bundle is made up of a fibrous sheath, vessels, fibers, phloem, and parenchymatous tissues. (Abdul Khalil *et al*., 2006).

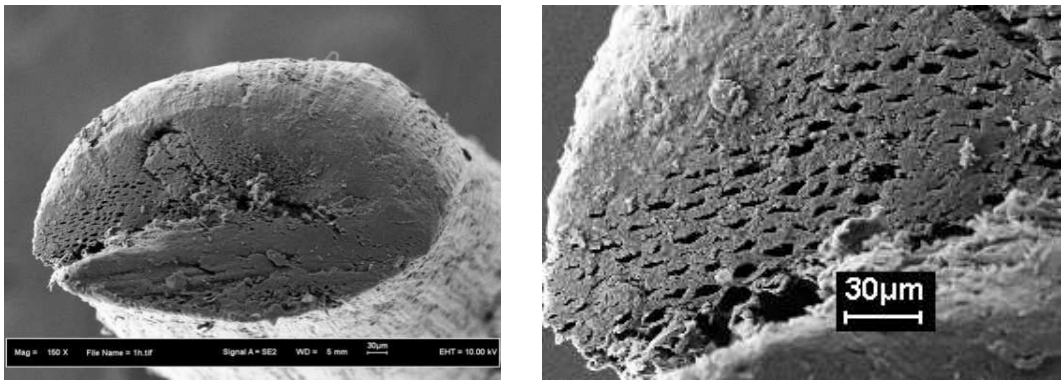


Figure 2.2: Cross-section view of a fibrous strand

Figure 2.4 and 2.5 shows the interior view of fibrous strand. The electron microscopic observations were mainly to the walls of the fibers within vascular bundle.

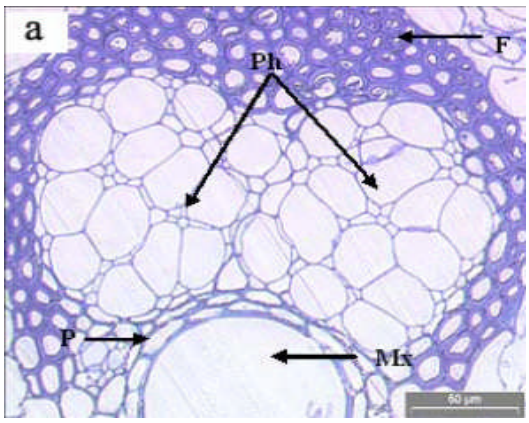


Figure 2.3: Tranverse section of oil palm frond fiber at high magnification (200x), (Abdul Khalil *et al.*, 2006).

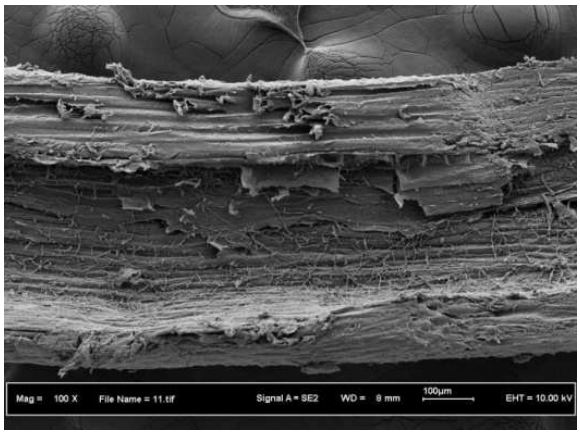


Figure 2.4: Interior longitudinal view at magnification 100x

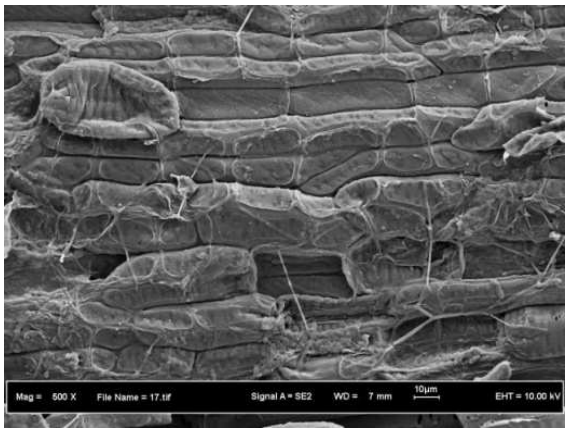


Figure 2.5: Interior longitudinal view at magnification 500x

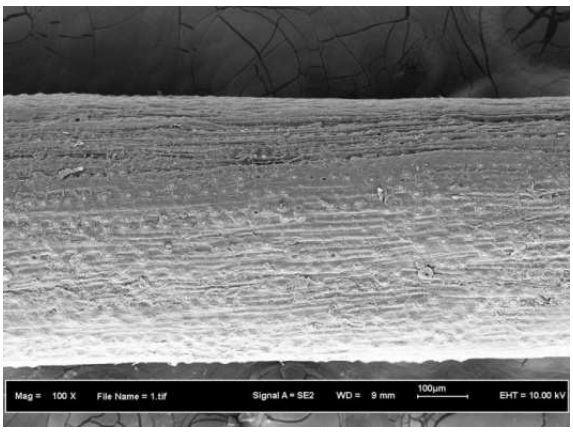


Figure 2.6: Longitudinal surface view at magnification 500x

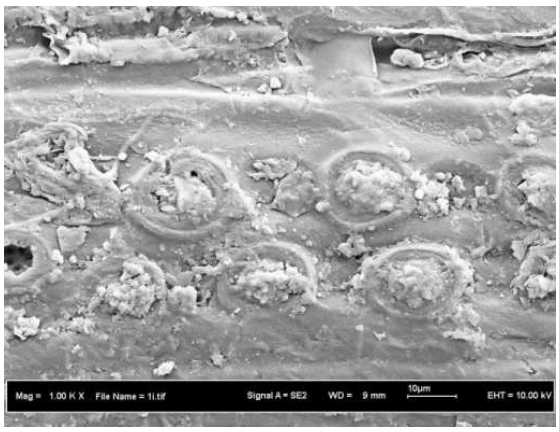
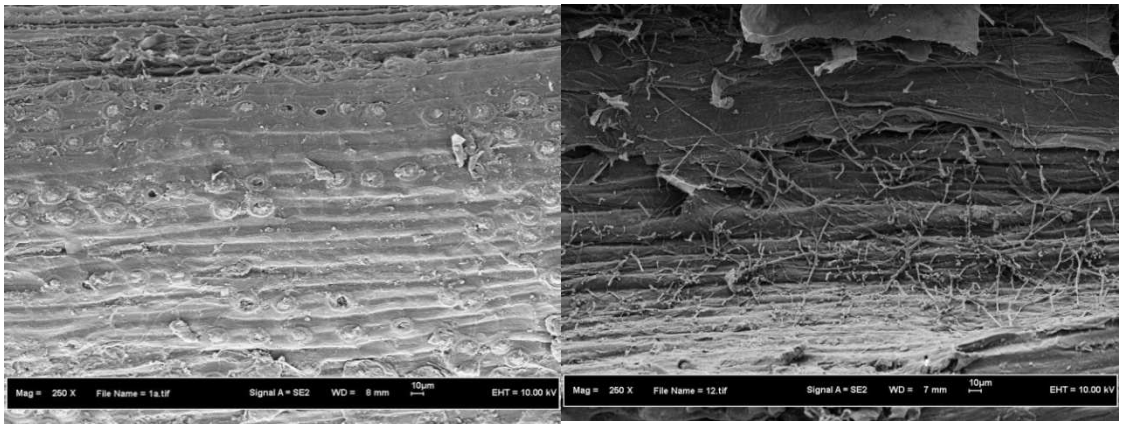


Figure 2.7: Longitudinal surface view at magnification 100x

Figure 2.6 shows the longitudinal surface view at magnification 500x and Figure 2.7 shows closer and clearer view of surface POF. As can be seen, Silica-bodies are found in great numbers on POF strand. They seem to attach themselves to circular crater which spread relatively uniform over the strand's surface, (Law *et al*., 2007). The Silica-bodies looked like rounded-shape, are measured about 10-20 µm in diameter.



(a) (b)

Figure 2.8: Surface view of POF at magnification 250x with silica-bodies (a) while the interior is lack visible silica-bodies (b).

Figure 2.8 (b) shows the silica bodies were disappear from the silica crater making it perforated. Law *et al*. (2007) reported that the removal of silica-bodies would enhance chemical penetration in pulping and the formation. These researchers also reported the formation of silica-bodies originated from the interior of fibrous strand through siliceous pathway (Figure 2.8, a)

2.3.2 Physical properties of Palm Oil Fiber

Physical properties of palm oil fiber elements are shown in Table 2.1, in comparison with other references.

Table 2.1: Physical Properties of Palm Oil Fiber

Properties	**Result From**	**References**	
	MPOB/UKM	Amar et al. 2005	Law et al. 2007
Average fiber length, mm	170.38		
Average fiber width, μm	291.44		
Average lumen width, μm	11.33	7.90	12.34
Average wall thickness, μm	4.90	2.30	3.38
Moisture content, %	10.57		
Ashes content, %	2.4		
Water absorption, %	15.97 *result from Amrec, SIRIM		

2.3.3 Chemical composition

Table 2.2 shows the percentages of various chemical components present in POF. Samples of Palm Oil Fiber were sent to MPOB/UKM to determine the chemical composition. Results in the table shown as in percentage unit were determine by Lignocellulosic analysis. The results obtained were almost similar compared with Law et al. (2007) for a few certain properties.

According to Joseph (1999), the characteristics of the fiber individual depend on the constituents, the fibrillar structure and lamellae matrix. The fiber is composed of numerous elongated fusiform fiber cells that taper toward each end. The fiber cells are linked together by means of middle lamellae, which consists hemicellulose, lignin and pectin. Dinwoodie (1981) summarizes the polymeric state, molecular

derivatives and function of cellulose, hemicellulose, lignin and extractives as shown in Table 2.3.

Table 2.2: Chemical composition

Constituent	**MPOB/UKM**	**Reference**		
		Law et al. 2007	*Khoo & Lee 1991*	*Law & Jiang 2001*
Extractives	**3.323**	3.7 ± 0.3	0.9	2.8
Acid-insoluble lignin	**20.917**	18.8 ± 0.3	17.2	17.6
Ash-free acid-insoluble	-	17.8 ± 0.2	-	-
Ash	-	1.3 ±0.2	0.7	3.8
Hot-water soluble	-	7.5 ± 0.8	2.8	9.3
1% NaOH soluble	-	14.5 ± 2.7	17.2	29.9
Holocellulose	**62.785**	82.4 ± 1.4	70.0	86.3
Cellulose	**39.405**	62.9 ± 2.0	42.7	-
Hemicellulose	**23.380**	28.0	32.5 (Leh, 2002)	-

Table 2.3: Cellulose, hemicellulose, lignin and extractives,polymeric state, molecular derivatives and function (Dinwoodie, 1981)

Content	**Polymeric state**	**Molecular Derivatives**	**Function**
Cellulose	Crystalline highly oriented large molecule	Glucose	Fiber
Hemicelluloses	Semi-crystalline smaller molecule	Galactose, Mannose, Xilose	Matrix
Lignin	Amorphous large 3-D molecule	Phenyl propane	Matrix
Extractives	Some polymeric; others nonpolymeric e.g. Terpentes	Polyphenols	Extraneous

Lignin

Complex chemical such as Lignin (lignen) is a compound usually derived from wood and is an integral part of the secondary cell walls of plants. Lignin fills the spaces in the cell wall between cellulose, hemicellulose, and pectin components, especially in tracheids, sclereids and xylem. The lignin content of fibers influences its structure, properties and morphology. Joseph (1999) reported that each cell of hard plant fibers is bonded together by lignin, acting as a cementing material. This researcher also reported that the lignin content in fibers influences its structural, morphology and properties.

Holocellulose

Holocellulose is a mixture of cellulose and hemicellulose in fiber. Holocellulose is also the fibrous residue that remains after the extractives, the lignin, and the ash-forming elements, have been removed.

Cellulose

The structural component of the primary cell wall of green plants, many forms of algae and the oomycetes is Cellulose. Cellulose is the predominant constituent of cotton, linen, and other plant fibers made for paper and cardboard and of textiles.

Hemicellulose

A hemicellulose can be any structural component present in almost all plant cell walls along with cellulose. As cellulose is crystalline, tough, and resistant to hydrolysis, hemicellulose has a random, amorphous structure with only a little strength.

2.3.3.1 Elements in Palm Oil Fiber

Figure 2.9: Area of analysis

Table 2.4: Elements of Palm Oil Fiber

Constituent	Result from UTM (% atomic)	**Reference**	
		Law *et al*. 2007	Singh *et al*. 1999
Oxygen	84.28		
Silica	0.64	1.8 (% atomic)	-
Copper	1.22	0.8 ±0.7 g/g	23 mg/L
Calcium	0.92	2.8 ± 0.1 g/g	0.25% (CaO)
Manganese		7.4 ± 0.4 g/g	48 mg/L
Iron		10.0 g/g*	473 mg/L
Sodium	1.62	11.0 ± 0.4 g/g	-
Potassium	11.32		

The elements in Palm Oil Fiber shown in Table 2.4 are the principal elements of a POF are determined by EDX analysis from FESEM, Mechanical Faculty, UTM. It shows the relative atomic percentage of each element and Figure 2.9 shows the area of analyzed specimen using FESEM. The results were compared with the findings by other researchers. The main components in Palm Oil Fiber are oxygen, silica, copper, calcium, sodium, potassium, manganese and iron.

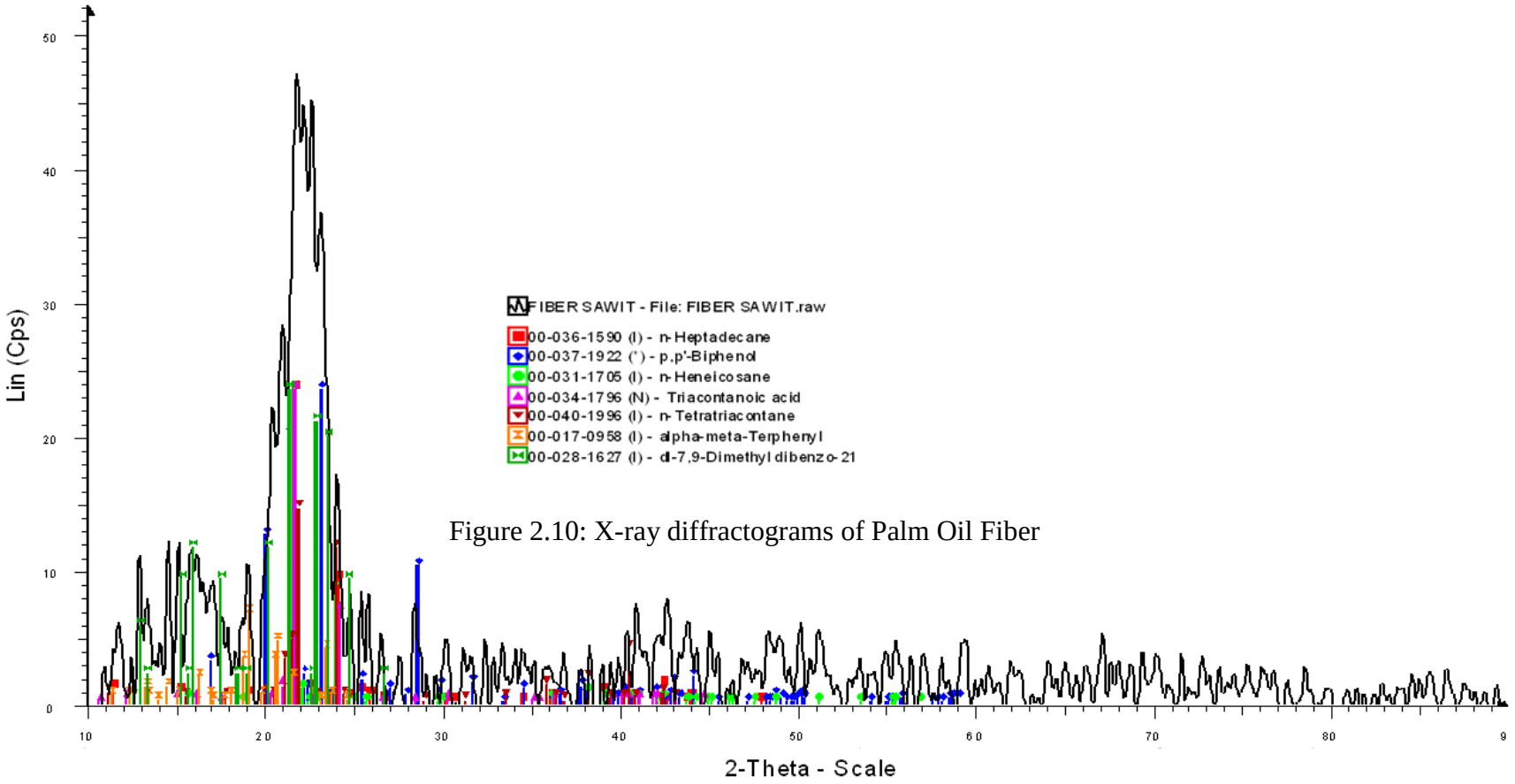


Figure 2.10: X-ray diffractograms of Palm Oil Fiber

Figure 2.10 shows the result of XRD analysis of a palm oil fiber. The test was conducted at Amrec, SIRIM. From the test result, the components in palm oil fiber were revealed. The peak is dominant by the major components consist of Heptadecane, Biphenol, Heneicosane, Triacontanoic acid, Tetratriacontane, and Dimethyldibenzo-21.

