INFLUENCE OF SORGHUM HUSK ASH AS MICRO-FILLER IN POLYMER CONCRETE

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ABSTRACT

Polymer concretes (PCs) are known to be less permeable to water, but its usage is less popular in tropical countries like Nigeria because its common binder, thermoset resins, are very sensitive to rising temperature. This sensitivity to temperature accelerates the polymerization process and this jeopardizes its early strength development, thereby producing a PC with low workability and high porosity. To address this, polymer inhibitors with addition of Methyl Methacrylate (MMA) was introduced. Firstly characterization work on binder formulation was carried out by introducing Methyl Ethyl Ketone Peroxide (MEKP) and cobalt Naphthenate (CoNp) into the polyester resin as accelerators. Properties like density, specific gravity, particle size distribution, surface area, morphology and chemical composition were carried out on fillers. Sorghum Husk Ash (SHA) and calcium carbonate ($CaCO_3$) were added as fillers. PC with optimum mix proportion at low binder (11%) and different filler contents (12, 14/ & 16%) were investigated under compressive test. Two types of PC (PC-SHA and PC-$CaCO_3$) with polyester binder were produced and its physical and mechanical properties were tested. PC-SHA gave highest compressive strength ($56.6N/mn^2$) at 14% filler contents and least water absorption of 0.26% at 16% filler contents after 28 days as compared to compressive strength value of PC-$CaCO_3$ ($49.8N/mm^2$) and water absorption of 3.1% at 16% filler contents after 28 days. Conclusively, SHA is seen to be highly promising filler for PC after being modified by grinding.

CHAPTER ONE

1.0 INTRODUCTION

1.1 Background to the Study

Generally, concrete-polymer composite is a concrete that contains polymers. Development of concrete-polymer composites such as polymer-modified concrete, polymer concrete and polymer-impregnated concrete aim to produce high-performing and versatile construction materials.

Polymer-modified concretes are composite materials comprising two solid phases - the aggregates that are intermittently dispersed via the materials and the binders in which itself contains a cementitious phase and a polymer phase (Gemert, 2007). The polymer introduced acts as polymeric admixtures/modifiers in normal concrete (Khudhair *et al.*, 2018). There are economically available polymeric admixtures/modifiers in Japan categorized into polymer dispersions, redispersible polymer powder, water-soluble polymers, and liquid polymers (Ohama, 2007). On the other hand, the term 'polymer-impregnated concrete' coined by Bhutta *et al.* (2013) refers to concrete produced by impregnating or infiltrating the hardened conventional concrete with a liquid monomer and afterwards completing the polymerization of the monomer in-situ. The area of application of polymer-impregnated concrete has been identified in precast products, though in less applications, majorly for enhancing the waterproofing ability and durability of concrete structure (Ohama, 2007).

The only non-cementitious concrete-polymer composite is polymer concrete. Polymer Concrete (PC) is manufactured by polymerizing dry aggregate and monomers (binder) after the addition of additive, catalyst or accelerator. The fresh

PC are then cured completely devoid of water and cement binder (Ohama, 2007). Its constituents depend on its designed application. PC has finite possible applications in the construction industry. It is useful in manufacturing box culverts, hazardous waste containers, trench lines, floor drains, and for the repair and overlaying of damaged concrete surface (bridge and pavements) (Bedi *et al*., 2013). However, some improvement and modification has to be made to improve the properties of PC. Due to this reason, many researchers have conducted several studies to develop materials that can be incorporated into PC, majorly by introducing fillers into the PC mixture (Barbuta *et al*., 2010).

In Japan, the concrete-polymer composites are applicable as sustainable construction materials and have been continually enhanced since the early 1920s (Ohama, 1997). Its application in the Japan construction industry is common, popular and dominant during the 1950s to 1970s (Ohama, 1997; Shaw, 1985). Other than Japan, the United States, United Kingdom, Russia and Germany have also published their standardization works of concrete-polymer composites with, occasionally, clear differences in the content (Khalid *et al.*, 2015). In Nigeria, the use of concrete-polymer composites is still not popular. This also applies to research on PC because polymers are very reactive to hot temperature and Nigeria is a tropical country. Therefore, the lack of technical know-how among local fabricators and material scientists has become a limitation for the production of PC.

Polymer concrete is produced from polymer resin-which acts as the only concrete binder, dry inert granular aggregates, and filler. Since it does not contain cementitious materials and water, its hardening follows the polymerization process when additives, catalysts, or accelerators are added. PC containing fillers has better mechanical properties due to its effective dispersal in the mixture and its ability to induce dense concrete mixture packing. However, the type of filling materials (natural, granulated, or synthetic) also plays a pivotal role in affecting the composite characteristics (Ates & Barnes, 2012).

In this study, polyester resin was employed as binder. Since this polymer binder is temperature sensitive, polymer inhibitor additive was applied into the binder formulation to initiate the designed modification. It was expected that this would give rise to lower, yet, sufficient binder concentration for PC. This study utilized low cost thermosetting polyester resin as binder. Also, this study used Sorghum Husk Ash (SHA) from agricultural waste as filler and added into concrete mix with low binder concentration. SHA was selected as the filler in this research work, since it is available and a bye product of agricultural waste in Nigeria. Sorghum husk, an agricultural waste from milling of guinea corn, is mostly disposed as agricultural waste in landfills. Its reutilization has the potential to generate sustainable and productive materials.

The Performance of PC incorporated with SHA filler was evaluated from an engineering viewpoint. Presently, there are no published findings and data for such PC incorporating agricultural waste in Nigeria. This study has conducted extensive experiments to develop and encourage innovative usage of such sustainable and intelligent material in the Nigeria construction industry.

1.2 Statement of the Research Problem

Polymer Concretes are regarded as less permeable concrete with improved strength (Sung *et al*., 1997). Its usage is usually employed in liquid containing structures (Mani *et al.* 1987). In PC, thermoset polymer resin is used as the binder. Thermoset resins that are economically available include epoxy, vinyl-ester, and unsaturated polyester resin. These are typical resins employed in the construction industry because of their higher strength and stiffness which makes them better than thermoplastic polymers. However, epoxy and vinyl-ester resins are costlier than polyester resins (Yang & Lee, 2001). Hence, Khalid et al., (2015) mentioned that, most researchers frequently choose unsaturated polyester resin, despite the fact that it is very sensitive towards temperature. Generally, high temperature can speed up the polymerization process, resulting in PC which fails to achieve its early strength and causing other problems such as poor workability and honey comb. Therefore, polymer modification should be considered to solve the aforementioned problem by prolonging the working life and giving sufficient time for PC production in ambient temperature.

Agricultural waste like sorghum husk, disposed on open fields can adversely impact our environment through pollution (Elbasiouny *et al*., 2020). Therefore, it is necessary that a study is carried out to suggest an alternative solution to this problem. This study is therefore conducted to re-utilize sorghum husk to serve as fillers in PC. Agricultural waste ashes can become a cost-effective material since mineral-filled PCs are costlier. However, not all agricultural waste ashes have the potential to become PC filler. The wrong selection of filler material may lead to worsened PC quality, also it affects the PC's workability and process ability (Bignozzi *et al.,* 2000).

On the other hand, air voids entrained or entrapped in hardened PC during the mixing and placing of fresh PC can significantly influence the permeability of the hardened PC. These air voids can be easily identified as visible pores on the hardened concrete. An increasing number of pores can reduce the strength of the PC (Rashid & Mansur, 2009), but the development of air voids can be efficiently reduced by using suitable micro-filler (Khalid et al., 2019). However, some modifications on the raw materials including the filler are essential to enhance their properties and improve their engineering behaviours.

1.3 Aim and Objectives of the Study

The aim of this research is to evaluate the influence of sorghum husk ash, an agricultural waste as micro-filler in polymer concrete with a view to reducing visible pores in hardened PC to enhance its strength and permeability.

The objectives of the study are listed as follows, to:

i. Formulate binder using polymer
ii. Characterize fillers under physical and chemical examination
iii. Determine the physical properties of PC containing SHA as micro-filler.
iv. Evaluate the compressive strength property of PC containing sorghum husk as micro filler.

1.4 Scope of Study

The scope of this study was to accomplish the objectives stated above and concentrate predominantly on experimental works. The testing method and work procedures were specified according to the Japanese international standard (JIS), Eurocode standard (BS-EN), American society for testing and materials (ASTM) and other recommended test procedures proposed by previous researchers.

All cement hydrate binders of conservative mortar or concrete were replaced in this study with polymer binders to produce PC. Hence, PC concrete are concrete without cement and water but made of resin binder only. The major component of PC used as polymer binder in this research is the thermosetting polyester resin. The chemical mixed with the resin was limited to 0. 5% Cobalt Naphthenate (CoNp) and 1% Methyl Ethyl Ketone Peroxide (MEKP) as recommended in earlier research findings in the literature

(Rebeiz et al., 1992; Gorniski et al., 2004 & Gorninski et al., 2007) to fabricate the proposed binder design for this research. Though, following the work of Li and Lee (2002) varying percentages of inhibitor additive of Methyl Methacrylate (MMA) were added to the resin mix to have sufficient working time in producing the PC. To get consistent outcomes, the PC specimens were cast in room temperature with the relative humidity around 66 ± 2%. After that, all specimens were post-cured. Control specimens had been produced where no filler was incorporated.

SHA was utilized as the filler in PC and it was used to substitute Calcium Carbonate in conventional filled-PC. SHA filler was grinded and physically modified to obtain finer particles. Inert granular materials such as coarse and fine aggregates were utilized as well and this is similar to conventional concrete. After that, all specimens were post-cured in sunny environment. The characteristics of SHA and Calcium Carbonate fillers were examined to determine its performance from an engineering perspective and the assessment of PC on the engineering properties with optimum mix design and mix proportion were done because it is an important factor in developing the potentially valuable construction materials.

1.5 Justification For the Research

SHA can be found in abundance as agricultural waste that is often uncontrollably dumped in landfills and burnt in most environment causing air pollution which is hazardous to human health and affecting the ozone layer leading to global warming. Recycling SHA as a potential filler in PC helps in turning waste to wealth and contributing to a sustainable and cleaner environment. Nevertheless, not all waste ashes have the potential to become PC filler. The wrong selection of filler material may lead to worse PC quality. Not only that, it affects the PC's workability and process ability (Bignozzi *et al*., 2000). Most natural waste source from agricultural plant is cellulose (Raveendran *et al.*, 1996; Kaddami *et al*., 2006) which has a structure that attracts liquid into PC. This can lead to excessive resin consumption, which is not cost effective, and thus jeopardise the production of PC even when filler is used. Until today, no study has so far been done on the incorporation of SHA as PC filler because of the aforesaid potential setbacks.

This research is to give an insight into the potential incorporation of SHA as micro-filler into PC, gauged through microstructure and strength examination.