2.4 Mechanical Properties of Palm Oil Fiber

Properties of POF determined by this study are very useful in the evaluation of new fiber at the research and development level. Because of their nature, fiber does not have a unique strength rather a distribution of strength. This test result obtained from the strength of a single fiber. Tensile strength is calculated from the ratio of peak force and the cross-sectional area of a plane perpendicular to the fiber axis, at the fracture location or in the vicinity of the fracture location, while Young's Modulus is determined from the liner region of the tensile strain curve.

The percentage of amorphous and crystalline components of natural fiber is a determining factor in the mechanical behavior of natural fiber and also due to the organic compound. (Sreekala *et al*., 2000). The tensile stress relaxation behavior of individual oil palm empty fruit bunch fiber was investigated.

Table 2.5 shows the result of mechanical properties of palm oil fiber. The mechanical properties such as tensile strength, elongation and the modulus of elasticity of fiber were determined in accordance with standards ASTM C1557, in an Tinius Olsen machine at a cross-head speed of $8x10^{-6}$ m/min. The tensile test lengths were 100 mm. Five fibers were tested for each parameter setting. The graph of the mechanical properties of fibers can be seen in Figure 2.11.

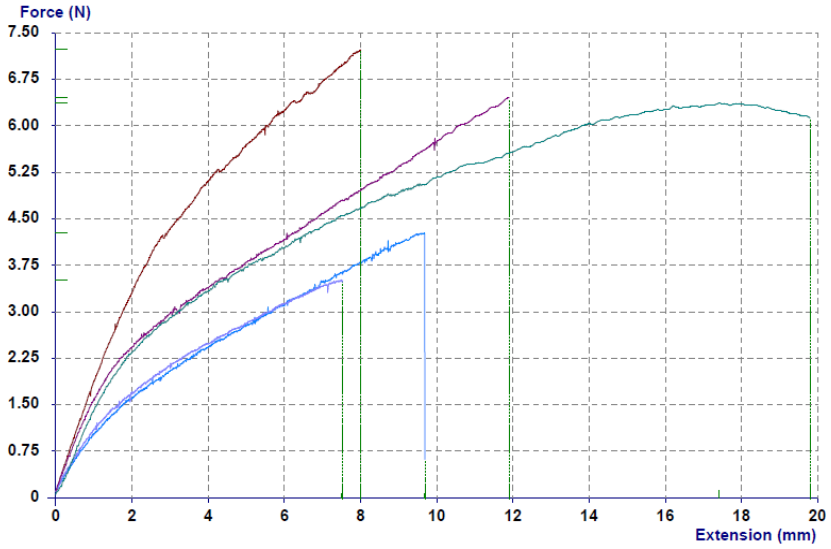


Figure 2.11: Ultimate tensile force vs. extension graph (Result from SIRIM)

Table 2.5**:** Mechanical Properties of POF

Technical Attributes	**Reference**	**Result from**	
	Megandran,2007	FKKKSA,UTM	Amrec, SIRIM
Tensile Strength (MPa)	21.2	113.43	83.1
Elongation at break (%)	0.04	0.10	0.11
Compression of Strength (MPa)	36.4		
Direct Screw Withdrawal (N)	1420		
Nail Withdrawal (N)	310		
Bending Stress (MPa)	37.6		

2.5 Fiber Reinforced Concrete

Many researchers have conducted investigations to study the different characteristics of fiber reinforced concrete in the past. The addition of fibers to concrete considerably improves its structural characteristics such as static flexural strength, impact strength, tensile strength, ductility and flexural toughness (Mohammadi, 2006).

Fiber-reinforced concrete offers a solution to this problem of cracking by making the concrete tougher and more durable, by incorporating three-dimensional reinforcement within the concrete, (Somayaji, 2001). Fibers are usually used in concrete to control plastic shrinkage cracking and drying shrinkage cracking. They also lower the permeability of concrete and thus reduce bleeding of water. Some types of fibers produce greater impact, abrasion and shatter resistance in concrete. Generally, fibers do not increase the flexural strength of concrete, so it cannot replace moment resisting or structural steel reinforcement. Some fibers reduce the strength of concrete.

Workability of palm oil fiber modified concrete decreases with the increase of fiber content in the concrete mix. This is due to the water absorption characteristic of palm oil fiber which absorbed water during mixing process and gave low slump during the slump test, (Megandran, 2007). Workability has been decreased significantly with the addition of fibers.

The main properties influencing toughness and maximum loading of fibre reinforced concrete are;

i. Type of fibers used.
ii. Volume percent of fiber.
iii. Aspect ratio (the length of a fiber divided by its diameter).
iv. Orientation of the fibers in the matrix.

2.5.1 Compressive Strength

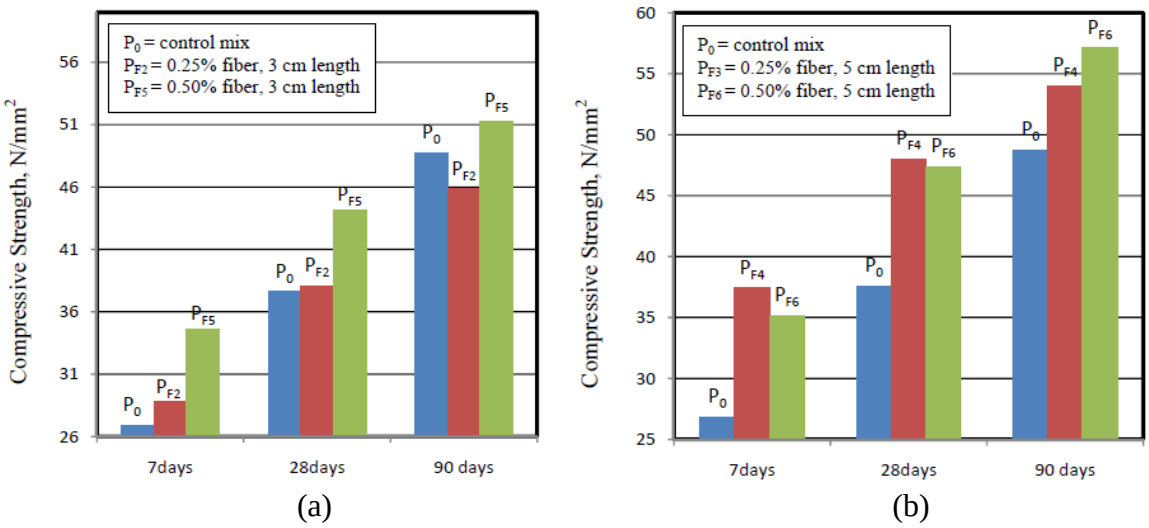


Figure 2.12: Effect of different concrete ages with respect to compressive strength for (a) 3 cm fiber length and (b) 5 cm fiber length, (Huzaifa, 2008).

Previous researchers have stated that compressive strength of concrete increased with addition of palm oil fibers. Huzaifa (2008) tested 100 x 100 x 100 mm cubes using size fiber of 1, 3 and 5 cm compared with control specimen P_0. The compressive strength of specimens also tested by using different mix of fiber content as 0.25% and 0.50% as shown in Figure 2.12. The results show that with addition of fiber length ranging from 1 cm to 5 cm, the compressive strength increases at 7, 28 and 90 days. Also it has been reported, the highest increases in strength can be up to 37%. The study reported that by adding fiber in higher percentage and longer strand helps to develop compressive strength in concrete. According to Megandran (2007), the increase of compressive strength is only up to certain fiber content (percentage).

2.5.2 Indirect Tensile Strength

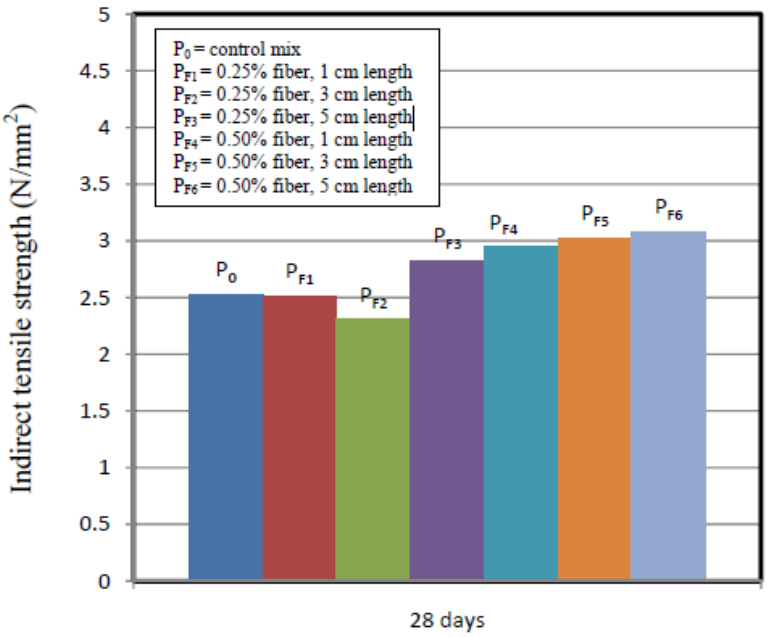


Figure 2.13: Effect of fiber length and content on the indirect tensile strength, (Huzaifa, 2008).

Huzaifa (2008) tested 100 x 200 mm cylinders using fiber length ranging from 1 to 5 cm compared with control specimen P_0 and reported that by adding fiber in higher percentage and longer strands helps to develop tensile strength. From the study, the fiber considered to be like an absorber that absorbs stress when load is applied on the body. With uniformly distribution of fiber the stress is said to be transferred in a complex way that it fails after higher loading is applied. The author reported that P_{F6} yields 22% increase from the control mix and yields twice the strength of P_{F3}.

2.5.3 Flexural Strength

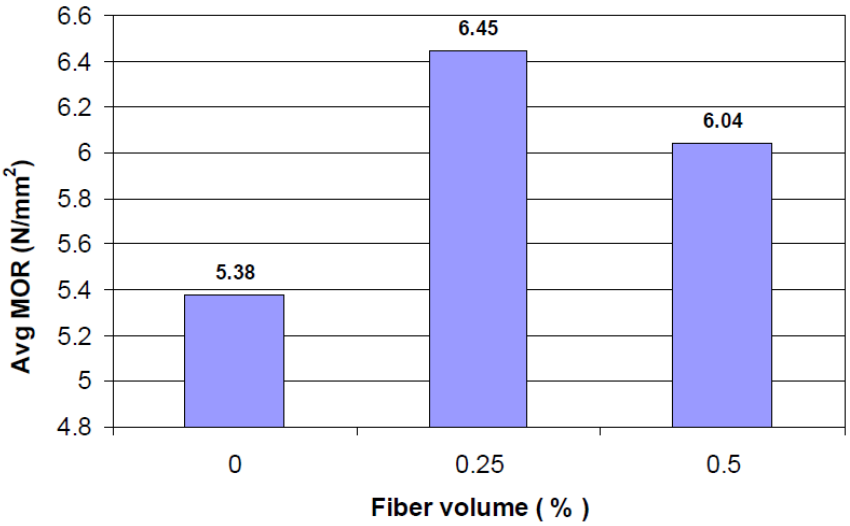


Figure 2.14: Average Modulus of Rupture vs. Volume of fiber, (Megandran, 2007)

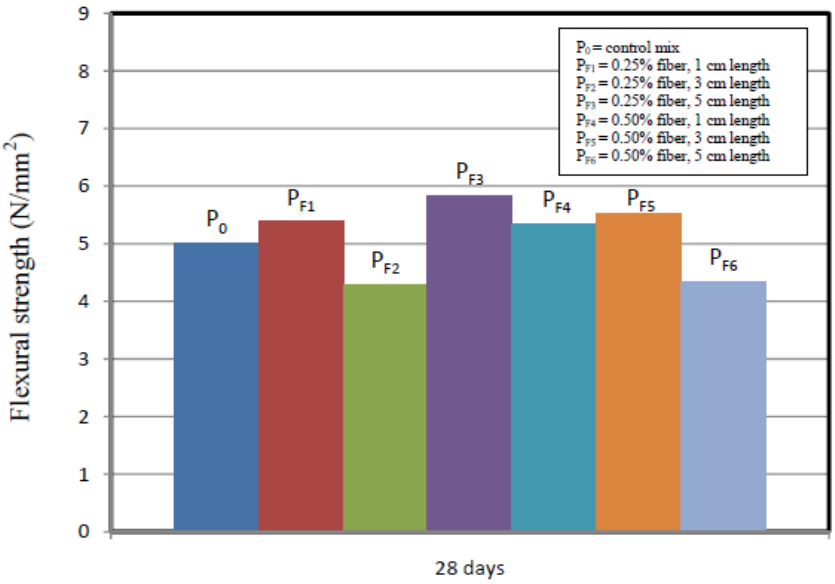


Figure 2.15: Effect of fiber length and fiber content on the flextural strength, (Huzaifa, 2008)

In the research done by Megandran (2007), the modulus of rupture of specimens does not increase linearly with increasing fiber content (percentage). The increase in strength is only up to certain fiber content. The researcher concluded that the congestion of fiber may lead to reduce of bonding and disintegration.

Investigation of Huzaifa (2008) indicated that P_{F6} which has a content of 0.5% fiber and 5 cm length of fiber shows the lowest flexural strength compared with others specimens. The researcher concluded that the reduction might be due to the length of fiber used. The 5 cm fiber for P_{F6} is unsuitable because it tend to give low value of flexural strength due to too much of fiber that contributes to weaker bonding between particles.

2.6 Length size of palm oil fiber used in concrete

The size of the fiber in concrete is important. It has advantages and disadvantages, depending on the length size of the fiber. Though, in fresh concrete properties, the use of small fibers can give more uniform dispersion in the concrete mix as compared to longer fibers. Furthermore, excessive fiber balling was reduced, resulting in better workability of the concrete mix containing shorter fibres, (Muhammadi, 2006)

Adding palm oil fiber to the composites increases the strength of the composite after 28 days. Even though adding fiber contributes to the increase of strength, the strength does not increase with increasing fiber content (percentage), (Megandran, 2007). This study shows that the content of the fiber can give an increment on the strength of hardens concrete. However, the congestion of fiber may lead to a reduce in bonding and disintegration and to reduce the structural integrity in harden concrete.

It is found that the use of fiber in concrete can give a developed higher matrix bonding in the concrete compared with the control mix. This can assist to increase the strength of concrete. The optimum length for the fibers when added in concrete as 0.25% fiber content is 5 cm and when 0.50% of fiber is used the optimum fiber length is 3 cm. With this fiber content, it can lead to an increase of the properties needed, (Huzaifa, 2008).

2.7 Durability

2.7.1 Acid Attack

Portland cement concrete usually does not have good resistance and is vulnerable to acid attack because of its alkaline nature. The components of the cement paste breaks down during contact with acids. Acids attack concrete by dissolving both hydrated and unhydrated cement compounds as well as calcareous aggregate. In most cases, the chemical reaction forms water-soluble calcium compounds, which are then leached away. Siliceous aggregates are resistant to most acids and other chemicals and are sometimes specified to improve the chemical resistance of concrete.