The significant findings of this research will inspire the production of PC in regions under tropical temperature climate and promote its usage amongst Nigeria fabricators of building and civil engineering elements. A notable novelty database of concrete polymer composites and its application in the construction industry will be provided. The outcomes of this work will be beneficial to researchers and engineers working in the field of cellulose filler in PC. Fabricators and engineers will be updated in improving the quality of materials and providing an established database for design works in the future. Value-added products from local resources will be developed to promote green materials in the construction industry via this composite production. This research will facilitate the introduction of materials with proven performance to contractors and provide-significant market value where the final product can be commercialized.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 Polymer Concrete

Polymer Concrete (PC) is produced solely from polymer resin that acts as the only concrete binder, dry inert granular aggregates and filler. PC is sometimes mistaken for geopolymer concrete, but it should be pointed out that PC is made by replacing all cement hydrate binder of conventional concrete with polymer binder. The hardening of PC occurs without any water, but it follows the polymerization process when additives such as catalysts or accelerators are added.

2.1.1 Polymer binder

The major component material commonly used in PC is thermosetting resin, which is a satisfactory material itself for structural applications. Thermosetting resins such as unsaturated polyester, acrylic and epoxy resins are preferred over thermoplastic resins though its selection is still dependent on specific applications. Thermosetting resins initiate changes to molecular structure and, sometimes, cause individual molecular chains to link up in an irregular fashion to form a solid infusible network. This is called ‘curing’ or ‘cross-linking’ and can be activated by special chemicals, heat or irradiation. Most processes of thermosetting resin are crosslinked and undergo an irreversible liquid to solid transition; this is unlike thermoplastics resin which undergoes a reversible liquid to solid transition (Jackson *et al.,* 2003).

Polyester resin is commonly used in the composites production industry and is the cheapest among thermosetting resins. Also, the resin can be easily handled, pigmented, filled, and fibre-reinforced in liquid form (Yang & Lee, 2001; Kueh, 2014). Polyester resins are the product of polycondensation reactions of dicarboxylic acids with dihydroxy alcohols (Carraher, 2007). Polyester polymer chain is shown in Figure 2.1. Using different reactants, Isophthalic and Orthophthalic polyester can be produced. Isophthalic polyester resin is produced from Isophthalic acid while Orthophthalic polyester resin is produced from phthalic acid, and thus both resins are different diacids (Carraher, 2007).

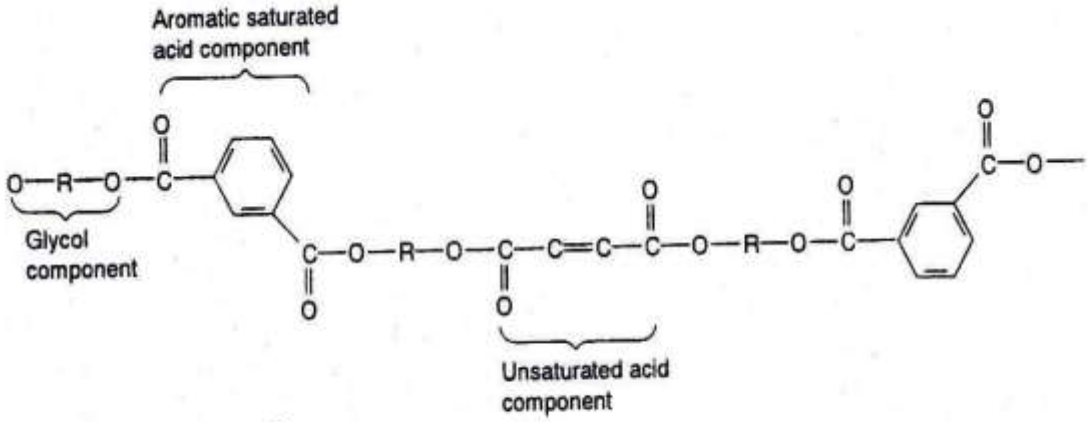


Figure 2.1: Polyester Polymer Chain

Source: Carraher (2007)

In terms of cost, Orthophthalic polyester resin is cheaper than Isophthalic polyester resin and has been previously used as traditional polyester resin (Soraru & Tassone, 2004). Nevertheless, Isophthalic polyester resin is a common binder for PC due to its excellent mechanical properties as shown in Table 2.1. The reason is that Isophthalic polyester resin has higher molecular weight chain, as supported by Hollaway (1994) and Gorninski *et al.,* (2007). Even though both polyester resins have similar density, their viscosity is different where the viscosity of Isophthalic polyester resin is higher (Table 2.1). The viscosity is an important parameter because it directly affects the workability of the PC especially when the binder content is low.

Table 2.1: Properties of Isophthalic and Orthophthalic Polyester Resin

Properties	**Isophthalic polyester**	**Orthophthalic polyester**
Viscosity (Brookfields) 25^{o}C, 60 rpm (cps)	400-650	250-350
Tensile modulus (MPa)	3200 min	2400 min
Flexural strength modulus (MPa)	3500 min	3800 min
Density (g/cm^{3})	1.1	1.1
Compressive strength (MPa)	117.3	91.4
Flexural strength (MPa)	29.5	17.2

Source: (Gorninski *et al.,* 2007)

The optimum binder content used by most researchers is 12% to obtain high strength PC (San-José *et al.,* 2005; Gorninski *et al.,*2007; Mahdi *et al.,* 2009). Hwang *et al.,* (2009) tested the binder content of 9% to 12% for unsaturated polyester resin and concluded that, when the binder content was 12%, the pore size of PC particles shrunk and the number of these small pores increased in number due to the decomposition or thermal degradation of polymer binder. An increase in binder content therefore decreases the porosity of PC (Hwang *et al.,* 2009; Haidar *et al.,* 2011) and indirectly reduces the water absorption ability of PC (Haidar *et al.,* 2011).

2.1.2 Polymer additive

Polymer additives are normally used in small quantities and are added to polymeric formulations to enhance the value of materials by improving their processing performance and appearance during manufacture and in use. Sufficient hardening process of polymer resin can be achieved after adding polymer additive into polymer formulation. The hardness can be measured and verified through scientific means to assess the relative degree of curing for most polymer composites materials (Varughese & Chaturvedi, 1996; Ateş & Barnes, 2012; Ilday *et al.*, 2013; Erickson *et al.*, 2014). Multiple studies have shown that, hardness can be measured either during resin development or using the final form of polymeric materials.

2.1.2.1 Curing agent (catalysts)

Peroxides are used as curing agent (catalysts) for unsaturated polyester resins, generating free radicals and causing cross-linking and acting at either elevated or ambient temperature. Development of peroxide and peroxy ester systems for room temperature curing of thermosets is a priority. Polyester resins are usually cured by adding chemicals for several purposes as follows:

i. To be decomposed to free radicals
ii. To offer a simple controlling technique
iii. To provide rate and length of cure.

Polyester resins can also be cured by heat or irradiation and newest technology proclaimed curing under the effect of ultraviolet (Murphy, 2001). Cross-linking can be positive or negative depending on the extent and the intended result. Chemical cross linking generally renders the insolubility of the material. At this point, it often enhances the strength of the cross-linked materials but decreases flexibility and increases the brittleness. The mixture is deemed as “gelled” at this stage where it becomes elastic and begins to feel like a rubber. Additionally, most chemical cross linking is not easily reversible (Carraher, 2007).

Methyl Ethyl Ketone Peroxide (MEKP) is widely and commonly used as a curing agent for unsaturated polymer resin to mold products. Chemical structure of MEKP and cross-linking process of polyester resin by peroxide curing agent are presented in Figures 2.2 and 2.3, respectively. Plate I shows that cross-link chains had formed in the hardened polyester resin after using MEKP and this image is captured under scanning electron microscopy (SEM) image.

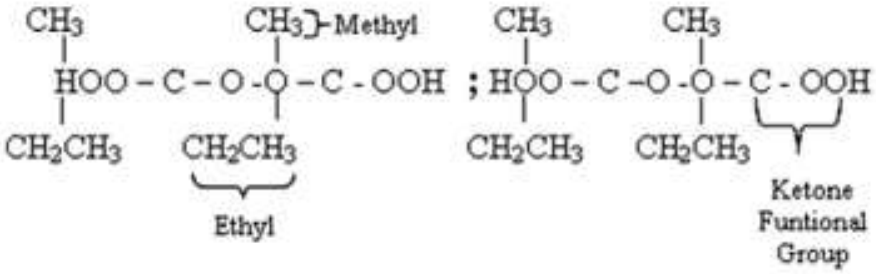


Figure 2.2: Chemical Structure of MEKP
Source: Lim *et al.*, 2009.

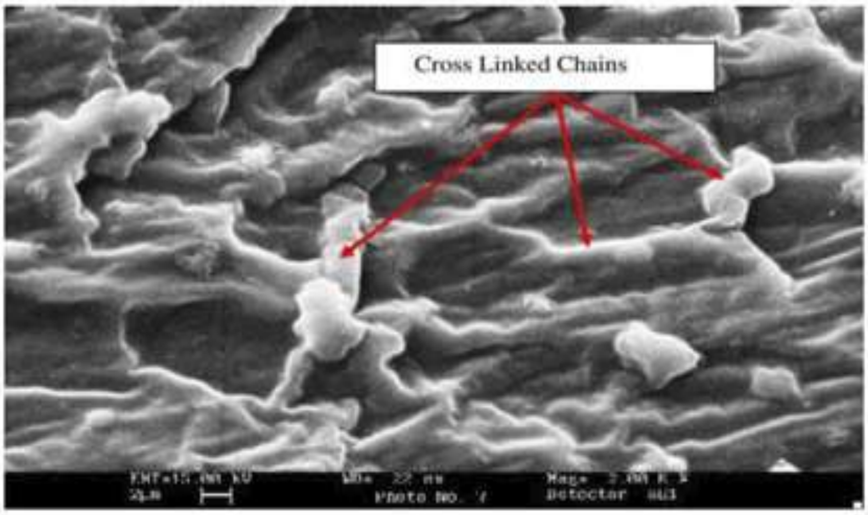


Plate I: Cross linking process of polyester resin by peroxide curing agent
Source: Lim *et al.*, 2009

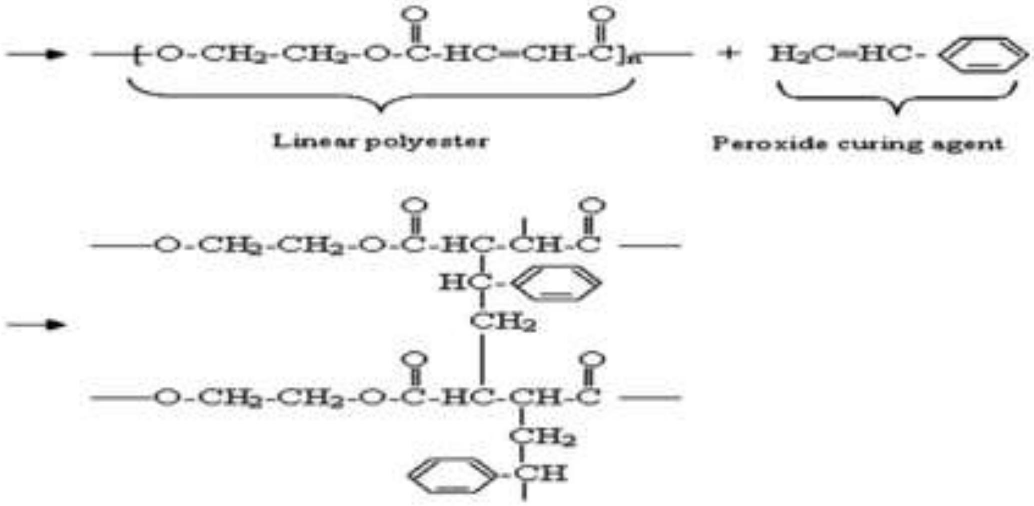


Figure 2.3: SEM of hardened polyester resin using MEKP
Source: Mahdi *et al.*, 2009.

2.1.2.2 Curing agent (accelerator)

The most popular method in curing below 100°C is by using accelerators or pre-accelerated resins and post curing is normally required. Ketone peroxide (either methyl ethyl ketone, cyclo hexanone, or acetyl acetone) with cobalt naphthenate (CoNp) or octoate (CoOc) are some combinations for such normal curing system (Carraher, 2007) with the former being the more popular combination at room and elevated temperature as well as the fastest for PC. The best formulation has been designed by Rebeiz *et al.,* (1992), Gorninski *et al.,* (2004) and Gorninski *et al.,* (2007) for making PC by adding 1% of MEKP and 0.5% of CoNp into the polymer binder formulation.

2.1.2.3 Inhibitors

Inhibitors, also referred to as retarders are polymer additives which can prevent undue polymerization of the resin. Typical inhibitors such as monohydric or polyhydric and some quinines are usually added into polymer formulation during manufacture of the resin to ensure stability in storage. Inhibitors that can further prolong the working life of the resin system which already contain peroxide and cobalt system include methyl methacrylate monomer (Li & Lee, 2002). Methyl Methacrylate (MMA) is a vinyl monomer for polyester resin which can reduce the resin’s viscosity (Fink, 2005).

2.1.3 Aggregates and filler

Thermosetting and thermoplastic materials are hydrophobic materials, therefore, presence of water and high moisture content in aggregates and filler has to be avoided especially during production of PC to enhance the compatibility and interaction between materials.

2.1.3.1 Aggregates

Crushed coarse aggregates and fine aggregates are used as the inert granular material for production of PC. Both aggregates have to be oven-dried at 100 ± 5^{o}C and the moisture content of the inert granular material has to be limited below 0.5% (Rebeiz, 1996; Mahdi *et al.,* 2007; Jo *et al.,* 2008; Mun & Choi, 2008). Aggregates must have low moisture content especially when incorporated in polymer concrete. As known, Polymer is hydrophobic materials, therefore the moisture and water should be minimized as possible and water is prohibited during production of polymer concrete to enhance the bonding between aggregates and polymer. To obtain high strength PC, smaller single size coarse aggregates (between 10 and 12 mm) can be used since Rashid & Mansur (2009) opined that the size of aggregates is not the governing factor.

2.1.3.2 Filler

Filler has been used in polymeric materials since its inception and their basic function is to ‘fill’ a composite. A good mixture is thus one which has homogeneous mix of filler and polymer. Generally, PC with filler has better mechanical properties due to its effective dispersal in mixture and ability to induce denser concrete mixture packing, though the type of filling materials (natural or synthetic) also plays a pivotal role in affecting the composite’s characteristics (Ateş & Barnes, 2012). The packing behaviour of particle in a polymer matrix determines the loading particle in polymer matrix. It becomes important and a critical factor in the understanding and design of any polymer composites, especially when dealing with highly filled system. Conventionally, particulate filler is used in polymeric materials, but the packing in dry state has been compromised due to irregular particle size and particle agglomeration at certain times. Due to this limitation, powder filler became an attractive alternative since it often packs considerably better especially when it is wetted by suitable liquid. The general approach to obtain high packing fraction powders is by using smaller particles to fit into the gaps between large particles and pores. Figure 2.5 gives a simple depiction of this and the approach is known as multimodal packing, which is a phenomenon continuously being studied to identify the optimum particle ratios and quantities of the fraction powders obtained. Thus, in PC production, the intention of using filler is to eliminate or minimize the gaps between materials to produce a denser structure.

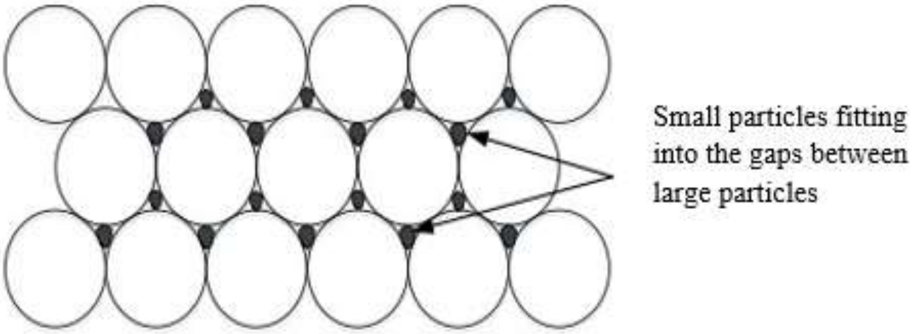


Figure 2.4: Classical bimodal packing effect
Source: Roger and Hancock, (2003)

The functions of using filler are to reduce cost since most fillers are relatively cheaper than polymer or to confer to property modification (Hristova *et al.,* 2002; Kandelbauer *et al.,* 2014). Functions of filler in polymer matrix are given as follows (Kandelbauer *et al.,* 2014):

i. To increase viscosity of liquid resin,
ii. To decrease curing shrinkage,
iii. To decreases peak exotherm on curing since filler acts as a heat sink,
iv. To enhance specific gravity of resin-filler mixture than unfilled resin if filler has higher density than resin is used, vice versa,
v. To increase cured hardness of resin-filler mixture,
vi. To decrease tensile elongation,
vii. To affect curing behaviour; gel time maybe accelerated or retarded.

Fillers normally come directly from the natural origin (mineral filler) or are synthetically produced and Table 2.2 shows some common filler in polymeric system. However, the depletion of natural resources has become a concern, and so, many researchers have shifted to synthetic fillers, though this choice is more expensive.

Table 2.2: Common Filler Used in Polymeric System

Type of filler from different origin	
Natural (mineral filler)	**Synthetic filler**
Natural calcium carbonate	Carbon black
Talc	Silica Fume
Clay	Hydroxides and basic carbonates
Alumina trihydrate	Perciptated calcium carbonate
Magnesium hydroxide	
Marble	
Quartz	
Wollastonic	
Mica	
Silica	
Stone dust	

Source: (Roger & Hancock, 2003; Sung *et al.*, 1997)

With consideration on both environmental issue and sustainable development, utilization and modification of waste materials has become an interesting topic of research in modern production of polymeric materials. This had been initiated in the 1980s when fly ash was first studied and then further explored in the 1990s. To date, the effectiveness of fly ash in enhancing the performance and durability of PC is proven, and it is often chosen since it is inexpensive, non-toxic, poses no health threat, has satisfactory original fineness with low thermal coefficient, is readily available and is compatible with other materials in resin (Atzeni *et al.,*1990; Gorninski *et al.,* 2007).

Fine tailings and rubber powder are some other potential waste filler in PC (Bignozzi *et al.,* 2000; Mun *et al.,* 2007). Aside from waste filler sourced from industries, waste filler from agricultural resources have also been studied (Hoseinzadeh et al., 2013). However, with adequate modifications, these fillers can be used in normal concrete (Shafigh *et al*., 2014; Hamada *et al*., 2019 and Maraveas, 2020).

Ndububa and Nuruden (2015) have been modifying SHA by grinding it to become finer and incorporating it in normal concrete. Similar approach has been reported by Alkamu (2017). Both ground materials were from agricultural resources and had proven their potential as a new variety of pozolanic material as well as a good supplementary cement material in concrete. Additionally, it has shown capability in controlling heat of hydration and improving mechanical properties of the concrete. The size of filler before and after modification is measured from particle size distribution using some techniques of measurement including sieving, sedimentation, optical scattering, and diffraction from particulate suspension. In this case, the finest filler after modification is the most idealistic (Jaya *et al.,* 2012; Awal & Shehu, 2013). As previously mentioned, filler surface plays a vital role in determining the process's behaviour and properties of polymer composites. Common surface treatment of filler uses coupling agent (bi-functional additive) to form very strong covalent bonds between filler and polymer through a variety of mechanism. The main reasons for incorporating coupling agents are (Rothon, 2003):

i. To improve filler production,
ii. To protect filler in storage and in the end of application,
iii. To improve processing,
iv. To reduce absorption of expensive additive,
v. To improve composites properties.

Other than type and size of filler, the shape of filler also affects important properties of polymer composites because it governs the rigidity of composites; the flow and rheology of the melt or liquid; and the tensile and impact strength and surface smoothness of the PC (Roger & Hancock, 2003). The shape is determined by the genesis of the filler, i.e., its chemistry, crystal structure, and processes undergone. Some typical particle shapes likely to be found include roughly spherical, blocky, irregular, platy, acicular, and porous shapes as illustrated in Figure 2.6. Generally, cellulose filler has porous particles.

Filler having good dispersion characteristic, gives extra advantages to the polymer composites system. The most common direct measurement of level of dispersion is by using Scanning Electron Microscopy (SEM) on a cross-section in microscopic scale. Additionally, good dispersing filler is a prerequisite for good mechanical and aesthetic properties. Multiple studies have shown that agglomerates of filler in composites system act as stress concentrator and reduce tensile and impact strength (Riley *et al.,* 1990; DeArmitt & Hancock, 2003). Other than that, good dispersion also gives smoother surface (Riley *et al.,* 1990; DeArmitt & Hancock, 2003). Dispersion and wetting of filler can also affect the permeability of the composites.

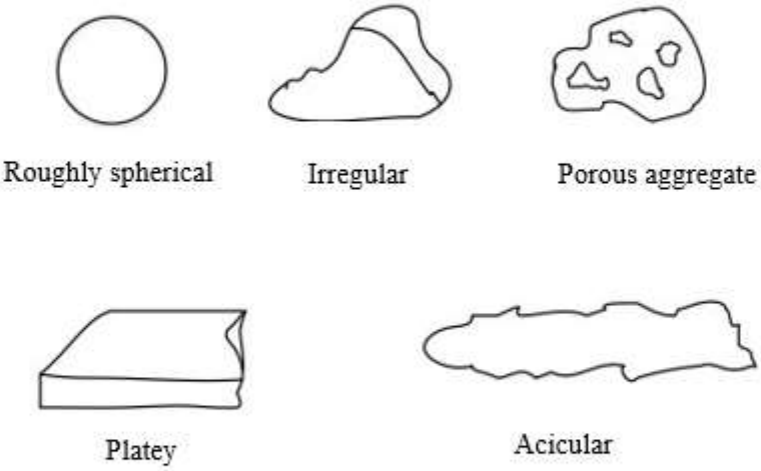


Plate II: Some particle shape in common filler
Source: Roger & Hancock, 2003.

2.1.3.2.1 Sorghum husk ash (SHA)

Over the last few decades, the sorghum corn industry has grown in Nigeria and become an important agriculturally based industry. Currently, Nigeria is one of the countries which has the most producers and exporters of guinea corn (United State Department of Agriculture, 2020). Undoubtedly, the industry also produces abundant waste alongside the guinea corn.