Degradation of the concrete microstructure occurs when unprotected concrete surfaces of sewer pipes, waste water treatment plants, cooling towers and other industrial constructions are attacked by acidic solutions. It will critically limit the service life of the construction components (Beddoe and Dorner, 2005).

Acids such as nitric acid, hydrochloric acid and acetic acid are very aggressive as their calcium salts are readily soluble and removed from the attack front. Other acids such as phosphoric acid and humic acid are less harmful as their calcium salt, due to their low solubility, inhibits the attack by blocking the pathways within the concrete such as interconnected cracks, voids and porosity. Sulphuric acid is very damaging to concrete as it combines acid attack and sulfate attack.

Normally, acidic ground waters are not common, it may be found in landfilled areas, places of mining operation, stock piling of mining tailings have occurred, agricultural and industrial waste (food and animal process industries). Some weak acids however can be tolerated, particularly if the exposure is occasional. Several concrete elements have been reported to be susceptible to the chemical attack of sulfuric, including foundation (groundwater containing sulfuric acid due to oxidization of pyrite in backfill), industrial floors of chemical plants,

basement walls of buildings near chemical plants, superstructures (due to acid rain) (Bassuoni and Nehdi, 2007; Granttan-Bellew, 1995). Table 2.6 shows the types of acid in many industry backgrounds.

Ahmed Budiea (2008) reported that POFA 10 μm concrete has a high quality and great resistance to deterioration in hydrochloric acid solution than control mix OPC. The use of POFA, as pozzalan for partial cement replacement in producing high durable concrete was proved successful.

Table 2.6**:** Common Acids with likely Occurrence (Mindess and Young, 1981)

Type of Acid	Occurrences
Hydrochloric acid	Chemical industry
Nitric acid	Fertilizer manufacture
Acetic acid	Fermentation process
Formic acid	Food processing and dyeing
Lactic acid	Dairy industry
Tannic acid	Tanning industry, peat waters
Phosphoric acid	Fertilizer manufacture

Most distinct is the dissolution of calcium hydroxide which occurs according to the following reaction:

$$2\ HX + Ca(OH)_2 \rightarrow CaX2 + 2\ H_2O$$

(X is the negative ion of the acid)

The decomposition of the concrete depends on the porosity of the cement paste, on the concentration of the acid, the solubility of the acid calcium salts (CaX2) and on the fluid transport through the concrete. Insoluble calcium salts may precipitate in the voids and can slow down the attack.

Corrosion of concrete due to hydrochloric acid can generally be characterized by the following reactions:

$$2HCl + Ca(OH)_2 \rightarrow CaCl_2.2H_2O \quad (1)$$

$$HCl + H_2O \rightarrow H_3O^+_{(aq)} + Cl^-_{(aq)} \quad (2)$$

Table 2.7**:** Acid attack and resistance of concrete

Acid attack increases with	Acid resistance increases with
Increase in acid concentration Constant and fast renewal of acidic solution at concrete/ liquid interface Higher temperature Higher pressure	High Ca++ content in a dense hardened cement paste (low w/cm-ratio) Low proportion of soluble component in concrete Creation of a durable protective layer of reaction products with low diffusion coefficient (transport properties)

Table 2.7 shows the characteristics of environment that can influence to increase acid attack and acid resistance. Concentration of acid plays a vital feature to encourage the acid attack into the concrete structure and also condition of temperature and pressure from environment.

2.7.2 Chloride Penetration

Chloride (Cl^-) is a common anion in soil, groundwater and sea water; in most cases being associated with sodium (Salt). However, the levels of chloride found in the ground are generally chemically innocuous; indeed, they may be beneficial since there is considerable evidence, from seawater studies, that the presence of chloride generally reduces sulfate attack.

A relative performance study of the effectiveness of POFA materials in controlling chloride ion penetration into concrete has been demonstrated by Abd Awal (1998). Incorporating POFA in concrete mix has been identified as a way to improve the resistance to chloride attack. Another researcher, Ahmed Budiea (2008)

also conducted the same study on high strength POFA concrete revealed chloride attack are reduced with replacement of very fine POFA.

The influence of the environment and concrete characteristics on chloride transport into concrete has been studied by the scientific community for several years. Concrete can be harmfully affected by chloride action due to leaching of the free calcium hydroxide which makes concrete porous and weak (Massazza, 1993). Chloride is believed to increase the risk of corrosion of the steel reinforcement. Actually the concrete itself generally provides the embedded steel with a high degree of protection against corrosion.

According to Qiang Yuan *et al.* (2008) silver nitrate colorimetric is used to measure the chloride penetration depth, the precipitate formed on the surface of concrete may be a mixture of silver oxide and silver chloride. It has been disclosed that 0.1mol/ silver nitrate solution is the most proper spraying solution. A simple colorimetric method to measure the depth of chloride penetration into the concrete by spraying $AgNO_3$ solution can be used. Although, this method cannot measure the chloride content in the contaminated area, generally it is an indicative of corrosion risk for reinforcement. (Meck and Sirivivatnanon, 2003)

The depth of cover should be about 75mm in order to protect reinforcement from chloride-induced corrosion (Somayaji, 2001), even a 19mm is enough to provide excellent protection. However, part of reinforced concrete structure that is continuously submerged under water or are outside the splash zone are commonly free from corrosion. Since prolonged immersion reduced the potential for corrosive action.

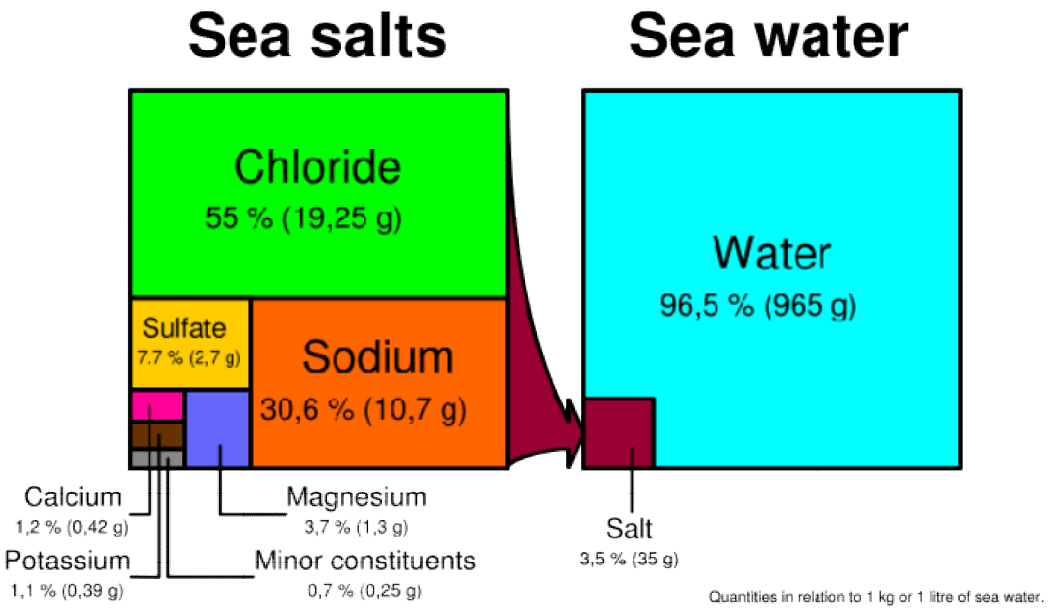


Figure 2.16: Concentration of components in sea water

Sea water contains about 2.7% NaCl, 0.32% $MgCl_2$, 0.22% $MgSO_4$ and 0.02% $KHCO_3$. The main aggressive compounds in sea water are the chlorides and sulphates combined with sodium and magnesium (Grattan-Bellew, 1995). Figure 2.16 shows the elements in proportion and concentration representative of ocean water.

CHAPTER 3

RESEARCH METHODOLOGY

3.1 Introduction

This chapter deals with the methodology that is used in the research that going to be carried out on palm oil fiber concrete. This research focuses on the durability aspects of the palm oil fiber concrete and the strength of the concrete with certain content of fiber. It will focus on their compressive strength, resistance to acid attack and penetration of chloride.

3.2 Methodology Flow Chart

This flow chart demonstrates the process of this research starting from the beginning of the choosing of a title till completion of the research.

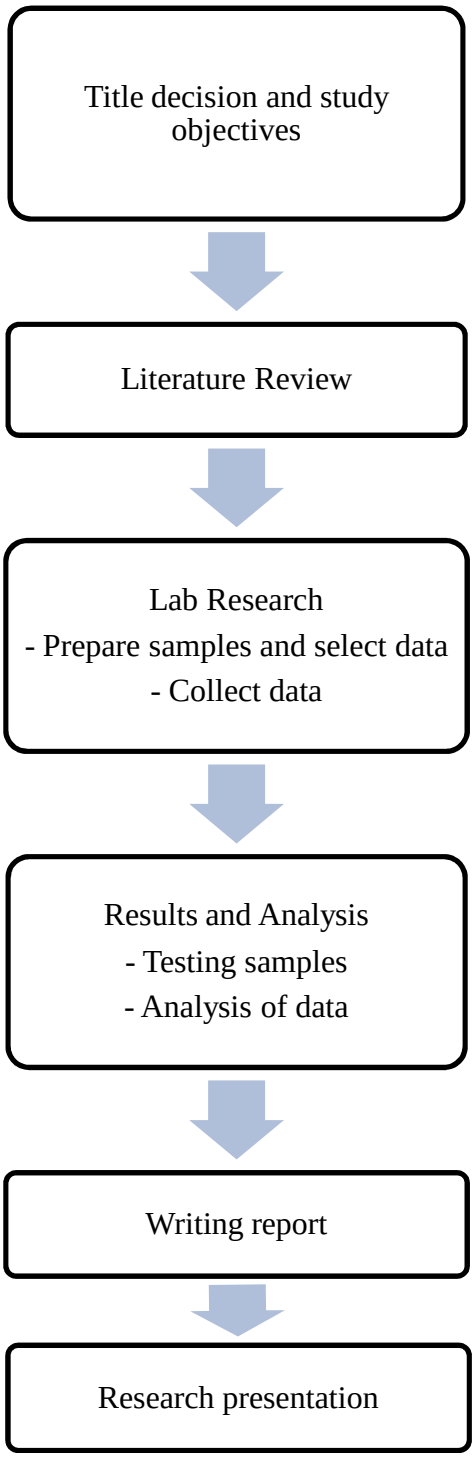


Figure 3.1: Research methodology flow chart

3.3 Materials Preparation

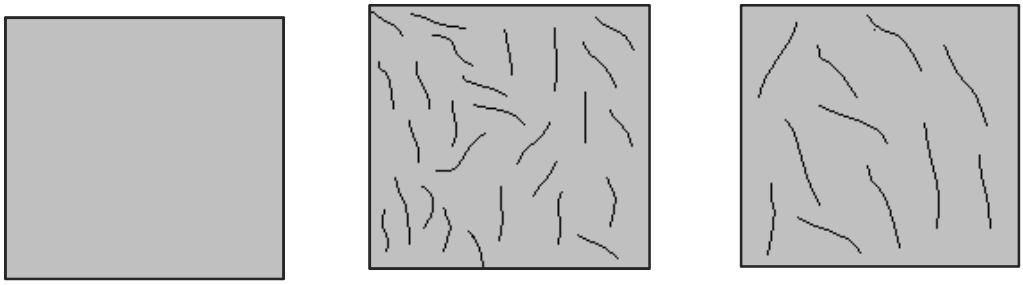
3.3.1 Palm Oil Fiber

Based on the past research (Huzaifa, 2008), this research carried out using fiber content and fiber length that has been recommended. In this research 3 cm and 5 cm fiber length with fiber content of 0.5 percent and 0.25 percent were chosen respectively as shown in Table 3. 1. This fiber is obtained from Fiber-X (M) Sdn Bhd.

Table 3.1: Fiber content in each concrete mix

Specimen	Fiber Content % *	Fiber length (cm)	W/C
Control Mix, P_0	-	-	0.45
P_{F1}	0.5%	3	0.45
P_{F2}	0.25%	5	0.45

*percent of fiber by of weight cement



P_0 P_{F1} P_{F2}

Figure 3.2: Illustration of specimens P_0, P_{F1} and P_{F2}

3.3.2 Cement Portland

Ordinary Portland Cement (ASTM type I) is commonly been used for this research and it came from manufacturer of the OPC which is Holcim (M) Sdn. Bhd with 50 kg for every bag. This cement is safely keep in dry place condition and the humidity is under control. Figure 3.3 shows the cement bag.

Figure 3.3: Cement bag

3.3.3 Water

Water used in this research is obtained from general supply water system in mixing, curing and others. The water is clean with no strange material in it that can disturb the hydration process of cement.

3.3.4 Fine Aggregates

Fine aggregates used in this research size 5 mm to filler. Fine Aggregate or sand works as filler in concrete should comply with coarse, medium, or fine grading requirements of MS: 30 Part 2, 1995.

In this study, sand used is air dried to obtain saturated surface dried. To obtain this condition, sand need to be dried in room condition for 24 hour. Sieve

analysis was done prior to using it to determine the fine aggregate passing 600 μm sieve.

3.3.5 Course Aggregates

In this study, crushed aggregates from quarry with the nominal size 10 mm in accordance to BS 882, 1992 are used. The course aggregate was dried to obtain saturated surface dried condition to ensure that water cement ratio was not affected.

The characteristics of the aggregates will affect the mix design, compressive strength, and workability is shape, texture, gradation and moisture content. The aggregate component of a concrete occupies 60 to 80 percent of the volume of concrete.

3.4 Concrete Mixtures

The method of mix design applied is according to the method published by Department of Environment UK (1988).

3.4.1 Method of Design

Stage 1:

Characteristic Strength	: 30 N/mm^2 at 28 days (Proportion Defective 5%)
Standard Deviation	: 8 N/mm^2
Margin	: (k=1.64), 1.64 x 8 =13.12
Target Mean Strength	: 30 + 13.12 = 33.12 N/mm^2

Cement type : OPC

Aggregate Type: Course : Crushed

Aggregate Type: Fine : Uncrushed

Free Water Cement Ratio : 0.45

Stage 2:

Slump : 30 - 60 mm

Maximum Aggregate Size : 10 mm

Free Water Content : 215.00 kg/m^3

Stage 3:

Cement Content : 215.00/0.45 = 478 kg/m^3

Stage 4:

Relative Density of Aggregate: 2.7

Concrete Density : 2360 kg/m^3

Total Aggregate Content : 2360 – 215 – 478 = 1667 kg/m^3

Stage 5:

Grading of the Aggregate : Percentage Passing 600 µm Sieve: 45 %

Proportion of Fine Aggregate : 45 %

Fine Aggregate Content : 1667 x 0.45 = 750 kg/m^3

Course Aggregate Content : 1667 x 0.55 = 917 kg/m^3

Volume of concrete for each batch = 20 x 0.001 + 9 x 0.0016

= 0.0344 m^3

Volume for each batch + 40% safety factor and other test = 0.0482 m^3

≈ 0.050 m^3

3.5 Mixing Process

This mix process is carried out using conventional mixer. In mixing process, fiber was added in portions at the final stage after all the concrete ingredients were mixed. This method of mixing can give and produce uniform dispersed fiber in concrete mix. Figure 3.4 shows the concrete mixer that been used.

Figure 3.4: Concrete mixer

3.6 Method of Curing

The curing process starts after demoulding, the specimens were cured in water in curing tank before testing for 28 days. Figures 3.5 to 3.7 show the curing process.