Sorghum husk ash (SHA) is a by-product from the guinea-corn milling industry and it is produced from combustion process of sorghum husk at high temperature of about 800 to 1000 degree Celsius. After completing the combustion process, SHA is collected and transported for dumping in open landfills. Agricultural waste disposal often causes health hazard and environmental pollution (Nagendran, 2011; Ziarati *et al.*, 2018 & Ismail *et al.*, 2010). Williams *et al.*, (2014) have characterized chemical composition and chemical properties of ordinary SHA as shown in Table 2.3.

Table 2.3: Chemical composition and properties of SHA

Chemical Composition	(%)
SiO_2	49.50
Al_2O_3	7.95
Fe_2O_3	14.94
MgO	0.49
P_2O_3	2.45
SO_3	0

Source: (Williams *et al*., 2014)

2.2 Curing Process of Polymer Concrete

There are two types of common curing methods for PC - air-dry curing and heat curing (post-curing). Other than that, Ohama and Demura (1982) has conducted water curing for polyester PC, but the results after 1, 5, 15 and 24 hours of water-curing indicated poorer compressive strength as compared to air-dry and post-curing method. The reason was that the saturated water condition had disturbed the exothermic reaction during polymerization process. Therefore, water curing is not appropriate for PC curing. Table 2.4 shows the summary of curing system that have been used for polyester resin-based concrete by previous researchers.

Table 2.4: Summary of Curing System

Air dry curing		Heat-curing		
Curing temperature (^oC)	**Curing period (hour)**	**Curing temperature (^oC)**	**Curing period (hour)**	**References**
20	24	70	1.5, 15, 24	(Ohama & Demura, 1982)
20	24	70	24	(Mani *et al.,* 1987)
20	36	20	168	(Sung *et al.,* 1997)
20	10	80	15	(Oshima & Hayashi, 1997)
20	168	-	-	(Soh, 1997)
*NM	*NM	40	*NM	(Soraru & Tassone, 2004)
-	-	105	10	(Aldrighetti *et al.*, 2005)
20	168	80	4	(Reis, 2006)
20	36	80	24	(San-Jose *et al.,*2008)

*NM - Not mentioned in the study

Most researchers kept the specimens of PC to air-dry curing (ambient room temperature) for long period of time (10 to 168 hours) before the specimens were heat-cured (post-curing). The long period of curing is because the ambient temperature of cold climates country is low. Additionally, PC is normally cast during winter or in cold conditions since it is very sensitive toward high temperature. Nonetheless, it has to be completely post-cured (heated) to attain its maximum compressive strength. In general, there is no standard curing temperature and period for heat curing as long as sufficient target strength of PC can be obtained.

2.3 Filler in Polymer Concrete

Roger and Hancock (2003) have stated that the properties of PC incorporating filler are significantly affected by several factors such as the type of filler and binder; the size and shape of filler; and also the amount of binder used. In this sub-section, effects of filler on physical and mechanical properties of PC are covered to achieve research objective. Incorporation of filler in PC is significant because PC normally has low binder content. Such incorporation can also replace dry fine aggregates to enhance the PC's properties.

2.3.1 Physical properties

The effect of filler on physical properties of PC can be obtained by conducting non-destructive test. The physical properties are mostly governed by water absorption and apparent density of PC.

2.3.1.1 Water absorption

Incorporating filler in PC significantly reduces the percentage of water absorption. Mani *et al.,* (1987) have conducted water absorption test on polyester concrete incorporating 0% to 5% filler content at different type of immersion - in cold and hot water. The researchers found that percentage of water absorption on PC incorporating 5% of filler content had obviously reduced more than unfilled PC.

Additionally, the PC immersed into cold water had the lowest water absorption. Hwang *et al.,* (2009) conducted water absorption test on polymer mortar incorporating calcium carbonate as filler and rapid-chilled steel slag as the fine particles. The researchers proved that water absorption had decreased significantly when the polymer binder and fine particle content increased. This happened because the fine particles had filled all gaps but left large air pockets. Another reason was that the particle itself has small water absorption. Generally, PC incorporating filler significantly reduces water absorption less than one percent. The findings from Mani *et al.,* (1987) and Hwang *et al.,* (2009) are given in Table 2.5.

Table 2.5: Water Absorption of PC Incorporating Filler

Type of concrete	Type of filler	Filler content (%)	Water Absorption (%)	References
		0	0.19 (cold water) 0.33	
Polyester –PC	Calcium carbonate	5	(Hot water) 0.11 (Cold water) 0.26-(Hot water)	(Mani *et al.*, 1987)
Ethylene Terephthlated based polymer mortar	Fly ash	20	<1	(Rebeiz *et al.*, 1992)

2.3.2 Mechanical properties

Generally, PC incorporating filler has better mechanical properties due to its effective dispersal in mixture and ability to induce denser concrete mixture packing, though the type of filling materials (either natural, granulated, or synthetic filler) also plays a pivotal role in affecting the composite characteristics. Basically, the effect of filler on physical properties of PC can be obtained by conducting destructive test.

Table 2.6 shows summary on the effect of various types of filler and filler content on mechanical properties of PC.

Table 2.6: Effect of filler on mechanical properties of PC

Types of Concrete	Types of Filler	Filler content (%)	Compressive Strength (MPa)	Flexural Strength (MPa)	Splitting tensile (MPa)	References
Polyester-PC	Calcium carbonate	0 5	41.2 68.0	12.2 19.3	10.8 12.1	(Mani et al., 1987)
Isophthalic based PC	Fly ash	8 20	80.4 96.8	17.9 22.8	- -	(Gorninski et al., 2007)
Orthophthalic based PC	Fly ash	8 20	72.5 86.8	18.0 20.5	-	(Gorninski et al., 2007)
Ethylene Terephthlate based polymer mortar	Fly ash	20	90.2	18.0	-	(Rebeiz et al., 1992)
Ethylene Terephthlate based PC	Fly ash	13	91.8	18.7	-	(Rebeiz et al., 1992)

From the summary, PC with highest filler content had significantly enhanced compressive, flexural and splitting tensile strength. However, filler has different fineness and higher fineness tends to give superior mechanical properties. This fact was supported by the work conducted by Atzeni *et al.,* (1990) and Gorninski *et al.,* (2007). Atzeni *et al.,* (1990) found that incorporating fly ash as filler in PC enhances the mechanical properties more than conventional quartz flour filler due to the greater fineness. This was supported by Soh (1997) where the researcher compared between fly ash and ground calcium carbonate as filler in PC. From the results, fly ash has better characteristics as filler as compared to ground calcium carbonate, giving the final PC superior mechanical properties. All-in-all, most studies concluded that, the higher the fly ash filler content, the denser the packing of PC's structure. It has also been proven that denser concrete with finer fillers undergoes delayed diffusion of aggressive agents (Gorninski *et al.,* 2007; Usman *et al.,* 2014)

2.4 Application of Polymer Concrete

Strong materials are needed to overcome the weak properties of conventional materials and to acquire high manufacturing standard in the construction industry. In this sense, polymer mortar and PC are advanced constructions materials which have been the dominating construction materials in Japan since the 1970s as shown in Table 2.7 for polymer mortar and Table 2.8 for polymer concrete (Ohama, 1997).

Table 2.7: Applications of Polymer Mortar in Japan

Application	**Location of work**
Floorings	Floors for houses, warehouses, offices, schools, hospitals, factories, shops, toilets, passages, stairs, garages, railway platform, train floors, and etc
Pavements	Roads (sidewalks and roadways), bridge decks, foot-bridge decks, parking lots, airport runways, and etc.
Anticorrosive linings	Effluent drains, chemical or machinery plant floors, grouts for acid-proof tiles, floors for chemical laboratories, electrolytic baths, pharmaceutical warehouses, septic tanks, hot spring baths, offshore structure, e.g. piers and sea berth, and etc.
Adhesives	Adhesives for floorings, walling materials and heat insulating materials, tile adhesives, adhesives for joining new cement concrete or mortar to old cement concrete or mortar, embedment of anchor bolts and etc.
Repair materials	Grout for repairing cracks and delamination of concrete structure, patching materials for damaged concrete structures, rustproof coatings for corroded reinforcing bars, resurfacings for managed concrete pavements, and etc.

Source: Ohama, 1997

Table 2.8: Application of PC in Japan

Application	Location of work
Integral waterproofing	Concrete roof floors, mortar walls, concrete blocks, water tanks, swimming pools, septic tanks, silos, and others.
Small-diameter automated shield tunneling systems	Telecommunication cable lines, sewage works, and others.
Structural precast products	Manholes and hand holes for telecommunication cable lines, electric power cable lines and gas pipelines, prefabricated cellars, or stack rooms, tunnel liner segments for telecommunication cable lines and sewerage systems, piles for pot or hot spring construction, forms for reinforced concrete structure, FRP-reinforced frames or panel for buildings, machine tool structure, e.g. beds saddles, works of art, e.g. carved statue and object d'art, tombs for Buddhists, and others.
Non-structural product	Gutter covers, U-shape gutter, footpath panels, terrazzo tiles and panels, and large-sized or curved decorative panels for buildings, partition wall panels, sinks, counters, washstands, bathtubs, and others.
Cast-in place applications	Spillway coverings in dams, protective linings of stilling basin in hydroelectric power stations, coverings of check dams, foundations of buildings in hot spring area, acid-proof linings for erosion control dams with acidic water, and others.

Source: Ohama, 1997

CHAPTER THREE

3.0 MATERIALS AND METHODS

3.1 Methodology

The research methodology in this study is divided in to three major phases:

Phase1: Characterization on raw materials of polymer binder and micro – filler,

Phase 2: Properties of polymer concrete incorporating micro- fillers,

Phase 3: Water absorption behaviour of poly concrete to normal concrete substrate.

Phase 1 required that proper characterization had been for the polymer binder followed by the filler. In order to achieve objective 1, the research frame work as shown in Figure 3.1 was used.

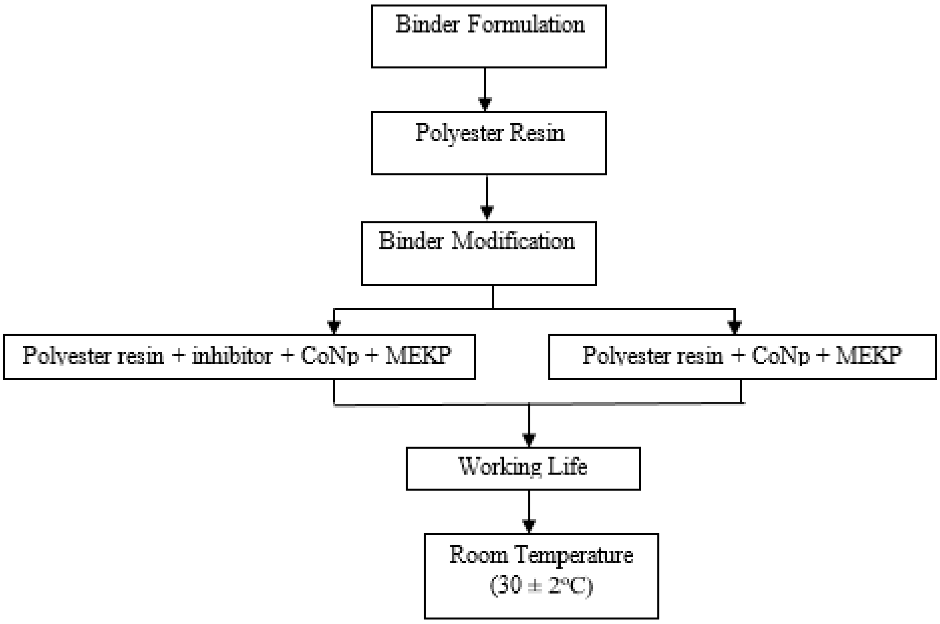


Figure 3.1: Characterizations on raw materials of polymer binder

To limit the scope, polyester resin was used as polymer binder because it is cheaper than other thermoset resins, especially epoxy resins (Lim *et al.*, 2009) due to high sensitivity of thermoset resins to hot temperature which speeds up the polymerization process, polymer inhibitor additive was introduced into the binder formulation to prevent the problem of premature gelation and provides sufficient time to complete the hardening process. Other main polymer additives which serve as curing agents include Cobalt Naphthenate (CoNp), which acts as the initiator, and Methyl Ethyl Ketone Peroxide (MEKP), which acts as the cross linker. For comparison, the binder formulation without inhibitor additive content was designed as the control binder formulation under the same condition. In this study, the proportions of CoNp and MEKP were fixed at 0.5% and 1% by binder weight, respectively. Physical observation was done on the fresh working life of the binder. The fresh binder working life was measured in ambient room temperature (30 ± 2^0c).

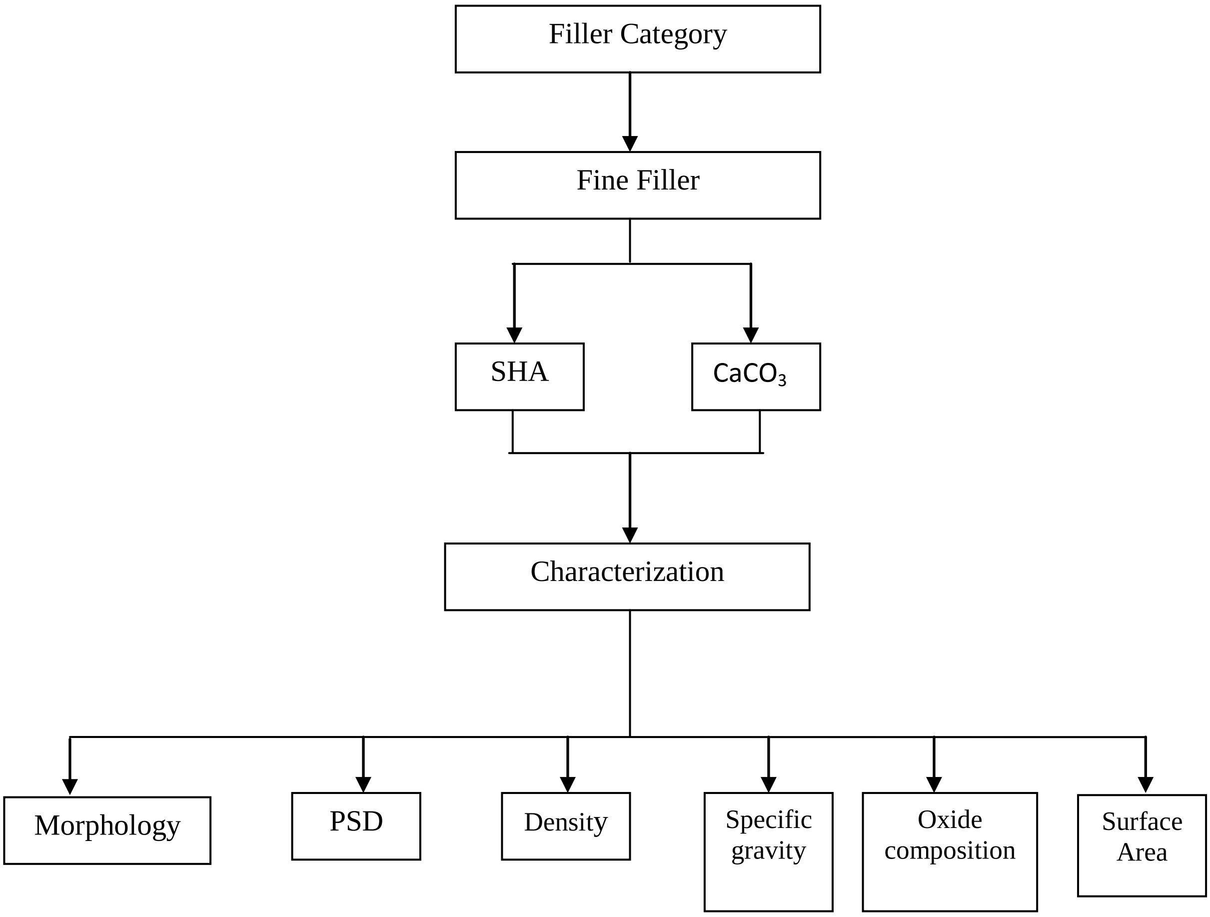


Figure 3.2: Development and properties of raw materials of filler

Figure 3.2 shown above describes the skeleton works on raw materials of filler. In this phrase, one category of filler was produced (fine filler). SHA was used to alternate Calcium Carbonate ($CaCO_3$), waste in conventional filled – PC. The filler and its size were limited to those passing through the sieve sizes of 300µm to 45µm. SHA was physically modified to obtain the particles passing through 45µm sieve size. Inert granular materials such as course and fine aggregates were utilized as well and this is similar to conventional concrete. To obtain a uniform mix and strength of PC, the size of coarse and fine aggregates was limited to those retained at 10 mm and passing 5mm sieves, respectively. All fillers and inert granular materials were oven dried and the moisture contents maintained below 0.5%. The characteristics of SHA as fillers in PC were examined to determine its performance from engineering perspectives. Calcium carbonate waste was included as filler to allow comparisons to be made with SHA. Fillers characterization which covered oxide composition by using ASTM C618-2017 standard, density test by following ASTM D1895-2003 standard, specific gravity test using ASTM C128-2007 standard, morphology examination using X-ray fluorescence (XRF), surface area using Brauer-Emmett-Teller Nitrogen Absorption (BET) and particle size distribution analysis by passing the fillers through micrometre size sieve.

Development and properties of PC were performed after the raw material had been identified as shown in Figure 3.3. This began with determination on the desired post-curing regime of PC, followed by the formulation of PC mix. This stage was taken as the pilot study.

Polymer binder, filler, fine aggregates and coarse aggregates were the main mix formulation for the PC. Polyester resin was employed in this study as the polymer binder. The polyester binder was blended with two types of filler; SHA and calcium carbonate ($CaCO_3$). In order to make an ideal comparison, PC incorporating SHA was paired with PC incorporating $CaCO_3$. The mix proportion was designed to have low binder content of 11% in accordance to previous researchers (Fowler, 1999; Reis, 2011; Varughese & Chaturvedi, 1996; Haidar *et al.*, 2011).

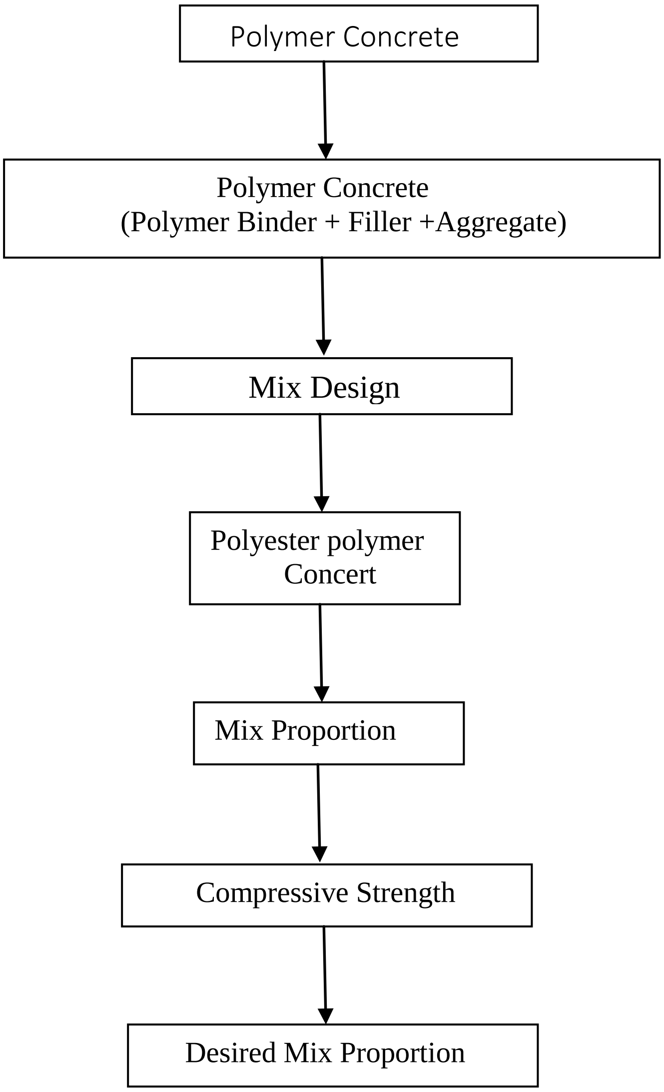


Figure 3.3: Development and Properties of PC

This design depends on the filler's absorbability to the liquid. Meanwhile, mix proportion for the filler content varied consistently from 12%, 14% to 16% and the constant fine aggregates content was prescribed at 30% to obtain predictable desired strength. The weight difference of polymer concrete mix was carried out to determine their density and water absorption behavior of PC was carried out at 3, 7, 14 and 28 days. Then, all PC mixes were subjected to compressive strength test to identify the optimum desired mix proportion.

At this stage, only the desirable optimum mix proportion was employed for further investigation on properties of PC. The skeleton of related works is presented in Figure 3.4. The test started with non-destructive test; density and water absorption of PC. Then, compression, test was employed to determine its mechanical property.

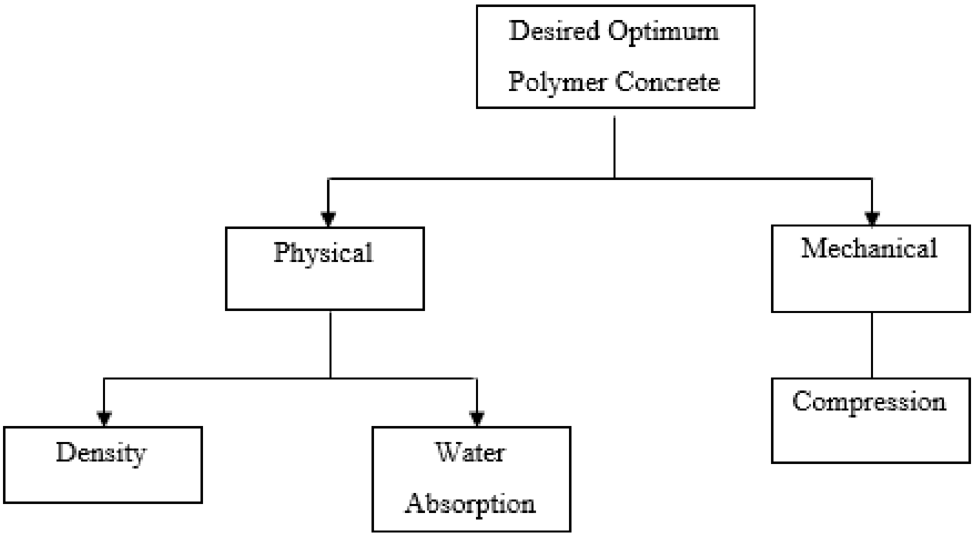


Figure 3.4: Properties of PC incorporating micro-filler

The effect of filler on physical and mechanical properties of PC was investigated after optimum desired mix proportions had been done.