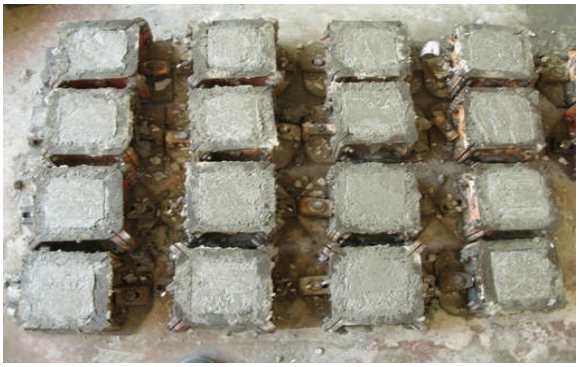


Figure 3.5: Concrete in moulds

Figure 3.6: Mixture cured by gunny sag

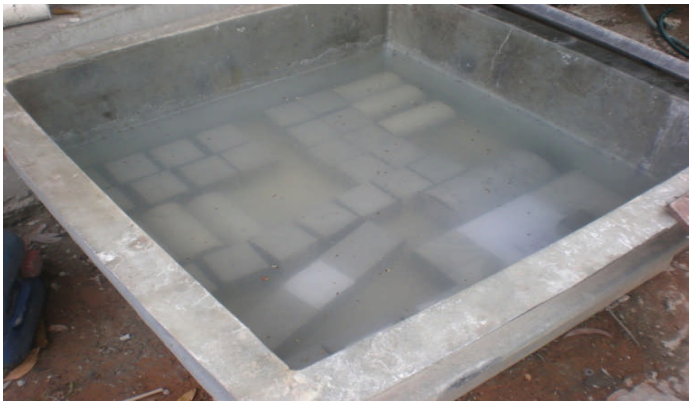


Figure 3.7: Concrete cured in water tank

3.7 Tests on Concrete

Tests on concrete consist of testing on fresh concrete and hardened concrete.

3.7.1 Tests on Fresh Concrete

Specimens of concrete are needed to be tested to represent the condition of the concrete mix. It spells out procedures for sampling various production systems and specifies a sample size of 1 cu ft except for routine Slump and Compacting factor tests. The specimen must be tested within 15 minutes and during testing, it must be protected from the weather.

3.7.1.1 Slump test

ASTM C143 test for slump of Portland cement concrete details the procedure for performing Slump tests on fresh concrete. A slump cone is filled in three layers of equal volume so the first layer is about 4 in. (76 mm) high, and the second layer is 6 in. (155 mm) high. Each layer is rodded 25 times with a tamping rod 24 in. (600 mm) long and 0.63 in. (16 mm) diameter, with a hemispherical tip with 16mm diameter. The rodding is uniformly distributed and full depth for the first layer and just penetrating previous layers for the second and third layers. Strike off the surface of concrete by a screeding motion and rolling the rod across the top of the cone. Figure 3.8 shows the slump of concrete mix.

In 5 ± 2 seconds, raise the cone straight up. Set the slump cone next to the concrete, and measure the difference in height between the slump cone and the original center of the specimen. With the rod set on the cone, this slump measurement can be read to the nearest 0.23 in. (6mm). The test from filling of

the slump cone to measuring the slump should take no longer than 2 minutes. Figure 3.9 shows the method to measure the slump. If two consecutive tests on a sample show a falling away of a portion of the sample, the concrete probably lacks the cohesiveness for the Slump test to be applicable.

Figure 3.8: Slump test

Figure 3.9: The slump was measure in terms of millimetre unit

3.7.1.2 Compacting Factor Test

This test is to measure the degree of compaction resulting from the application of a standard amount of work according to BS 1881-103, 1993. The apparatus as shown in Figure 3.10, which is commercially available, consist of a rigid frame that supports two conical hoppers vertically aligned above each other and mounted above a cylinder. The top hopper is slightly larger than the bottom hopper, while the cylinder is smaller in volume than both hoppers. To perform the test, the top hopper is filled with concrete but not compacted. The door on the bottom of the top hopper is opened and the concrete is allowed to drop into the lower hopper. Once all of the concrete has fallen from the top hopper, the door on the lower hopper is opened to allow the concrete to fall to the bottom cylinder. A tamping rod can be used to force especially cohesive concretes through the hoppers. The excess concrete is carefully struck off the top of the cylinder and the mass of the concrete in the cylinder is recorded. This mass is compared to the mass of fully compacted concrete in the same cylinder achieved with hand rodding or vibration. The compaction factor is defined as the ratio of the mass of the concrete compacted in the compaction factor apparatus to the mass of the fully compacted concrete.

Figure 3.10: Compacting factor test

3.7.2 Test on Hardened Concrete

3.7.2.1 Compressive Strength

Compressive strength test for this research is using iron mould size 100 x 100 x 100 millimetre. Specimens are tested after 7, 28 and 90 days. Figure 3.11 show the compressive strength setup and specimens after testing.

The compression test was conducted by using compression test machine at the material lab of Civil Engineering Faculty of UTM as specified in the test method BS 1881-Part 116,1983. An increasing compressive load was applied to the specimen until failure occurred to obtain the maximum compressive load. The specimen dimension was taken before the testing. The testing was carried out for 28 days specimen after curing.

$$\text{Compressive Strength} = P/A \qquad (3)$$

Where :

P : Ultimate compressive load of concrete (kN)

A : Surface area in contact with the platens (mm^2)

Figure 3.11: Compressive strength test setup and specimens after testing

3.7.2.2 Indirect Tensile Strength Test

The most commonly used tests for estimating the tensile strength of concrete is the ASTM C 496 splitting tensile strength of cylindrical concrete specimen. For this test, concrete cylinder is subjected to compression loads along two axial lines which are diametrically opposite. The specimen is applied constant load range of 0.69 to 1.38 MPa until the specimen fails.

In order to do the colorimetric method on the specimens, first, the specimen needs to be split. The test was carried out on specimens, so the specimens can expose its concrete inner and ready to be tested for determining chloride ingress. Figure 3.12 shows the indirect tensile strength test setup.

Figure 3.12: Indirect Tensile Strength Test

3.8 Determination of Chloride Penetration

The objective of test is evaluating the performance of palm oil fiber concrete in resisting the penetration of chloride ion. Nine cylinders of 200 x 100 mm were cast for each concrete mixture (P_O, P_{F1} and P_{F2}), then were immersed in 3% sodium chloride solution for period of 7, 28 and 90 days as shown in Figure 3.13. Before putting any specimen in the solution, specimen needs to be cured in water for 28 days.

At the specified period of emersion, the specimens were removed to test for chloride penetration. At that time, identical cylinder concrete was split along their length. Then, the exposed cross section was sprayed with 0.2 N silver nitrate solution. A change of white colour will appear due to the form of silver chloride, while area not penetrated will appear as a brown precipitate due to reaction of hydroxide ions to form silver oxide.

When the reading is not uniform, six reading were taken using vernier calliper on each cylinder along two side of a splitted specimen. Therefore, the average of 3 x 3 = 9 readings give the actual depth of chloride penetration for each condition. Ravindrarajah and Moses (1993), Abdul Awal (1998), and Ahmed Budiea (2008) have used similar method successfully to determine the depth of penetration of chloride ions.

Figure 3.13: Specimens in 3% chloride solution pond

3.9 Resistance to Acid Attack

To investigate this test, concrete cube specimens (100 x 100 x 100 mm) were used. These cubes were prepared and cured in water for 28 days before putting them into the hydrochloric acid solution. Figure 3.14 (left) shows the specimens immersed in 1% Hydrochloric Acid.

The durability performance of both OPC and POFC concrete specimen were determined by periodic measurement of weight losses of the samples continuously immersed in the solution. Figure 3.14 (right) shows a specimen being weighed. The immersion period is up to 1800 hours. The pH of the solution was controlled to 2. At the end of the day, compressive strength test was conducted to test the strength performance of POFRC in water cured versus immersed in acid solution.

Figure 3.14: Specimens immersed in 1% Hydrochloric Acid solution pond (left) and a specimen weighed after certain period of time (right).

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Fresh Concrete Testing

Fresh concrete testing involved slump test and compacting factor test. These tests conducted to determine workability and consistency of each concrete mix.

Workability is the ability of a fresh (plastic) concrete mix to fill the form/mold properly with the desired work (vibration) and without reducing the concrete's quality. The main factors that influence to workability of palm oil fiber reinforced concrete are water content, cement content, characteristic of aggregate and fiber content.

4.1.1 Slump Test

The slump test is prescribed by BS1881: Part 102: 1983. The Slump test results are shown in Table 4.1. The slump was measured in terms of millimetre unit. Figure 4.1 shows the results of the slump test. It shows that the slump value is within the design range, 30–60 mm. This may be due to the fact that the aggregates have

achieved the saturated surface dry (SSD) condition and no excessive water exist in the mix. The control fresh concrete mix, P_0 has the highest workability by show their highest value of slump.

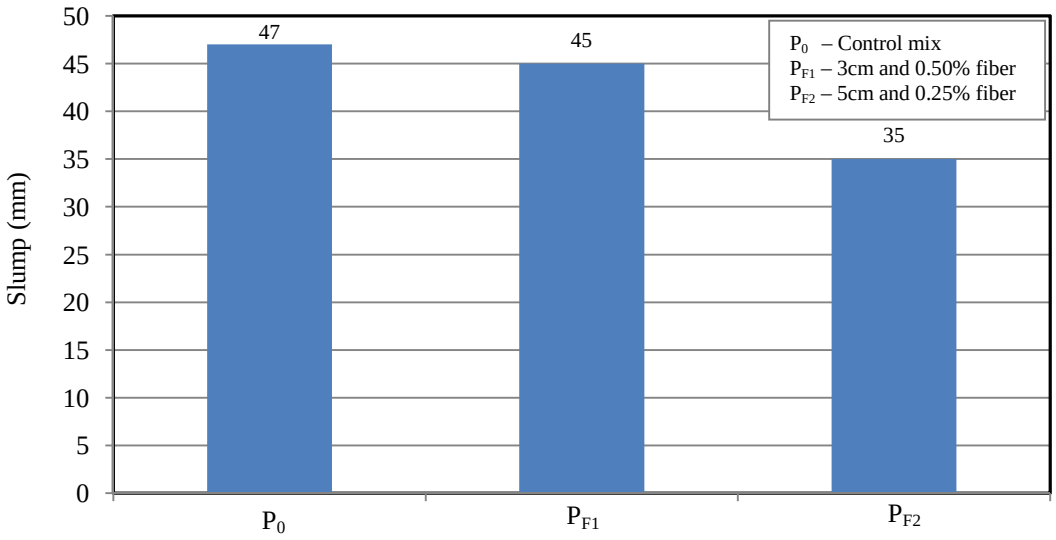


Figure 4.1: Height slump vs. concrete mix

4.1.2 Compacting Factor Test

Figure 4.2 shows the compacting factor results. The highest workability is for P_{F2} with compacting factor of 0.93. With an increase of fiber content in the mix, the workability becomes lower. The decreasing of workability is due to fiber dispersal in concrete mix that makes it more viscous.

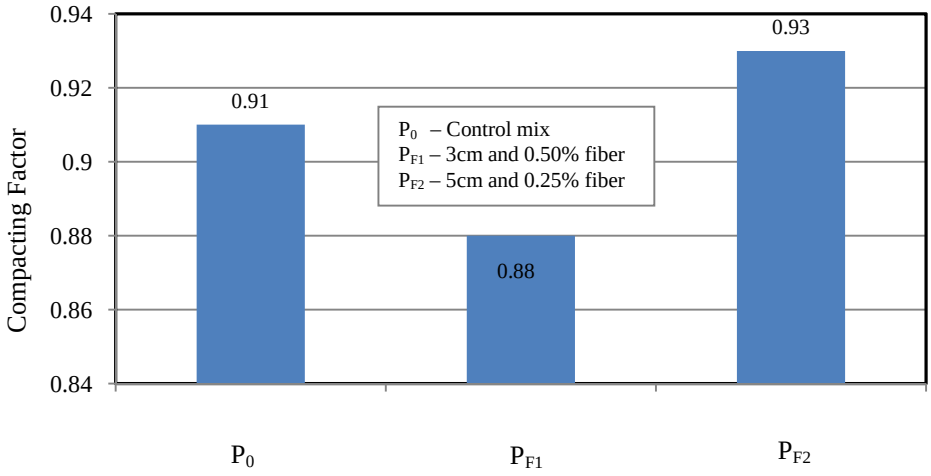


Figure 4.2: Compacting factor test result

4.2 Concrete Density

Table 4.1 shows the average density of the used concrete mixes. The design density was 2360 kg/m^3.

Table 4.1: Concrete Density for the used mixes

Concrete mix	Usage / Age	Cross Section Area (mm^2)	Density kg/m^3	Average Density kg/m^3
Control Mix P_0	Compressive / 28 days	1x10^4	2370	2390.6
		1x10^4	2400	
		1x10^4	2390	
		1x10^4	2380	
		1x10^4	2410	
	Acid Attack / 28 days	1x10^4	2395	
		1x10^4	2380	
		1x10^4	2400	
		1x10^4	2410	
P_{F1}	Compressive / 28 days	1x10^4	2420	2402.5
		1x10^4	2385	
		1x10^4	2385	
		1x10^4	2395	
	Acid Attack / 28 days	1x10^4	2420	
		1x10^4	2420	
		1x10^4	2385	
		1x10^4	2395	
P_{F2}	Compressive / 28 days	1x10^4	2380	2398.1
		1x10^4	2360	
		1x10^4	2405	
		1x10^4	2480	
	Acid Attack / 28 days	1x10^4	2390	
		1x10^4	2390	
		1x10^4	2385	

The control mix specimen, P_0 gives average result of 2390.6 kg/m^3, while P_{F1} gives an average of 2402.5 kg/m^3 and P_{F2} specimen gives an average result of 2398.1 kg/m^3. It is clearly shown that the control mix, P_0 has slightly lower density than both concrete mix with 0.25% and 0.5 % fiber. Concrete mix with fiber is more dense compared with normal concrete. It shows that higher percentage of fiber was added into concrete mix, resulting in higher density of concrete mix. This might

happen due to the fact that the fiber content filled in concrete mix make it more dense concrete. The results of concrete density are shown Figure 4.3.

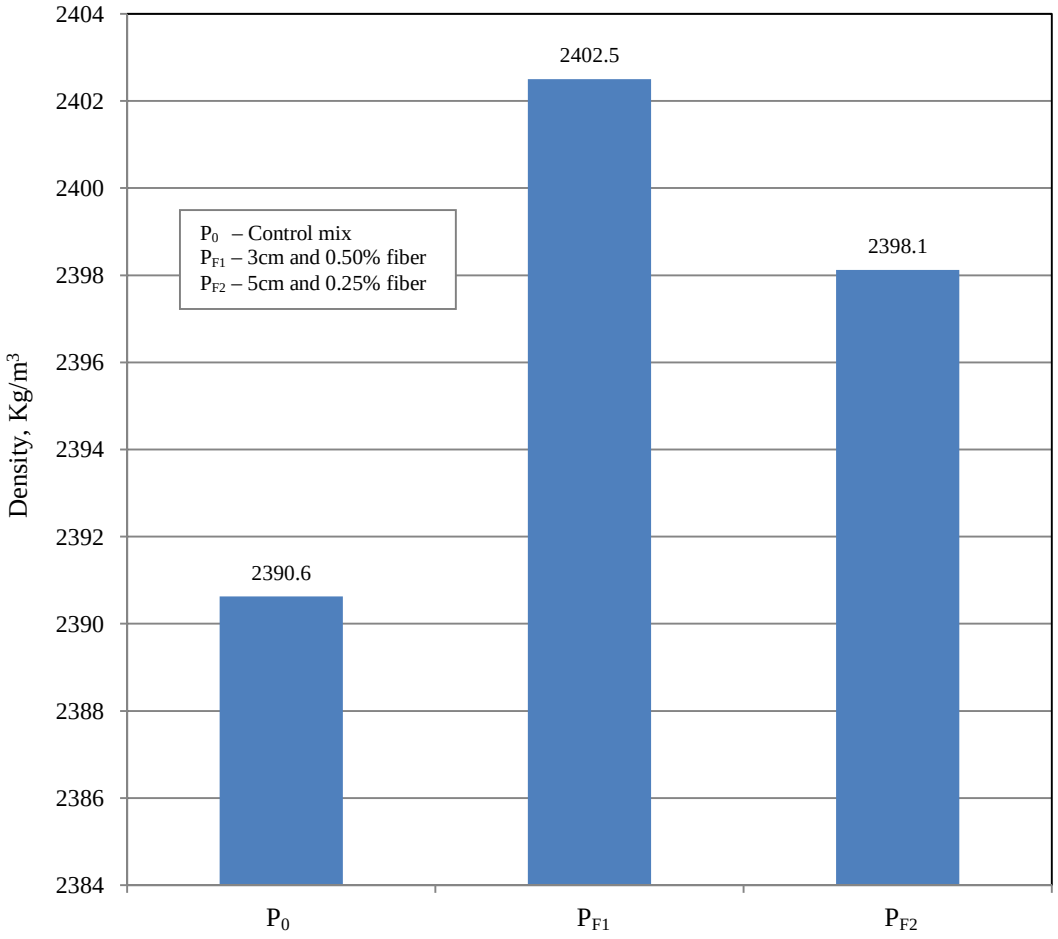


Figure 4.3: Density vs. Concrete Mix

4.3 Hardened Concrete Testing

The hardened concrete test conducted was compression test. They are conducted to control the quality of the concrete and to check specification compliance. The most common test performed on the hardened concrete is the compressive strength test. This is because it's fairly easy to perform and shows strength correlation between the compressive strength and many desirable properties, (Mamlouk, 2006).