Table 3.1: Type of testing, standard method and number of specimens

Phase 1: Characterizations of Raw Materials					
Type of testing (Binder)	**Standard Method**	**No. of Specimen**	**Type of Testing (Filler)**	**Standard Method**	**No. of Specimen**
Working life	JIS A 1181 (2005)	18	Density	ASTM D1895 (2003)	3
			Specific gravity	ASTM C128 (2007)	3
			XRF	ASTM E1621 (2013)	4
			SEM	*PR	4
Phase 2: Properties of PC					
Type of testing	**Standard Method**	**No. of Specimen**	**Type of Testing**	**Standard Method**	**No. of Specimen**
Density	ASTM C642 (2015)	105	SEM	*PR	4
Water – Absorption	ASTM C642 (2015)	105			
Cube Compression	BS EN 12390 – 3 (2009)	105			

Note: *PR is referred to previous research of standard method

3.2 Materials

The following are the materials used for this study: Calcium carbonate, Sorghum Husk Ash (SHA), Calcium Carbonate ($CaCO_3$), Polyester resin, Cobalt Naphthenate (CoNp), Methyl Ethyl Ketone Peroxide (MEKP), Methyl Methacrylate (MMA), Fine aggregates and Coarse aggregates.

3.2.1 Sorghum husk ash (SHA)

SHA is an incinerated ash derived from the incineration of sorghum husk after harvest of sorghum. The husk from sorghum was got at open fields dumped by local farmers at Maikunkele, Minna, Niger state. It was sun-dried for 24hrs and then burnt in the open-air method for 4 days with a locally fabricated incinerator available in the laboratory as presented in Abalaka and Okoli (2013). The resulting powders were pulverized in a local mill at Gidan-Kwano village of Minna and then sieved with a 75μm sieve. The production process of SHA filler is represented in Figure 3.5 as shown.

(a) Sorghum Husk

(b) Incinerator set-up

(c) Burning of Sorghum husk to Ash by open burning

(d) Sorghum Husk Ash system

(e) Grinding process of burnt ash to finer particles

(f) Sieving to 45 Micrometer particle size

Plate III: Production Process of SHA

3.2.2 Calcium carbonate

The Calcium Carbonate was purchased at HSHF Mining Company, Ukiwe Street, Kubwa, Abuja. All specifications on Calcium Carbonate were verified from the manufacture's detail to ensure they conform to the JIS standard. Figure 3.6 presents a sample of Calcium Carbonate used in the research work.

Plate IV: Calcium Carbonate

3.2.3 Polyester resin, hardeners and inhibitor

The polyester resin, hardeners and inhibitor are all purchased in Lagos State from a chemical dealing agent. All specifications on the resin formulations were verified from the manufacture's detail to ensure they conform with JIS standard.

3.2.4 Fine aggregates

Natural occurring river sand was used as fine aggregates in all the mixes, with maximum particles size of 4.75 mm (No 4) in the upper limit in accordance with ASTM delineation. The specific gravity of the sand was 2.64. The sand was oven-dried before use to ensure it is moisture free since polymer resin is hydrophobic.

3.2.5 Coarse aggregates

Locally available air-dried crushed granite of 12mm maximum size was used in all the mixes. Care was taken to ensure that the aggregate was free from moisture, organic matters such as dry muds, leaves and other deleterious materials.

3.3 Methods

3.3.1 Characterizations on raw materials

This sub-section describes the overall methods related to characterizations of raw materials of polymer binder and micro-filler.

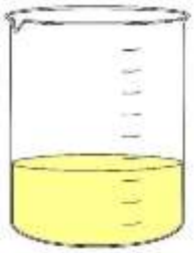
3.3.2 Characterizations on polymer binder

The physical property of interest of the polymer binder is described in this sub-section. Polyester resin was employed as the polymer binder. At this point of investigation, working life tests for fresh polymer binder were conducted to obtain the physical property.

3.3.2.1 Working life

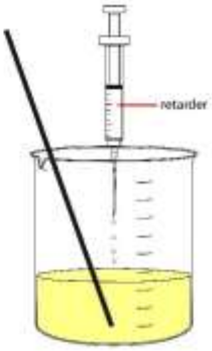
Working life is the time taken for polymer binder to reach a viscose stage where it becomes unworkable. A simple test of working life was conducted in accordance to JIS A 1181 (2005). In order to have sufficient time to produce PC at ambient room temperature of 30 ± 2^{o}C, polymer inhibitor additive was added into the binder formulation followed by 0.5% of CoNp. The resin and the additives were stirred until their colors became homogenously apparent. Then, immediately after the addition of 1% of MEKP, the resin was observed for apparent changes.

The stirring process continued until the resin threads appeared to stick at the mixer container. The polymer inhibitor was limited from 0% to 1% by resin weight to get a sufficient time to cast the PC and similar study have been conducted by Li and Lee (2002). In this study, the ‘sufficient time’ must cover the time during casting, pouring, compacting, and finishing of the fresh concrete. Nevertheless, it is a best practice that the time also covers the time for cleaning the tools before transferring the specimen into the oven for heat curing. Polymer binder without inhibitor was employed as control specimen. The details of test procedure are presented in Figure 3.7. Consistency of time recorded for working life was observed by monitoring the surrounding humidity.



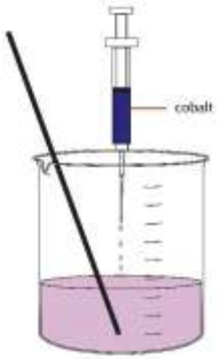
Step 1:

100 g of pure polyester resin was prepared**.**



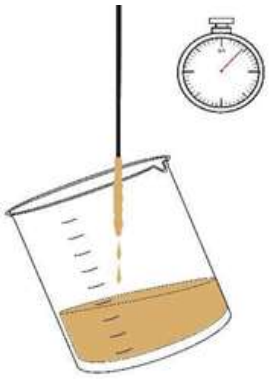
Step 2:

Polymer additive of inhibitor was added by resin weight into polymer resin. There was no change of pure resin colour when the resin was stirred.



Step 3:

0.5% promoter of CoNp by resin weight was added into polymer resin. The resin colour changed to light purple when stirred.



Step 4:

Stopper was started immediately when the MEKP was added into the polymer resin. The stirring process was continued by using skewer until the resin threads appeared on the stick mixer.

Plate V: Test procedure for working life test

3.3.3 Characterizations on micro filler

Sorghum Husk Ash (SHA) was used in this study. It was selected in consideration of environmental factors, ease of handling and being a non-toxic agriculturally based material and it was oven-dried at 100 ± 5^oC and the moisture content of SHA was kept constant below 0.5% because polymer is a hydrophobic material. In this study, the SHA was classified as fine SHA. Fine SHA was obtained directly by passing the SHA through 45 µm sieve to separate their natural substances and homogenize the filler sizes. Meanwhile, the fine SHA was obtained by using grinding machine to obtain finer filler. In this study, the fillers which passed through 45µm sieve were categorized as fine filler. Therefore, for comparison purpose, SHA and $CaCO_3$ will be jointly taken as the fillers.

3.3.3.1 Density

The density of filler is one of the important parameters to calculate the volume fraction later on. The test to determine apparent density of overall fillers strictly followed ASTM D1895 (2003). The details of test procedure are highlighted in Figure 3.8:

Step 1:

First, apparatus for density test was assembled.

Step 2:

Inside diameter and height of Appropriate container was measured.

Step 3:
Oven-dried powder (filler) was Poured into the funnel. The small end of the funnel was closed at this point.

Step 4:

After the powder had passed through the funnel, the excess at top of container was scrapped off using a straightedge without shaking the container.

Plate VI: Test procedure for density test of powder materials (filler).

The test was repeated for three specimens and an average of apparent density of material was calculated using Equation 3.1:

$$\rho = \frac{w}{v} \tag{3.1}$$

where,

ρ is apparent density in kg/m^3

W is mass of materials in g

V is volume of container in cm^3

3.3.3.2 Specific gravity

The importance of specific gravity test of fillers is paramount to determine the volume fraction of samples for mix design purpose. The test was conducted in accordance with ASTM C 128 (2007) using the cylinder tube for SHA and $CaCO_3$. All materials were oven-dried for accuracy. The specific gravity of the fillers was determined by the following steps:

Step 1:

The cylinder tube wit cover (air tight) was weighed on the scale balance as W_1

Step 2:

One-third (1/3) of the tube’s height was filled with dry sample, covered and weighed as W_2

Step 3:

The dry sample in the cylinder tube was filled up with water, covered and weighed as W_3

Step 4:

Cylinder tube filled up with water only covered and weighed on the scale.

Plate VII: Test Procedure for Specific Gravity of Powder Materials (Fillers)

The test was recorded for three specimens and an average was calculated using Equation 3.2:

$$S.G = \frac{W_2 - W_1}{(W4 - W1) - (W3 - W2)} \qquad (3.2)$$

3.4 Properties of Polymer Concrete Incorporating Micro-Filler

Physical tests involved were tests for density and water absorption while the mechanical test involved was compression test. These tests were conducted to investigate the effect of filler on the physical and mechanical properties of PC.

3.4.1 Mix proportion of polymer concrete

In this study, the key materials for PC were unsaturated polyester resin, fine aggregates, coarse aggregates, and filler as illustrated in Figure 3.13.

The mix proportion for polyester resin of PC incorporating SHA and Calcium carbonate is shown in Tables 3.3, 3.4 and 3.5, respectively. The overall binder content was limited to 11%. Unsaturated polyester resin was mixed separately with inhibitor additive of Methyl Methacrylate (MMA), 0.5% of Cobalt Naphthenate (CoNp) as the initiator and 1 % of Methyl Ethyl Ketone Peroxide (MEKP) as crosslinker, while the filler content was varied to 12%, 14% and 16% and the constant coarse aggregates content were prescribed with 30% to obtain predictable strength.