4.3.1 Compressive Strength

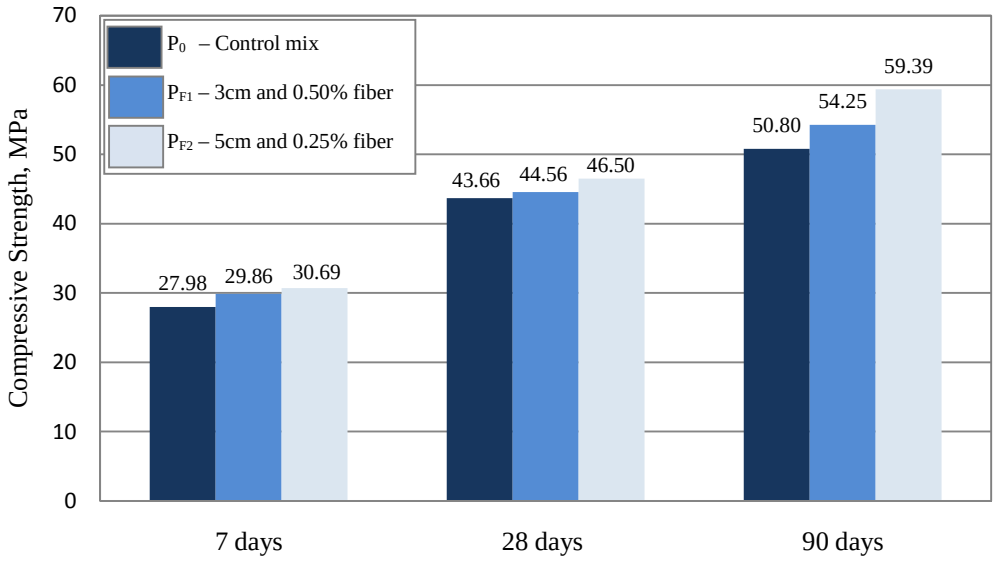


Figure 4.4: Chart of Compressive Strength at 7, 28 and 90 days

Figure 4.4 shows the results of the compressive strength of specimens used in the test, P_0 – control concrete mix, P_{F1} – concrete mix with 0.5 % and 3 cm fiber length, and P_{F2} concrete mix with 0.25 % and 5 cm fiber length. These tests were conducted at 7, 28 and 90 days in order to get their strengths. Concrete mix was designed with compressive strength of 30 MPa for 28 days.

The result of the experiment shows that adding palm oil fiber to the concrete mix increases the strength at 7, 28 and 90 days respectively compared with the control mix. The mix P_{F2} shows the highest average compressive strength (59.39 MPa) from the results. This result is in agreement with the research done by Huzaifa (2008). Although the additional of fiber can contribute to increasing compressive strength, the strength doesn't increase linearly with an increase of fiber content, (Megandran, 2007). However, with the increase of fiber length from 3 cm to 5 cm, the compressive strength increases for all duration of time.

From this figure, as can be seen, fiber mixed with the concrete increases the compressive strength slightly compared to normal concrete. According to Mamlouk (2006), the addition of fibers to concrete does not greatly increase the strength of concrete.

Fiber reinforced concrete helps to make the concrete more tough and durable to avoid the cracks from happening. While compressive strength test is conducted, time of concrete mix P_{F1} and P_{F2} to fail were longer than time of the control mix P_0. According to Mamluok (2006), fiber-reinforced concrete can sustain load even after initial cracking, which means that the time for micro-crack to occur are withhold when the fibers upholds the concrete binder and course aggregate are good, and prolong the failure time.

The results also show that longer fiber gives higher strength, with the understanding that the longer fiber is more effective in arresting micro-cracks. According to Mohammadi *et al*. (2006), he observed that the post-cracking resistance of fiber-reinforced concrete was considerably influenced by the length, orientation and also stiffness of fibers used.

4.4 Durability Performance

4.4.1 Resistance to Acid Attack

After 28 days curing in water, 4 specimens from mixture were immersed in a 1% hydrochloric acid (HCl) solution for 1800 hours days in a container. In order to minimise the evaporation, these specimen were kept covered throughout the testing period. And also, similar specimens from each batch were kept in water up to 1800 hours days to compare strength values after 28 water curing.

The pH of the solution was controlled to 2. The pH value of the solution observed was increased with time due to decomposition of the concrete specimens in to the solution.

Acid resistance was evaluated by determining the weight loss (WL) and compressive strength loss (SL) of the specimens using this equation:

$$\text{WL}\ (\%) = \frac{w1 - w2}{w1} \times 100\ \% \tag{4}$$

where w_1 and w_2 are the weights of the specimens (in kilograms) before and after immersion

$$\text{SL}\ (\%) = \frac{fc1 - fc2}{fc1} \times 100\ \% \tag{5}$$

where f_{c1} represents 1800 hours compressive strength of control specimens and f_{c2} is the compressive strength of the specimen after exposure to 1% hydrochloric acid (HCL) solution for 1800 hours.

4.4.1.1 Weight Loss

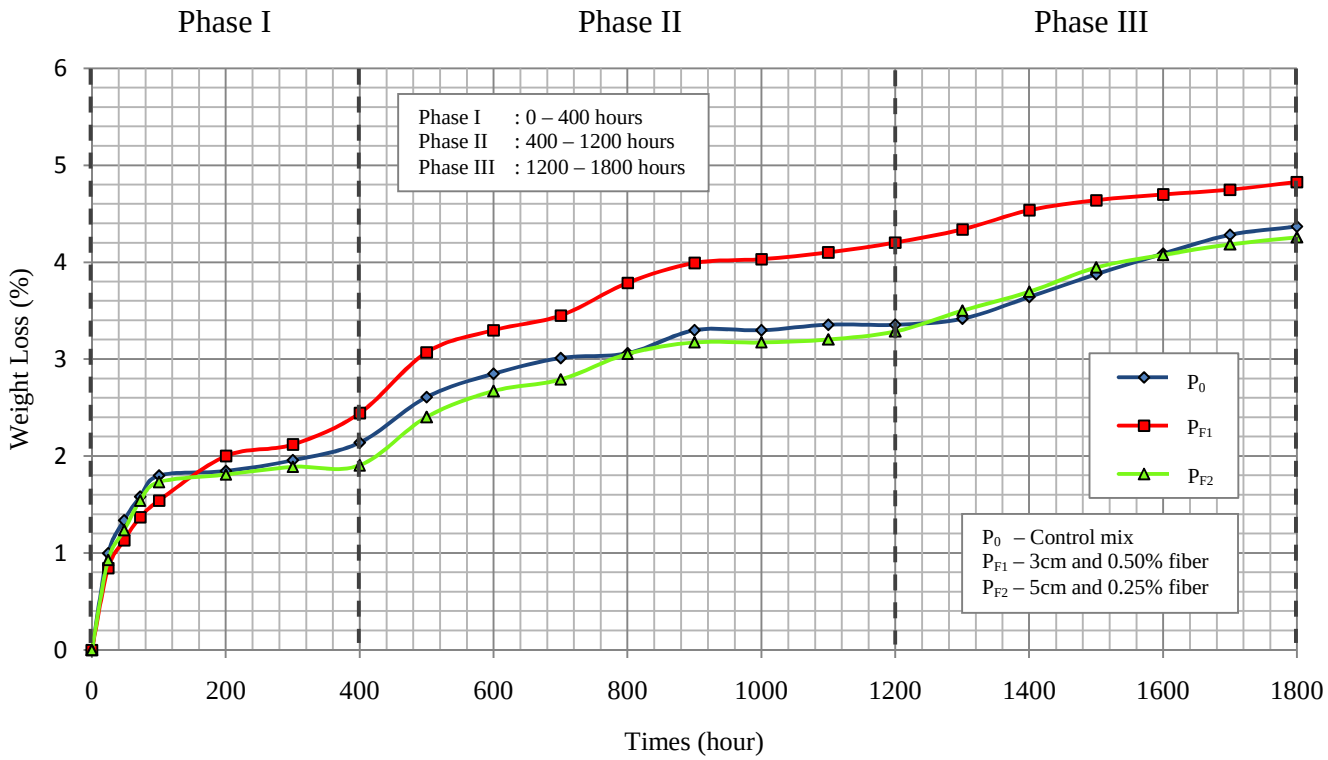


Figure 4.5: Rate of weight loss over the time

The weight loss results for all specimens are shown in Figure 4.5. The graph is divided into 3 phases, based on replacement of new acid solution. It can be observed that at time from 200 until 400 hours weight loss of specimens remains constant, same as at 900 – 1200 hours weight loss also remains constant. As a result no weight loss is observed. Initial pH measured was less than 2, pH increase to more than 3 as the immersion time increases. The increase in pH is the result of leaching of calcium hydroxide from the cement matrix into the solution. The presence of hydroxyl ions, OH^- will increase the pH from less than 2 to more than 3.

$$Ca(OH)_2 \rightarrow Ca^{2+} + 2OH^- \quad (6)$$

The constant weight indicates that the system has achieved equilibrium, whereby the solution has reached a saturation limit, hence dissolution of the cement matrix ceased to occur.

At the Phase I, the difference in weight loss for mixtures P_0, P_{F1} and P_{F2} was almost constant. However P_{F1} shows the lowest weight loss during that stage. During the second stage, P_{F1} shows significant loss in weight over P_0 and P_{F2}. At the same time, weight losses of P_{F2} and P_0 are constantly increasing. At the end of the phase III, the weight loss ranged up to 5%. Similar rate of weight loss of P_0 and P_{F2} can be observed. Thus, P_{F2} shows great potential to resist deterioration same as control specimen P_0.

Hydrochloric Acid solution reacts with fiber through decomposition. Fiber decomposed with this solution will allow the solution to penetrate more depth into the concrete mix. Concrete mix P_{F1} which contains the highest fiber content (0.50%) might be exposed to this solution more than other specimens. So chances to loss their weight is higher than other specimens.

It was observed that some fibers were dislodged from the surface of specimens due to dissolution/leaching during the very severe acidic exposure of the solution. Also it was observed that the fibers were decomposed and disintegrated; and fibers crumble on the concrete surface. Thus, in acidic environments, the criterion of weight loss may relatively overestimate the degradation rate of concrete mixtures incorporating fibers.

According to research performed by Bassuoni and Nehdi (2006), the role of fiber reinforced in controlling cracks and increase the tensile capacity of the cementitous matrix can lead to improved matrix integration in acidic environments by limiting swelling and disruptive pressure from voluminous reaction product. The authors suggested to do microanalysis so the initiation and propagation of microcracks in the cementitous matrix due to gypsum and ettringite formation resulting from the acid attack were revealed.

4.4.1.2 Compressive Strength Loss at 1800 hours

After 1800 hours exposure, specimens were tested for residual compressive strength, which was calculated using the original cross-sectional area, determined the strength loss with reference to the strength of concrete without exposure.

The residual compressive strength of test cube after immersing in hydrochloric acid was determined and the result were shown in Figure 4.6. The control specimen, P_0 is the highest compressive strength compared with other specimens, P_{F1} and P_{F2}, when immersed in acid solution. However, the P_{F1} specimen had the lowest compressive strength. As discussed earlier, this can be mainly due to the highest of deteriorate fiber content relative to the P_{F1} specimen. The function of fiber reinforcement in increasing the compressive strength can lead to opposite results, which means the highest of fiber content concrete specimen can lower the strength.

Figure 4.7 shows the compressive strength of each specimen at 1800 hours which is conducted same time with specimens immersed in the acid solution. The result pattern is still similar to the compressive strength at 90 days. At 1800 hours, the P_{F2} produces the highest strength of 60.26 MPa which is 14% higher than control mix P_0 (52.91 MPa).

The percentages of compressive strength loss for all mixtures are shown figure 4.8. The results were obtained using Equation 2, where shows the different compressive strength between in acid and water cured at 1800 hours. The rate of weight loss increased due to addition of fiber and resulted in weight losses of 50.1%, 66.1% and 57.9 % for P_0, P_{F1} and P_{F2} respectively at 1800 hours. From the figure, the residual compressive strength of P_{F1} drops sharply in hydrochloric acid compared with other specimens.

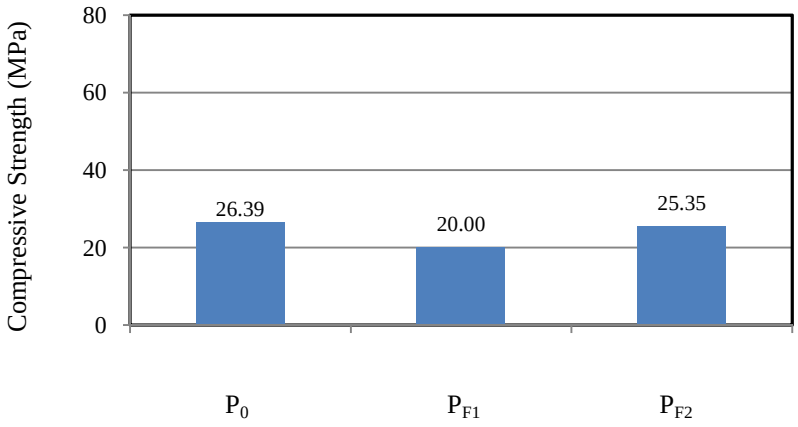


Figure 4.6: Compressive Strength at 1800 hours in 1% Hydrochloric Acid solution

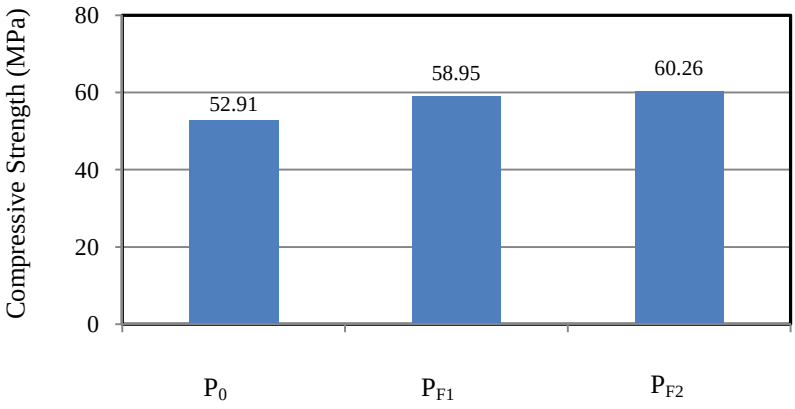


Figure 4.7: Compressive Strength at 1800 hours in water cured

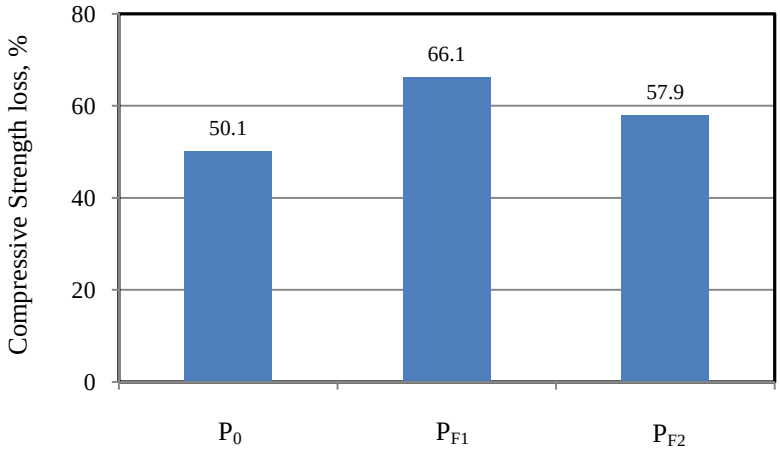


Figure 4.8: Compressive Strength Loss at 1800 hours

4.4.1.3 Relationship between weight loss and compressive strength loss

The relationship between weight loss and compressive strength is shown in Figure 4.9 as a percentage. It had been suggested by Bassuoni and Nehdi (2006) that there was a correlation between weight loss and compressive strength after exposure hydrochloric acid solution. Most of the available literature on the resistance of concrete to sulfuric acid attack has not discussed the correlation between weight loss and strength loss.

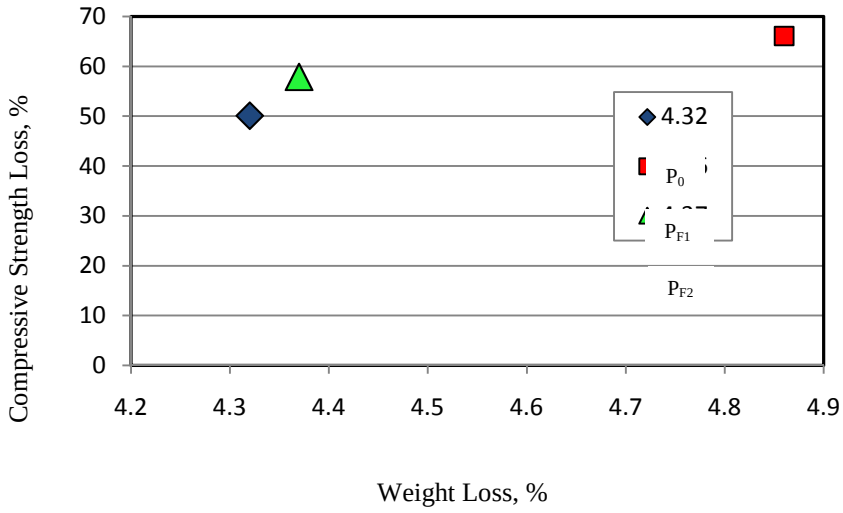


Figure 4.9: Weight loss (%) versus compressive strength loss (%) after 1800 hours

It can be observed that data points are scattered and give a trend. Compressive strength did have a direct relation with weight loss of specimen under hydrochloric acid attack. P_{F1} shows high of compressive strength loss and weight loss compared with control mix P_0 and P_{F2}. In most cases, it may be suitable to express the rate of concrete deterioration due to hydrochloric acid attack in terms of compressive strength loss.

As for conclusion, specimen P_{F1} shows worse effect against acid solution. Thus, it can be concluded that P_{F2} and P_0 exhibited better resistance against to acid. Hence, P_{F2} can be widely used as concrete structure in low concentration of acidic environment.

4.4.1.4 Visual Inspection

Visual Inspection on specimens was conducted to determine any physical changes in the specimens. Generally, there is not much difference in the physical appearance for specimens after being immersed in acid solution except foredges and corner losses that can be noticed for all specimens. In addition, gray solids could be observed at the bottom of the container presumably originate from the broken corners. When the specimens are exposed to HCl solution, the acids will dissolve the surface. C-S-H of cement as well as leaching the calcium hydroxide in cement. According to Ahmed Budiea (2008), colour change for control mix P_0 should be observed. In this research, the colour change could not be observed since the specimens were weighed every 100 hours, hence any colour change was washed away due to frequency of weighing the specimens. Figure 4.10 shows specimens before being exposed to the acid solution and Figure 4.11 shows the specimens at 1800 hours after being exposed to 1% hydrochloric acid solution.

(a)

(b)

(c)

Figure 4.10: The specimens before exposure to hydrochloride acid solution (a) control specimens P_0 (b) P_{F1} and (c) P_{F2}

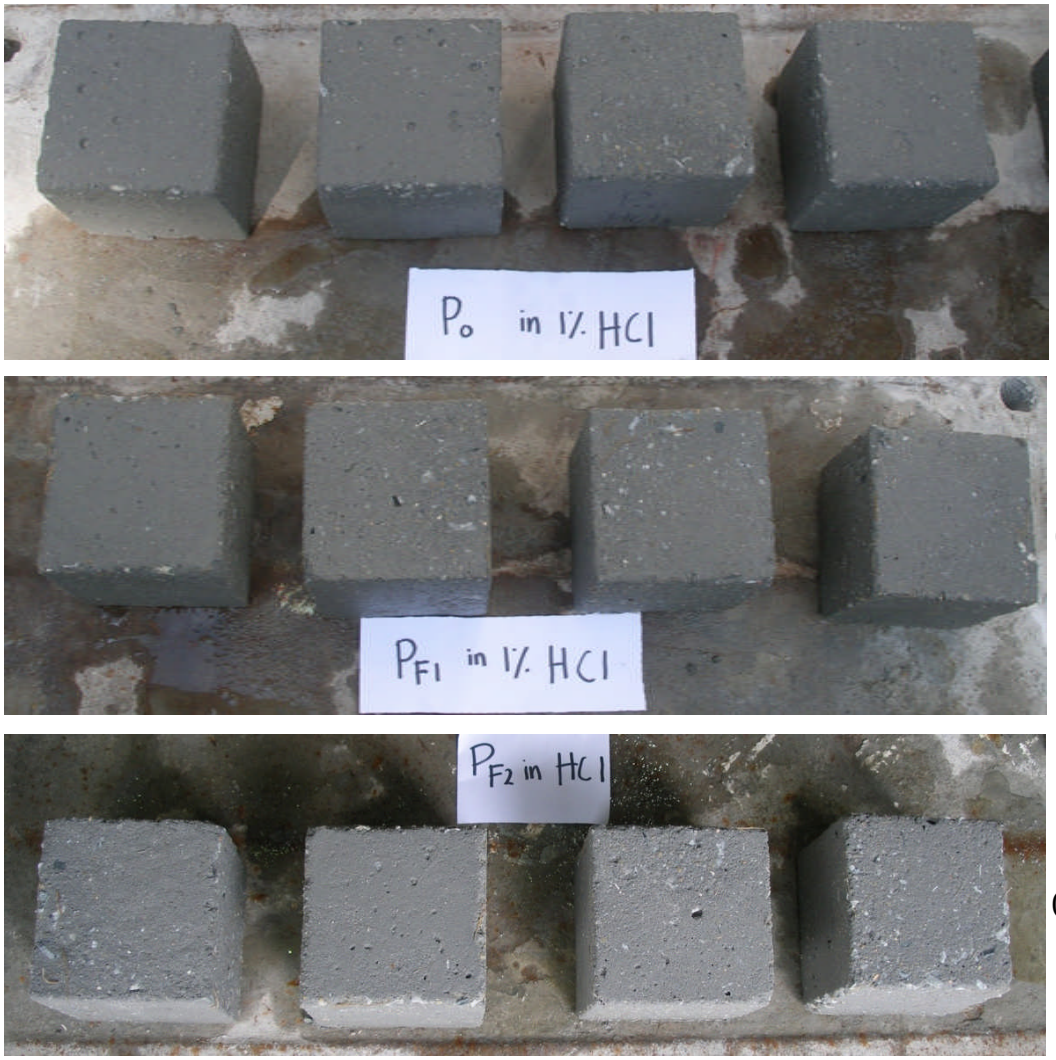


Figure 4.11: The specimens after 1800 hours exposure to hydrochloride acid solution (a) control specimens P_0 (b) P_{F1} and (c) P_{F2}

Figure 4.12: P_0 at 1800 hours in water cured (left) and hydrochloric acid (right)

Figure 4.13: A piece of P_{F1} after 1800 hours immersed in hydrochloric acid

Figure 4.12 shows the specimens P_0 after conducting compressive strength test at 1800 hours. Specimen concrete which was immersed in hydrochloric acid revealed 2 layer of deterioration. The inner layer shows the brown layer and the out side layer shows the grey layer. The morphology of the damaged layers can be seen under FESEM and while the elemental analysed was determined by EDX analysis.

4.4.2 Resistance to Chloride Penetration

4.4.2.1 Chloride Penetration

The silver nitrate colorimetric method is a very easy and quick way to measure the free chloride penetration depth in concrete. In this method, silver nitrate solution with a concentration of 0.2N is sprayed onto the freshly fractured cross-section of concrete. Consequently, the silver ions react with the chloride ions to form a white precipitate of silver chloride. In the chloride free or low chloride ion area, the silver ions react with the hydroxyl ions to form a brown precipitate of silver oxide. There is a very obvious boundary between the grey and brown areas.

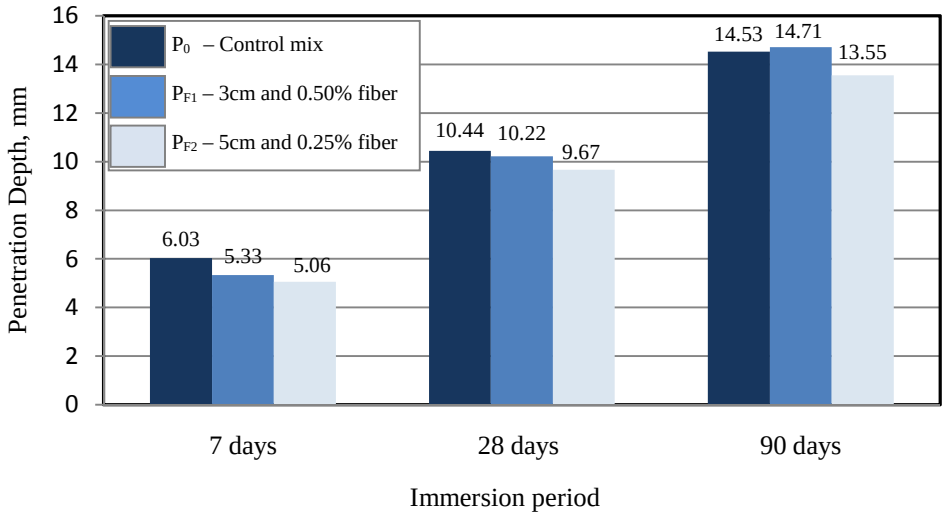


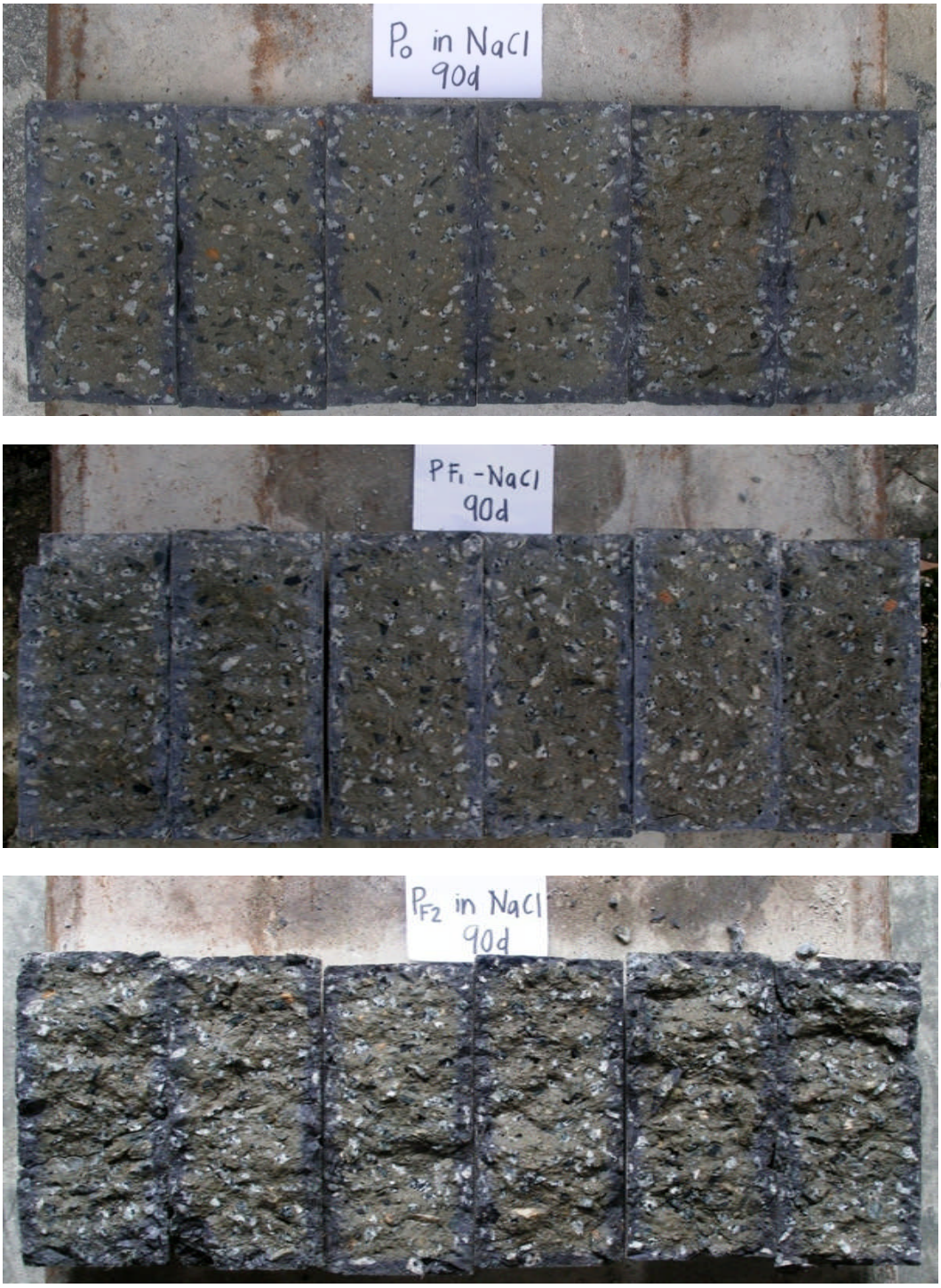
Figure 4.14: Depth of chloride ion penetration through various concrete mixes

The depth of chloride ion penetration in concrete at the colour change boundary can obviously be identified. Six readings were taken on each cylinder along two side of a split specimen using vernier calliper. Figure 4.14 shows the results of chloride penetration into various concrete mixes. From the results specimen P_0 shows the highest depth of penetration 10.44 mm at 28 days. However, the different depth compared with the lowest at 28 days is around 8% only. At 90 days, P_{F2} shows the lowest of penetration 13.55 mm but it doesn't show significant result between the values with specimen P_0 of 14.53 mm.

It was observed that the maximum chloride ingression into the control mix specimens P_0 is 14.53 mm. This is inconsistent with previous research (Ahmed Budiea, 2008) which reported that the chloride ingression into P_0 should be more than 20 mm.

Generally, the chloride diffusion for the concrete containing fiber was clearly reduced. This data also proves that by adding fiber in higher percentages and longer fiber strand have good effect on resisting chloride ion ingress. Since no microstructural analysis was carried out for the P_0 sample, a possible explanation for this observation can perhaps be related to the compressive strength of the specimens. Specimens with fiber, P_{F1} and P_{F2} showed higher compressive strength values than P_0, this indicates that the specimen is denser in terms of its microstructure compared to specimen P_0. Hence, it should be more resistance against chloride ingression. Figures 4.15 - 4.16 show the result for all specimens after being sprayed with silver nitrate $AgNO_3$.

Figure 4.15: Measurement of depth of chloride penetration on P_0, P_{F1} and P_{F2}.



(a)

(b)

(c)

Figure 4.16: The penetration of chloride into Specimen (a) control specimens P_0 (b) P_{F1} and (c) P_{F2}

4.5 Microstructural Analysis

A microstructural investigation is important to determine the cause of deterioration and understand the mechanism of deterioration. The analysis investigated the piece of concrete broken from the structure which will represent the bulk concrete either after compressive strength or tensile strength test. The external surfaces of the samples exposed to acid were studied by using the FESEM using Low Electron Scanning.

Grattan-Bellew (1996) investigated the behaviour of Portland cement concretes due to deterioration of aggressive environment. The author suggested that an understanding of the interrelationships of the components of the concrete with the environment is necessary to determine the causes of the deterioration when it occurs.