Table 3.2: Mix Proportion of PC Incorporating SHA

Mix Proportion							
Up binder		**SHA Filler**		**Fine Aggregates**		**Coarse Aggregates**	
%	kg/m^3	%	kg/m^3	%	kg/m^3	%	kg/m^3
11	1210	12	278.4	46	1214	30	750
		14	324.8	44	1161		
		16	371.2	42	1108		

Note: Polyester binder was mixed with inhibitor, 0.5% CoNp and 1% MEKP
*Up = Unsaturated Polyester

Table 3.3: Mix Proportion of PC Incorporating Calcium carbonate ($CaCO_3$)

Mix Proportion							
Up binder		**$CaCO_3$ Filler**		**Fine Aggregates**		**Coarse Aggregates**	
%	kg/m^3	%	kg/m^3	%	kg/m^3	%	kg/m^3
11	1210	12	324	46	1214	30	750
		14	378	44	1161		
		16	432	42	1108		

Note: Polyester binder was mixed with inhibitor, 0.5% CoNp, and 1% MEKP

Mix proportion by volume fraction of PC was design in accordance with the work of Khalid *et al.* (2015):

$$W_f = \rho_f V_f \tag{3.3}$$

where,

W_f is weight of material in kg

ρ_f is density of material in kg/m^3

V_f is volume of mould in m^3

Table 3.4: Mix Proportion of Polyester Binder Formulation

Mix Proportion							
Up binder		**MMA**		**CoNp**		**MEKP**	
%	Kg	%	Kg	%	Kg	%	Kg
11	1210	0.6	7.26	0.5	6.05	1	12.10

3.4.2 Mix proportion of normal concrete

The main materials for making normal concrete in this study were cement, coarse aggregates, fine aggregates, and tap water. Additionally, mix proportion of normal concrete is presented in Table 3.5. A slump of 30-60 mm was designed for the normal concrete with a characteristic's strength of 30 N/mm^2.

Table 3.5: Mix Proportion of Normal Concrete

Concrete materials	**Mix proportions (kg/m^3)**
Cement	427
Coarse aggregates	950
Fine aggregates	810
Water	213

3.4.3 Preparation of Polymer Concrete

Preparation of PC strictly followed JIS A 1181 (2005). There are two types of mixes involved: dry mix and wet mix. Step-wised preparation is listed below.

i. Dry mix consisting inert granular aggregates (fine and coarse aggregates) and filler was prepared and weighed according to mix proportion. Then, all dry mixes were mixed together using concrete mixer.

ii. The wet mix had additional polyester binder mixed together with its inhibitor of MMA, initiator of CoNp and crosslinker of MEKP.

iii. Once crosslinker of MEKP was added into the binder formulation, time was recorded. Then, the wet mix was poured into the dry mix and immediately mixed together until all materials had blended well.

iv. Preparation of PC had to strictly follow the duration of working life. Therefore, the moulds had to be prepared beforehand, tightened and cleaned from undesirable substances.

v. Releasing agent was applied onto the mould surface for easy demoulding.

vi. Fresh concrete was poured into the mould and then compacted. This was repeated three times until the mould had been fully filled. PC was compacted using vibrating table for 10 seconds every layer.

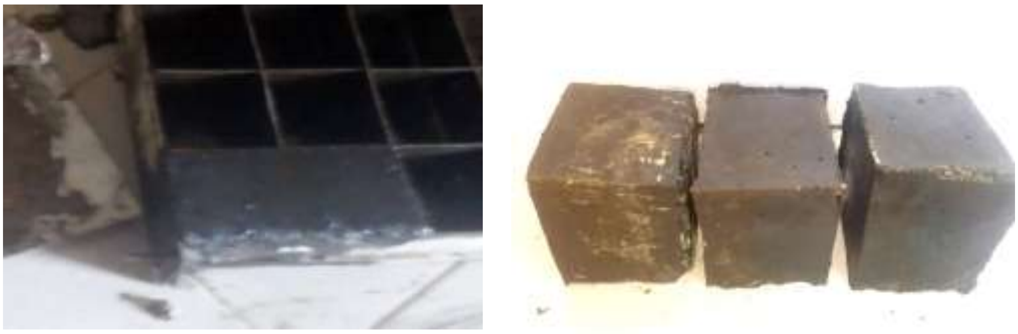
vii. The specimens were post-cured through oven at $70^{o}C$ for three hours. Curing temperature and period were determined according to the pilot study.

viii. All tools were cleaned with acetone and pieces of paper.

Figure 3.10 illustrates polymer concrete incorporated with sorghum husk ash (PC-SHA) and PC-$CaCO_3$ preparation and hardened PC cube after being post cured in oven.

(a) Fresh concrete poured into the mould and then compacted

(b) Casting PC-$CaCO_3$



(c) Casting PC-SHA

(d) Hardened PC-SHA

Plate VIII: Preparation of Polymer Concrete

3.4.4 Post-Curing system of polymer concrete

The post-curing regime of unsaturated resin-based PC with and without incorporating any filler was investigated. A total of 105 specimens with dimensions of 100 mm x 100 mm x 100 mm were prepared according to JIS A 1181 (2005). The specimens were left at ambient room temperature of 30 ± 2^{o}C for an hour and then oven-dried as a form of post-curing to complete the curing process. The specimens were pre-cured for an hour in ambient room temperature based on the study of working life of the polymer binder as discussed in sub-section 3.3.2.1. At that point, the specimens were post-cured at different curing temperature of 30^{o}C, 50^{o}C and 70^{o}C and at different curing period of 1, 3, 6, 16 and 24 hours. Dry density, water absorption and compression test were carried out on the PC cube. All specimens were tested according to the design of curing temperature and period. The compressive strength was obtained from the average of three specimens for each condition. Then, the little part of crushed specimen was investigated under scanning electron microscopy (SEM).

3.4.5 Desired mix proportion of polymer concrete

The mix proportion of PC in this study was investigated under cube compressive strength. Design of mix proportions are followed according to mix proportion with low binder content.

3.4.6 Properties of polymer concrete

Physical properties of polymer concrete in this study were obtained under density and water absorption tests while only the compressive strength properties were carried out under the mechanical properties as it is known that the basic performance characteristic of any material is hinged on its compressive strength. The morphological properties were also tested. The details of tests conducted in this research are explained in the next sub-section.

3.4.6.1 Density

Density is mass per volume and the mass of cured PC were measured by using digital weighing scale. PC's density is a basic parameter to be measured before conducting any destructive test. Consistency of PC's production can be monitored from the basic density and it can be calculated from Equation 3.5:

$$\rho_C = W_C / V\mathrm{c} \tag{3.5}$$

Wc is mass of cured PC in kg

Vc is volume of cured PC in m^3

3.4.6.2 Water absorption of polymer concrete

The density and water absorption of hardened PC was jointly determined using the test procedure specified in ASTM C 642 (2015). The details of test procedures are listed.

i. Hardened PC specimens were immersed in the water bath. The specimens were kept fully submerged for 3 days.
ii. Wet specimen was drained and surface-dried. The weight of specimen was recorded. The surface-dried mass after immersion is designated as B.
iii. The test was repeated for 7 days, 14 days and 28 days

Generally, water absorption takes long time to complete the periodic test. However, the measuring data was stopped when it reached differences between data of about less than 0.5%. Water absorption of PC was calculated using Equation 3.6.

$$Absorption\ (\%) = \frac{B-A}{A} \times 100 \qquad (3.6)$$

A is mass of oven-dried of PC in air in g

B is mass of surface-dried of PC in air after immersion in g

3.4.6.3 Cube compressive strength

The compressive strength of all cubes with the dimensions of 100 mm x 100 mm in cross section were determined according to BS EN 12390-3:2009. Compression test was carried out using compression testing machine. The compressive strength was calculated using Equation 3.4:

$$f_c = \frac{F}{A_c} \qquad (3.4)$$

where,

fc is compressive strength in MPa (N/mm^2)

F is maximum load in N, Ac is cross section of the specimen in mm^2

(In this case, cross section of specimen is 100 mm by 100 mm)

3.4.6.4 Microstructure properties

Microstructure properties of PC in this study were obtained under morphology image of Scanning Electron Microscopy (SEM).

CHAPTER FOUR

RESULTS AND DISCUSSION

4.1 Characterization of Raw Materials

Characterization of raw materials of polymer binder and micro-filler were covered in this sub-section.

4.1.1 Characterization of polymer binder

All additives included into polymer binder such as CoNp and MEKP were constant, except for inhibitor additive. Therefore, the effect of inhibitor additive on working life of polymer resin was investigated. This point of investigation was conducted to obtain objective 1.

Table 4.1: Property of Polyester Resin

Properties	**Polyester resin**
Density (g/cm^3)	11.1

The Table 4.1shows the density of polymer binder as specified by the manufacturer's details

4.1.1.1 Working life

Working life of fresh polymer resin was visually observed to record the duration taken to become viscous before the curing process was completed. Figures 4.1 shows the duration of fresh working life with different inhibitor additive contents.

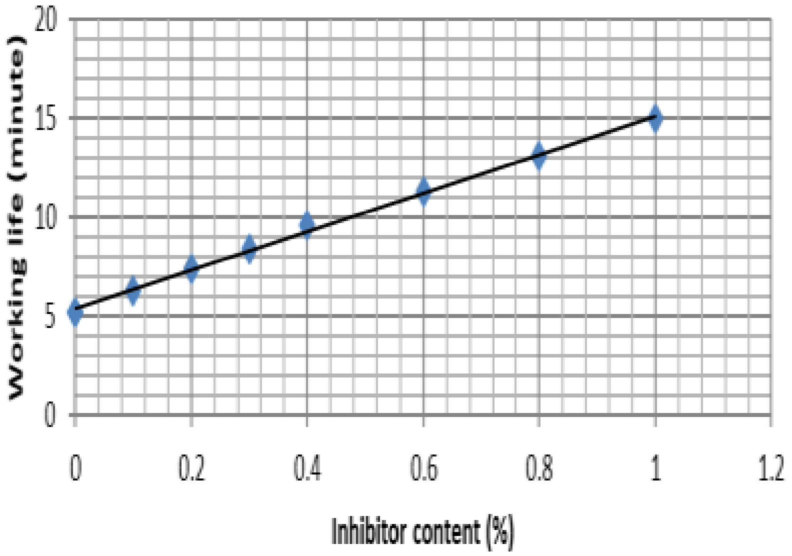


Figure 4.1: Effect of inhibitor on working life at ambient room temperature

The whole results revealed that the fresh working life had progressively increased when the inhibitor content was increased. The duration of working life in ambient room temperature managed to be prolonged approximately 5 minutes without inhibitor content and at approximately 15 minutes at 1.0% inhibitor content. However, the working life of polyester resin without inhibitor content was shorter than the polyester resin with inhibitor content. This happened because higher temperature had accelerated the polymerization process. Thus, this phenomenon indicates that the addition of polymer inhibitor is workable to prolong the duration of fresh working life in ambient temperature condition. Similar outcomes have been achieved by Li and Lee (2002).

Working life time without inhibitor additive was very short at 5 minutes and 18 seconds in ambient room temperature. Within this time duration, application of polymer concrete is limited which is considered as insufficient time period when thermoset polyester resin was employed as binder in PC. Since casting of concrete takes time to mix, pour and compact the fresh concrete as well as finishing and cleaning works, adding the inhibitor additive in binder formulation is important to prolong the working life of polymer resin in ambient temperature. Consequently, curing process and strength properties of polymer resin will not be interrupted. At this stage of investigation, the crosslinking of polymer completed very well with inhibitor additive of Methyl Methacrylate (MMA) up to 1.0%. This is because the resin has a unique chemical characteristic which interrupts the curing temporarily before the polymerization starts and the cross-linking is complete. At this stage, the addition of inhibitor additive of MMA is significant in ambient room temperature (30 ± 2°C).

4.1.2 Characterizations of filler

Two types of fillers were used in this research work viz. SHA and $CaCO_3$. Characterizations of filler were done before these fillers were incorporated into the PC. All tests related with this characterization are as shown in Figure 3.2. This point of study was conducted to achieve objective 2.