Figures 4.17 and 4.18 show the micrograph of P_{F1} immersed in water and in hydrochloric acid solution. The micrograph showed rough idea of the different surface behaviour between both regimes curing at 500x magnification. Figure 4.17 shows a very dense structure of cement paste. While, Figure 4.18 shows a porous structure, in strong acid like HCl cement paste dissolves completely. It is found that the acid is extremely aggressive towards the cementitious component in the concrete. This is in agreement with statement stated from Grattan-Belew (1996).

The effect of palm oil fiber in concrete when immersed in water and hydrochloric acid can be observed. Fibers have been deteriorated easily by hydrochloric since it is a natural fiber. The fibers which comprised of hemicelluloses, holocellulose and cellulose (refer to Table 2.3) is susceptible to acid attack whereby the cellulosic material will be hydrolysed by the acid, (Atanu Biswas *et al.*, 2006)

The author revealed that acid attack is limited to the surface layer. This is because the reaction products tend to block the pores and prevent any further penetration of the acid. Figure 4.19 shows the changes in layer of concrete surface in

cross section view after been immersing the acid. In this material, the zone had deteriorarated, increases of the porosity as being dissolution of portlandite by that acid.

According to previous researcher (Randell and Jaubertie., 1999), concrete being attacked by the acid should show the formation of gypsum. But in this case, gypsum needle-like crystal was not found. This might be happen due to different application of acid type in the study compared with this researcher. This researcher was using sulphuric acid to investigate acid attack.

The presence of sodium chloride in the sample can be observed in the micrograph picture of concrete surface (Figures 4.21 - 4.22) and proven in the EDX analysis of the sample as shown in Table 4.3. Figure 4.21 shows the micrograph surface of specimen P_{F1} after being exposed to the sodium chloride at high magnification 2500x. Figure 4.22 shows the surface of the specimen magnified 250x.

Figure 4.23 shows the surface of hydrated cement paste of specimen PF1. Figure 4.24 shows the micrograph of the surface at high magnification (10,000x). calcium silicate hydrate (C-S-H) can be found a lot in the specimen. According to Huzaifa (2008) the hydrated cement paste in control mix is different from in concrete incorporating fiber in terms of the hydration product.

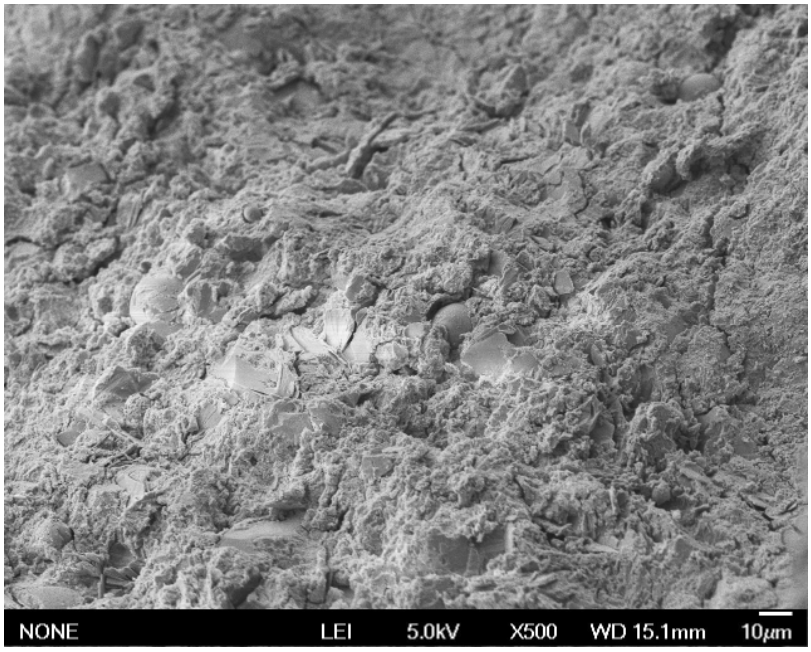


Figure 4.17: P_{F1} in water at 1800 hours

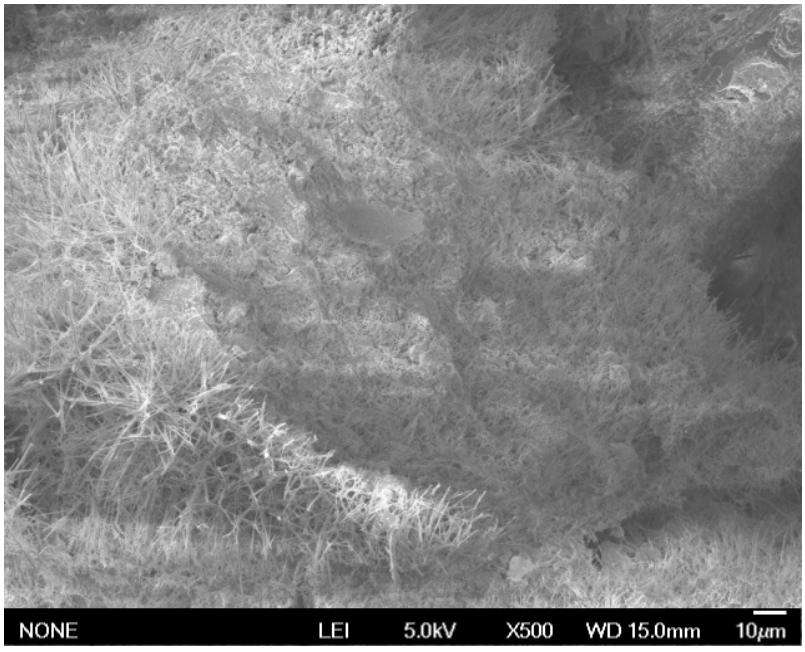


Figure 4.18: P_{F1} in hydrochloric acid at 1800 hours

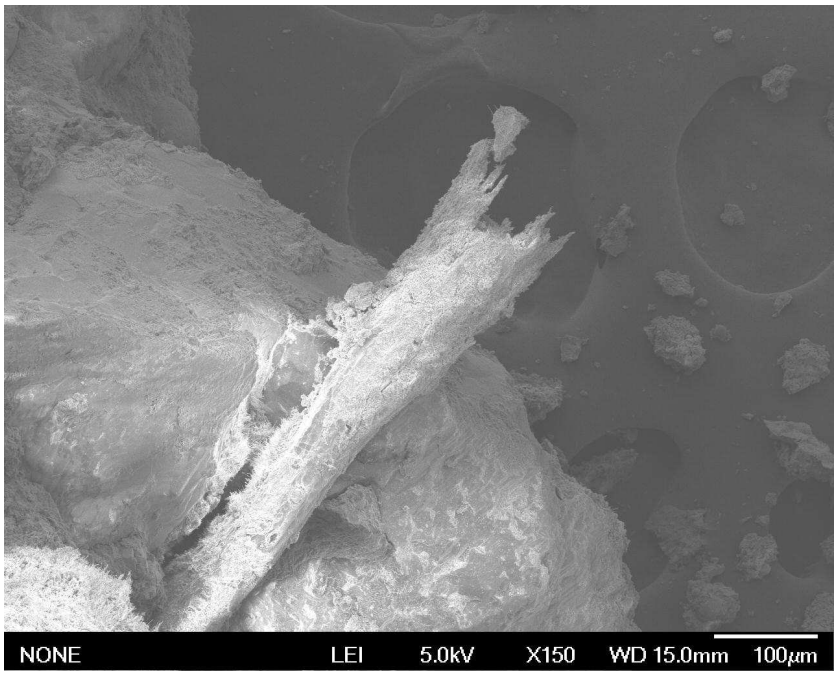


Figure 4.19: Fiber of P_{F1} in hydrochloric acid at 1800 hours

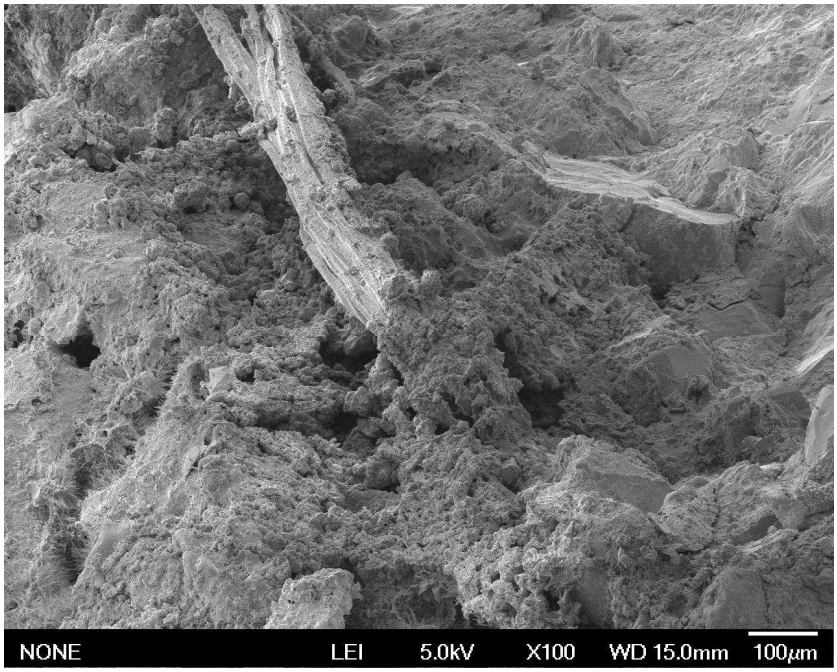


Figure 4.20: Fiber of P_{F1} in water at 1800 hours

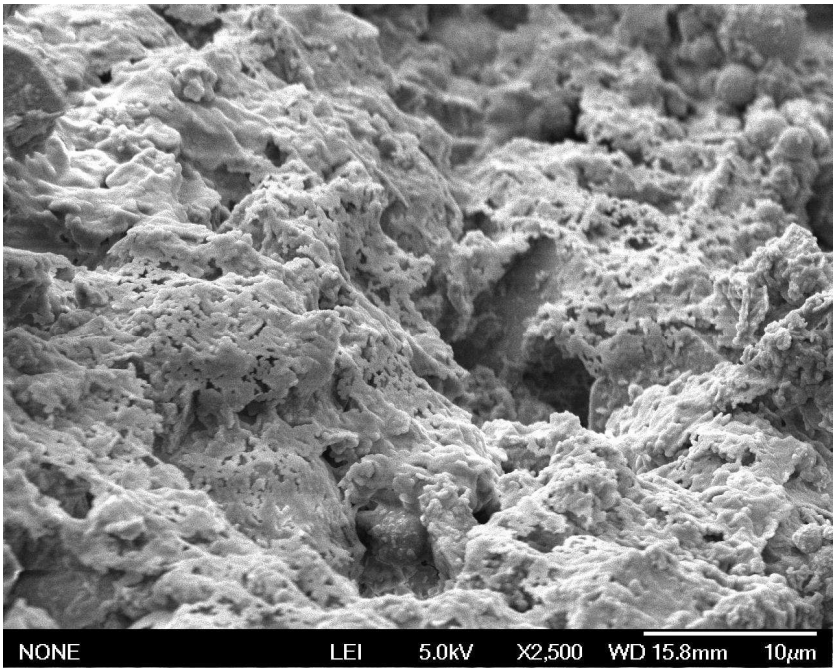


Figure 4.21: The surface of P_{F1} in sodium chloride after 90 days

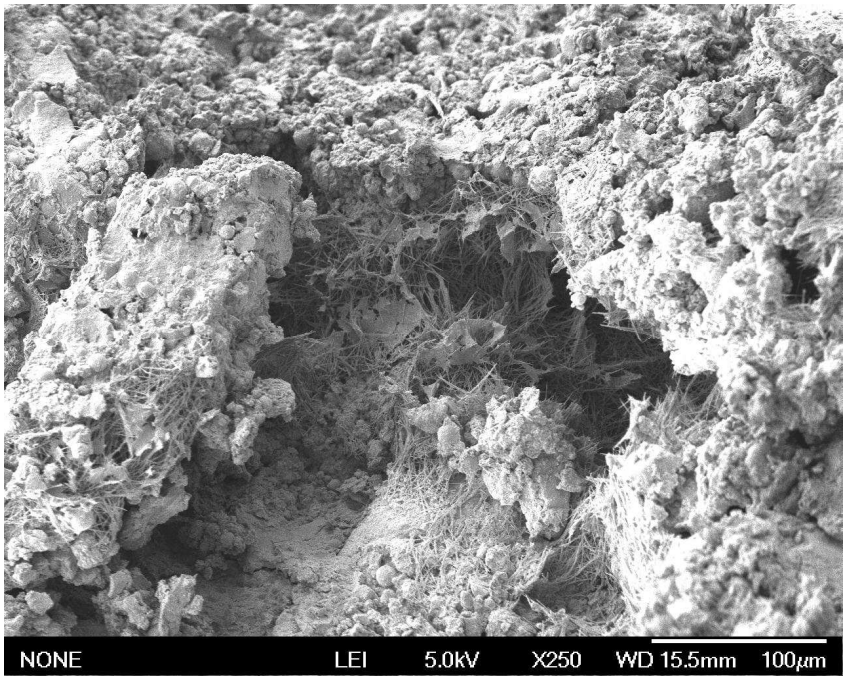


Figure 4.22: The surface of P_{F1} in sodium chloride after 90 days

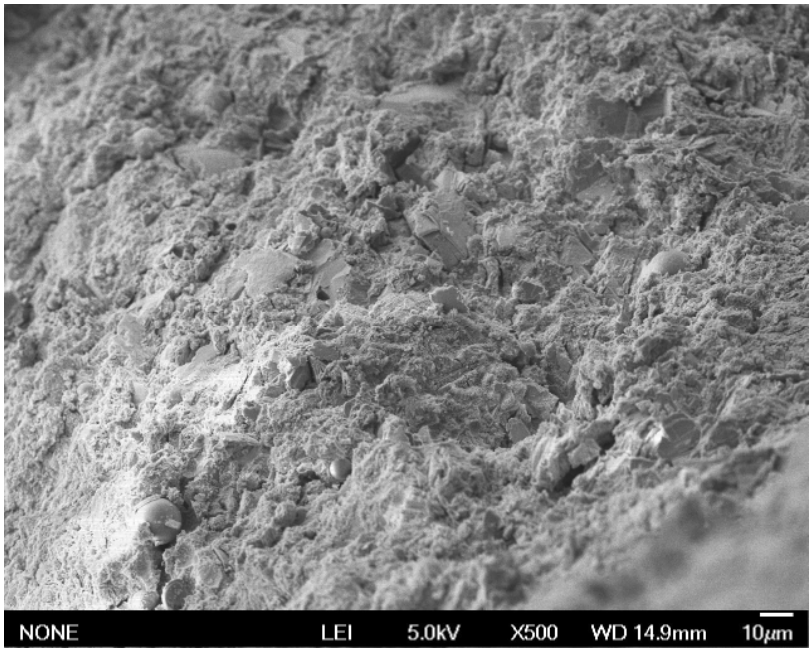


Figure 4.23: The surface of hydrated cement paste P_{F1} (500x)

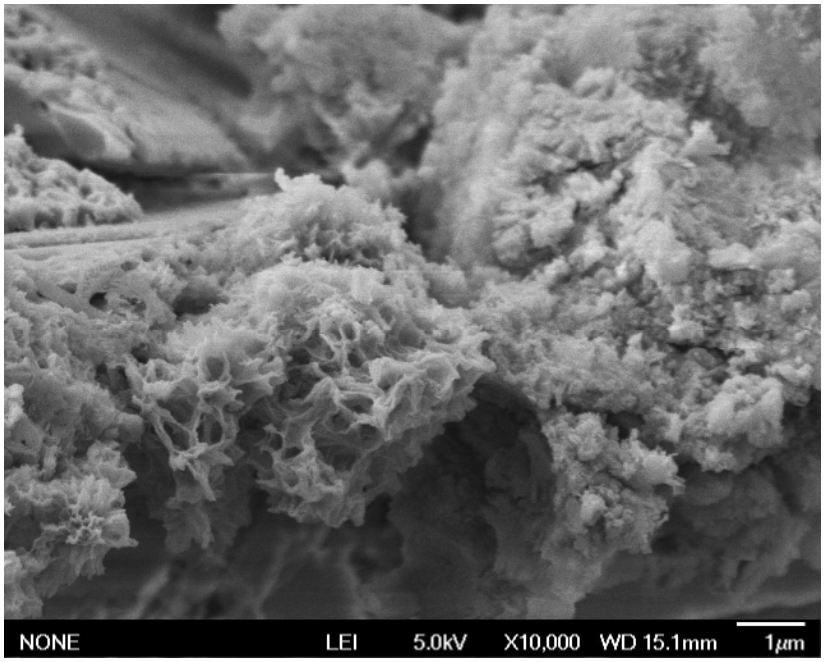


Figure 4.24: The surface of hydrated cement paste P_{F1} (10,000x)

4.6 EDX Analysis

4.6.1 Acid Attack

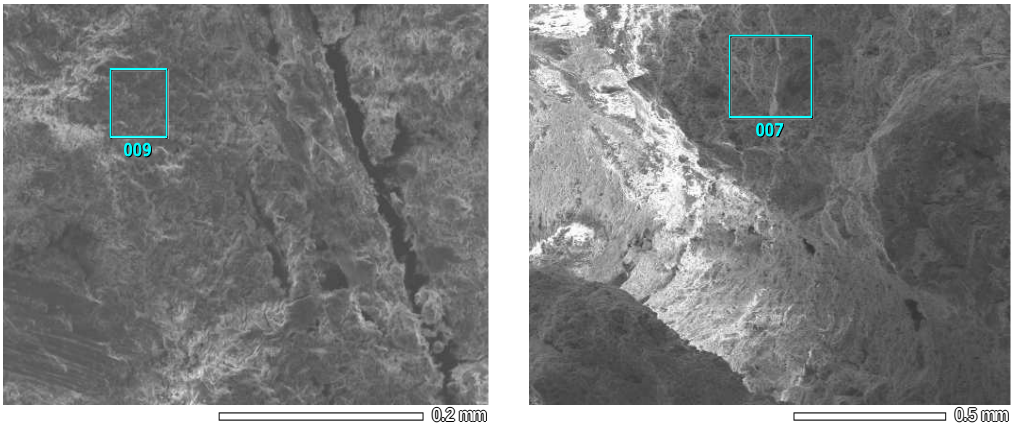


Figure 4.25: P_{F1} at 1800 hours in water cured (left) and hydrochloric acid (right)

Table 4.2: EDX Analysis of Specimen P_{F1} in water and in acid

Element	Mass (%)		
	In Water	In Acid	
		Outside layer	Inner layer
Calcium	20.07	0.23	10.61
Chloride	0.23	1.23	1.40

Figure 4.25 shows the area of concrete which was analysed using EDX Analysis for the specimen immersed in acid, they had been divided by 2 layers of deterioration categories due to colour change. Table 4.2 shows the result of the relative mass percentage of calcium and chloride. However, in the brown layer the relative content of calcium is quite high than the outside layer. In most cases, the percentage of calcium was decreased after being exposed to acid solution. This might be due to the leaching or dissolution of calcium hydroxide (CH) and calcium silicate hydrate (C-S-H) upon exposure to the hydrochloric acid solution. The presence of chloride ion from hydrochloric acid solution is shown by the increase in the relative mass percentage of chloride from EDX analysis.

4.6.2 Chloride Penetration

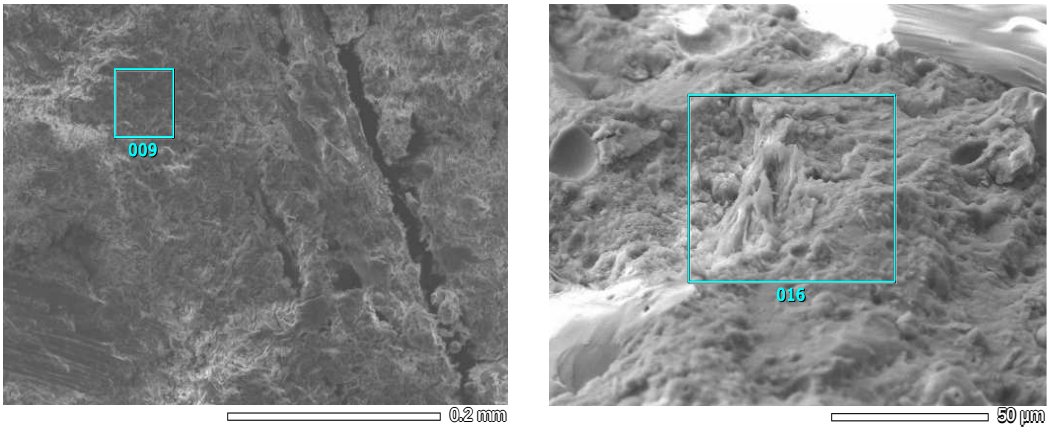


Figure 4.26: P_{F1} at 1800 hours in water cured (left) and sodium chloride (right)

Table 4.3: EDX Analysis of specimen P_{F1}in water and sodium chloride

Element	Mass (%)	
	In water	In sodium chloride
Calcium	19.35	20.13
Chloride	0.23	2.44
Sodium	0	0.53

Figure 4.26 shows the area of concrete were being analysed using EDX analysis. Sodium chloride solution penetrated and crystallized in the specimen as sodium chloride which was identified by EDX analysis. Table 4.3 shows the result of the relative mass percentage of calcium, chloride and sodium. It shows that the percentage of chloride was increased and the presence of sodium after exposure to sodium chloride solution.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

Based on the research conducted, conclusions can be drawn as below:

1. The optimum length and percentages of fiber used in concrete is 5 cm and 0.25% since specimen P_{F2} has achieved the maximum compressive strength for the whole period.

2. From the acid attack test, concrete specimens incorporating 3cm fiber length and 0.5% fiber content (P_{F1}) which is highest in palm oil fiber content show the highest percentage of mass loss and also compressive strength loss.

3. Specimen P_{F2} shows the same behavior with control mix P_0 after being exposed against hydrochloric acid solution.

4. Test results of chloride penetration revealed that palm oil fiber in concrete does not influence the rate and depth of the chloride ion ingress.

5. Generally, palm oil fiber reinforced concrete can be utilized as concrete material as long as not being exposed against severe exposure environment and specimen P_{F2} shows the great potential to be use widely as concrete material.

5.2 Recommendations

Based on the result of this study, the following recommendations would be useful:

1. To do further study about various durability aspects. By compiling all this results, better conclusions can be made.

2. To do further study on the strength and durability aspects of palm oil fiber reinforced concrete incorporating with other replacement and admixtures materials.

3. Before fiber being used in concrete material, the palm oil fiber should be well treated so it will suit with humid environment and any vulnerable characteristics could be lessened.

4. Study alone about the morphology, physical, chemical and mechanical of the palm oil fiber should be conducted to get better understanding about their characteristics.

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APPENDICES

Appendix A1

Compressive Strength test data at 7 days

Mix	Cross Section Area (mm^2)	Maximum Load (kN)	Avg. Maximum load (kN)	Compressive Strength (MPa)
Control Mix, P_0	$1x10^4$	272.6	279.8	27.98
	$1x10^4$	291.2		
	$1x10^4$	281.1		
	$1x10^4$	274.3		
P_{F1}	$1x10^4$	297.5	298.6	29.86
	$1x10^4$	298.6		
	$1x10^4$	292.9		
	$1x10^4$	305.4		
P_{F2}	$1x10^4$	318.4	306.9	30.69
	$1x10^4$	312.1		
	$1x10^4$	301.8		
	$1x10^4$	295.3		

Appendix A2

Compressive Strength test data at 28 days

Mix	Cross Section Area (mm^2)	Maximum Load (kN)	Avg. Maximum load (kN)	Compressive Strength (MPa)
Control Mix, P_0	$1x10^4$	467.6	436.7	43.67
	$1x10^4$	455.1		
	$1x10^4$	431.1		
	$1x10^4$	392.9		
P_{F1}	$1x10^4$	448.2	445.5	44.55
	$1x10^4$	465.3		
	$1x10^4$	394.5		
	$1x10^4$	474.4		
P_{F2}	$1x10^4$	458.3	460.5	46.05
	$1x10^4$	416.1		
	$1x10^4$	523.0		
	$1x10^4$	444.4		

Appendix A3

Compressive Strength test data at 90 days

Mix	**Cross Section Area (mm^2)**	**Maximum Load (kN)**	**Avg. Maximum load (kN)**	**Compressive Strength (MPa)**
Control Mix, P_0	$1x10^4$	537.8	508.0	50.80
	$1x10^4$	385.7		
	$1x10^4$	565.2		
	$1x10^4$	543.4		
P_{F1}	$1x10^4$	483.5	542.5	54.25
	$1x10^4$	569.4		
	$1x10^4$	556.1		
	$1x10^4$	561.1		
P_{F2}	$1x10^4$	607.9	593.9	59.39
	$1x10^4$	591.5		
	$1x10^4$	579.1		
	$1x10^4$	597.2		

Appendix B1

Weight of specimens – Acid Resistance (0 – 800 hours)

Mix	Sample No	Time	0	24	48	72	100	200	300	400	500	600	700	800
P_0	P_{0A1}		2.4110	2.3865	2.3785	2.3730	2.3675	2.3665	2.3635	2.3595	2.3485	2.3425	2.3385	2.3335
	P_{0A2}		2.3940	2.3695	2.3625	2.3565	2.3515	2.3505	2.3480	2.3440	2.3325	2.3265	2.3230	2.3180
	P_{0A3}		2.3785	2.3575	2.3485	2.3425	2.3375	2.3365	2.3340	2.3295	2.3185	2.3130	2.3095	2.3245
	P_{0A4}		2.3985	2.3730	2.3645	2.3585	2.3530	2.3515	2.3490	2.3440	2.3325	2.3270	2.3225	2.3125
P_{F1}	P_{F1A1}		2.3955	2.3770	2.3700	2.3655	2.3615	2.3510	2.3575	2.3345	2.3275	2.3225	2.3155	2.3105
	P_{F1A2}		2.4210	2.4015	2.3930	2.3845	2.3820	2.3690	2.3650	2.3655	2.3485	2.3425	2.3375	2.3310
	P_{F1A3}		2.4225	2.4015	2.3965	2.3935	2.3865	2.3765	2.3695	2.3620	2.3505	2.3450	2.3435	2.3335
	P_{F1A4}		2.3870	2.3645	2.3575	2.3505	2.3475	2.3370	2.3300	2.3290	2.3040	2.2985	2.2975	2.2865
P_{F2}	P_{F2A1}		2.4820	2.4620	2.4545	2.4465	2.4420	2.4395	2.4385	2.4380	2.4285	2.4175	2.4145	2.4075
	P_{F2A2}		2.3895	2.3565	2.3495	2.3430	2.3385	2.3375	2.3350	2.3345	2.3215	2.3165	2.3135	2.3070
	P_{F2A3}		2.3895	2.3715	2.3645	2.3585	2.3540	2.3525	2.3505	2.3505	2.3380	2.3330	2.3305	2.3245
	P_{F2A4}		2.3850	2.3665	2.3585	2.3495	2.3445	2.3420	2.3400	2.3395	2.3265	2.3215	2.3185	2.3125

Appendix B2

Weight of Specimens – Acid Resistance (800-1800 hours)

Mix	Sample No	Time	800	900	1000	1100	1200	1300	1400	1500	1600	1700	1800
P_0	P_{0A1}		2.3335	2.3310	2.3310	2.3295	2.3295	2.3285	2.3230	2.3170	2.3125	2.2945	2.2915
	P_{0A2}		2.3180	2.3165	2.3165	2.3145	2.3145	2.3130	2.3085	2.3025	2.2975	2.2980	2.2965
	P_{0A3}		2.3245	2.3030	2.3030	2.3020	2.3020	2.3005	2.2945	2.2895	2.2840	2.2830	2.2810
	P_{0A4}		2.3125	2.3155	2.3155	2.3145	2.3145	2.3125	2.3070	2.3015	2.2965	2.2960	2.2945
P_{F1}	P_{F1A1}		2.3105	2.3060	2.3055	2.3045	2.3025	2.2990	2.2940	2.2930	2.2900	2.2875	2.2855
	P_{F1A2}		2.3310	2.3260	2.3255	2.3245	2.3220	2.3180	2.3130	2.3095	2.3090	2.3085	2.3065
	P_{F1A3}		2.3335	2.3280	2.3265	2.3225	2.3215	2.3190	2.3145	2.3135	2.3125	2.3115	2.3100
	P_{F1A4}		2.2865	2.2820	2.2805	2.2800	2.2755	2.2740	2.2680	2.2635	2.2625	2.2615	2.2595
P_{F2}	P_{F2A1}		2.4075	2.4045	2.4045	2.4030	2.4010	2.3955	2.3905	2.3845	2.3825	2.3780	2.3755
	P_{F2A2}		2.3070	2.3045	2.3045	2.3035	2.3020	2.2965	2.2910	2.2850	2.2795	2.2770	2.2765
	P_{F2A3}		2.3245	2.3215	2.3215	2.3210	2.3190	2.3140	2.3100	2.3045	2.3015	2.3000	2.2980
	P_{F2A4}		2.3125	2.3095	2.3095	2.3095	2.3070	2.3025	2.2980	2.2915	2.2895	2.2875	2.2855

Appendix C1

Compressive Strength test data at 1800 hours in water

Mix	**Cross Section Area (mm^2)**	**Maximum Load (kN)**	**Avg. Maximum load (kN)**	**Compressive Strength (MPa)**
Control Mix, P_0	$1x10^4$	543.9	529.1	52.91
	$1x10^4$	483.3		
	$1x10^4$	580.0		
	$1x10^4$	509.1		
P_{F1}	$1x10^4$	617.6	589.5	589.5
	$1x10^4$	579.5		
	$1x10^4$	593.2		
	$1x10^4$	567.7		
P_{F2}	$1x10^4$	634.5	602.6	60.26
	$1x10^4$	598.3		
	$1x10^4$	578.5		
	$1x10^4$	599.1		

Appendix C2

Compressive Strength test data at 1800 hours in 1% hydrochloric acid solution

Mix	**Cross Section Area (mm^2)**	**Maximum Load (kN)**	**Avg. Maximum load (kN)**	**Compressive Strength (MPa)**
Control Mix, P_0	$1x10^4$	254.2	263.9	26.39
	$1x10^4$	283.5		
	$1x10^4$	235.4		
	$1x10^4$	282.3		
P_{F1}	$1x10^4$	199.2	200.0	20.00
	$1x10^4$	199.2		
	$1x10^4$	213.0		
	$1x10^4$	188.7		
P_{F2}	$1x10^4$	245.4	253.5	25.35
	$1x10^4$	246.1		
	$1x10^4$	263.6		
	$1x10^4$	258.9		

Appendix D

Mix	7 days			28 days			90 days		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
P_0	6.32	6.42	7.16	10.26	11.63	9.56	14.56	15.54	15.28
	5.44	4.12	5.56	11.12	10.68	9.54	15.22	14.64	13.22
	5.32	7.66	6.54	12.78	7.66	10.56	16.68	15.12	14.52
	5.68	6.36	4.48	11.24	11.64	9.64	12.78	14.68	15.26
	6.78	8.12	5.42	10.76	11.60	10.18	15.66	15.22	13.12
	5.02	7.62	4.52	10.62	10.02	8.44	14.62	10.12	15.3
	6.03			10.44			14.53		
P_{F1}	4.86	6.42	5.42	9.68	11.46	13.52	15.68	12.13	14.96
	5.12	5.3	4.42	8.66	10.44	10.24	12.66	15.12	14.74
	4.6	5.44	6.12	6.78	8.56	12.68	16.63	12.68	13.76
	5.88	5.48	5.32	12.86	8.42	10.56	13.76	12	16.76
	5.78	3.98	6.56	9.56	9.56	9.68	15.72	14.98	16.54
	5.9	4.66	4.69	10.44	10.34	10.52	15.74	15.74	15.18
	5.33			10.22			14.71		
P_{F2}	5.56	3.52	5.42	10.54	10.18	7.3	15.22	13.72	14.32
	5.52	3.42	5.52	10.66	12.24	10.54	12.74	15.56	15.54
	4.5	5.44	3.86	5.22	11.38	9.14	14.12	12.46	15.9
	3.24	5.46	4.02	10.82	10.54	9.68	13.02	11.1	13.18
	7.02	4.32	6.18	8.64	10.12	9.38	11.62	14.32	11.74
	4.28	5.64	8.16	10.12	9.22	8.34	13.42	13.54	12.38
	5.06			9.67			13.55		