4.1.2.1 Density

The density of SHA and Calcium carbonate were presented in Table 4.1. The two samples gave average density of 0.24 g/cm^3 for SHA and 2.71 g/cm^3 for Calcium carbonate. From the results, SHA was seen to be lighter than Calcium carbonate material. This revealed that SHA filler has lesser moisture contents less than 0.5% in them when compared to Calcium carbonate. Also, this refilled that SHA filler has the tendency of producing lighter polymer concrete than $CaCo_{3.}$ SHA filler is kept in an air tight material and store in dry environment. The standard deviation (SD) and coefficient of variation (COV) for the two fillers are given in Table 4.1.

Table 4.2: Density of filler used

Type of filler	Density (g/cm^3)	SD,	CoV (%)
SHA	0.24	0.04	1.72
Calcium carbonate	2.71	0.02	0.68

4.1.2.3 Specific gravity

The specific gravity of SHA and Calcium carbonate are presented in Figure 4.1. SHA and Calcium carbonate gave average specific gravity of 2.32 and 2.70 respectively, which conform to standard as specified by British Standard (BS 812: part 107).

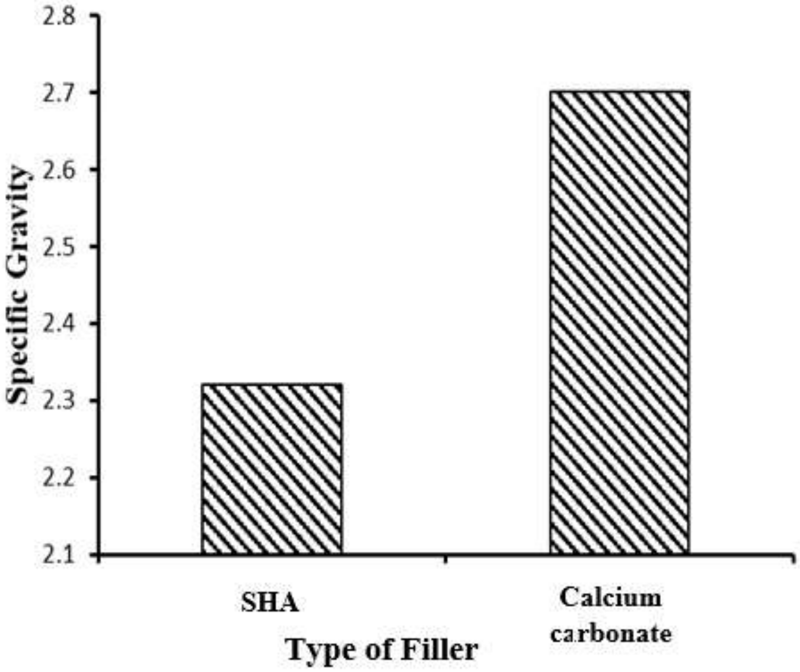


Figure 4.2: Average specific gravity

4.1.2.3 Chemical composition

Table 4.2 shows the chemical composition of SHA and Calcium carbonate compared with CEM 1 (42.5 N) Portland cement with brand name Dangote 3X. From the results, SHA has more oxide components than Calcium carbonate.

Table 4.3: Chemical Compositions of SHA, Calcium carbonate and Portland Cement (CEM 1-42.5N)

Chemical composition (%)	Type of filler		
	CEM 1	**SHA**	**Calcium Carbonate**
SiO_2	21.30	83.00	-
Al_2O_3	6.12	20.10	-
Fe_2O_3	3.47	10.89	-
TiO_2	0.25	1.16	
K_2O	0.23	2.45	-
MgO	1.24	2.05	-
CaO	65.0	0.76	89.2
Na_2O	0.10	0.45	10.0
P_2O_5	0.05	0.15	-
SO_3	1.02	2.38	-
MnO	0.05	0.00	-
Loss of Ignition	0.80	5.76	-

From the chemical composition of the oxides obtained, it is determined that the constituent materials of the SHA used are in harmony with the requirements of ASTM C 618 standard, and the material is an exceptional pozzolana, as it shows viable pozzolanic characteristics.

4.1.2.4 Particle size distribution

Figure 4.2 illustrates the particle distribution of the fillers used in this study. This test is predicated in other to examine the fineness of the pulverized SHA and the acquired $CaCO_3$. The SHA and calcium carbonate are categorized as fine fillers. All fillers were compared in terms of the fineness and particle size distribution curves of the two fillers, as shown in Figure 4.2. The distribution showed that the two fillers examined in this study contained well-graded fine. More than 90% particle of the two fillers had passed through 150 µm sieve. Further explanations on the filler's physical changes are in the morphology section of sub-section 4.2.2.5.

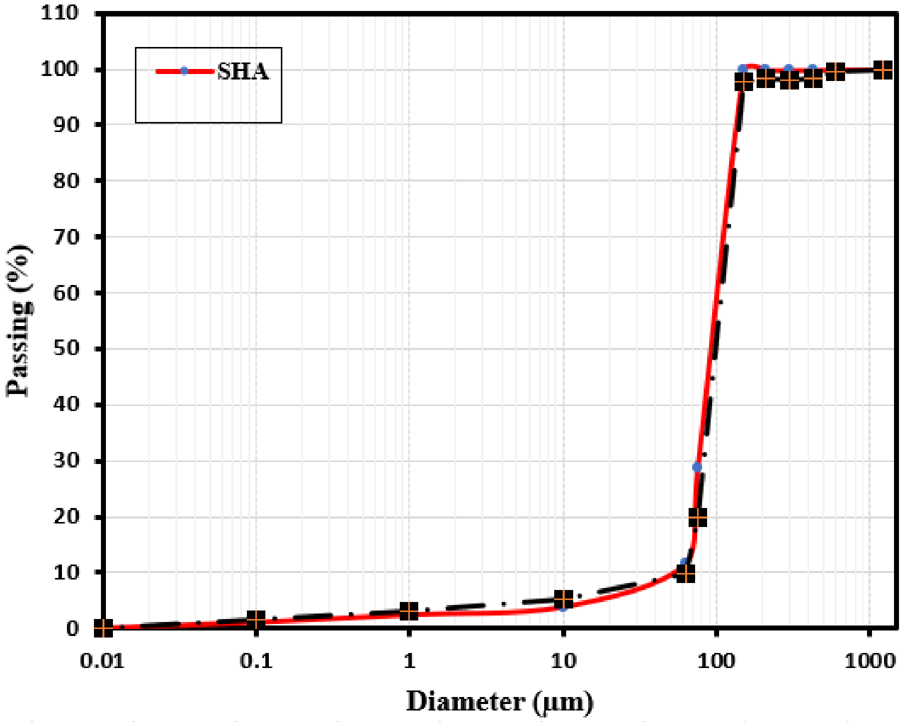


Figure 4.3: Particle size distribution of SHA and $CaCO_3$ fillers materials

4.1.2.5 Surface area

Surface areas of the two fillers examined in the study were obtained from Brunauer-Emmett-Teller nitrogen absorption (BET) test and the results are presented in Table 4.3. This finding is important to gauge the difference between the two fillers, it was found that the surface area of SHA was larger when compared with $CaCO_3$. This means that the finer filler has larger surface area since the surface area of SHA (3420 cm^2/g) was 1.20 times lesser than $CaCO_3$ (4091 cm^2/g). From the overall results, SHA and Calcium carbonate can be paired as fine filler because of their similar surface area. The characteristics of SHA is similar to what Bakar *et al.*, (2011) reported for RHA and Hafizah (2015) reported for POFA.

Table 4.4: Fineness of fillers

Type of Filler	Blaine surface area (cm^2/g)
SHA	3420
$CaCO_3$	4091

4.1.2.6 Morphology image of the micro fillers

Figure 4.3 presents the morphology images of two micro-fillers (SHA AND $CaCO_3$) used in this research. In general, no pores were spotted under SEM with 10000 times magnifications, but a more porous cellular structure similar to that found by Noorv and *et al.* (2013) using SEM was observed. The pores' size, which was within the average of 10 μm, was anticipated to affect the workability of fresh Polymer Concrete since it would lead to a higher propensity to absorb liquid resin, as stated by Noorvand *et al.* (2013). It was also expected to increase the amount of voids and capillaries, thus reducing the density of packed structure (Noorvand *et al.*, 2013; Mahjoub *et al.*, 2014). The SHA pore size was identical to those of naturally cellulose based plant. The physical grinding procedures that the SHA had undergone earlier had made the open cellulose structure to collapsed. This made the SHA to possess a high potential for usage for PC filler.

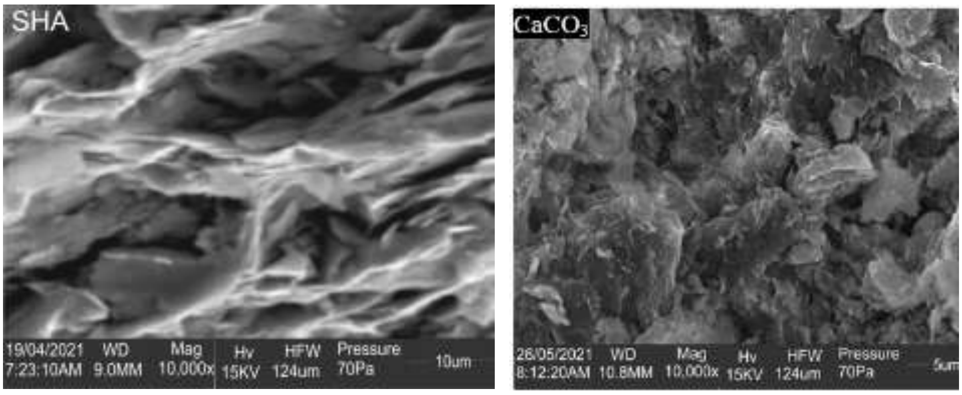


Plate IX: Morphology images of micro-filler (a) SHA (b) Calcium Carbonate (1000 times magnifications)

4.2 Properties of Polymer Concrete Incorporating Micro Filler

This section describes the properties of PC incorporating micro-filler, especially on the effect of filler on engineering properties. This began with determination of the mix proportion and properties of PC incorporating micro-filler.

4.2.1 Post-Curing of polymer concrete

In determining the desired post-curing temperature (heat curing) and curing period for the PC, fresh polymer concrete without filler was exposed to post-curing process. Compressive strength for all PCs without fillers at varying curing temperature of 30°C, 50°C, 70°C, and varying curing period of 2 hours to 24 hours at an interval of 2 hours was determined. From the experimental outcome, a non-linear relationship was observed between compressive strength of all the PCs without fillers cured at different temperature regime of 30°C, 50°C, 70°C, and their corresponding curing period as presented in Figure 4.4. The compressive strength progressively increased with increasing curing period and became constant after a specific curing period. During the first hour of curing period, the compressive strength for PC with curing temperature of 30^0C was negligible. However, after being cured for up to 16 hours, the compressive strength progressively reached approximately about 39 N/mm^2. In the case of PC cured at 50^0C and 70^0C, the study outcomes demonstrated that the highest compressive strength of 39 N/mm^2 was achieved also after 16 hours of curing period, this experimental outcome value is closely related to the value reported by Hafizah (2015). We can therefore deduce that compressive strength increased with prolonged curing period.

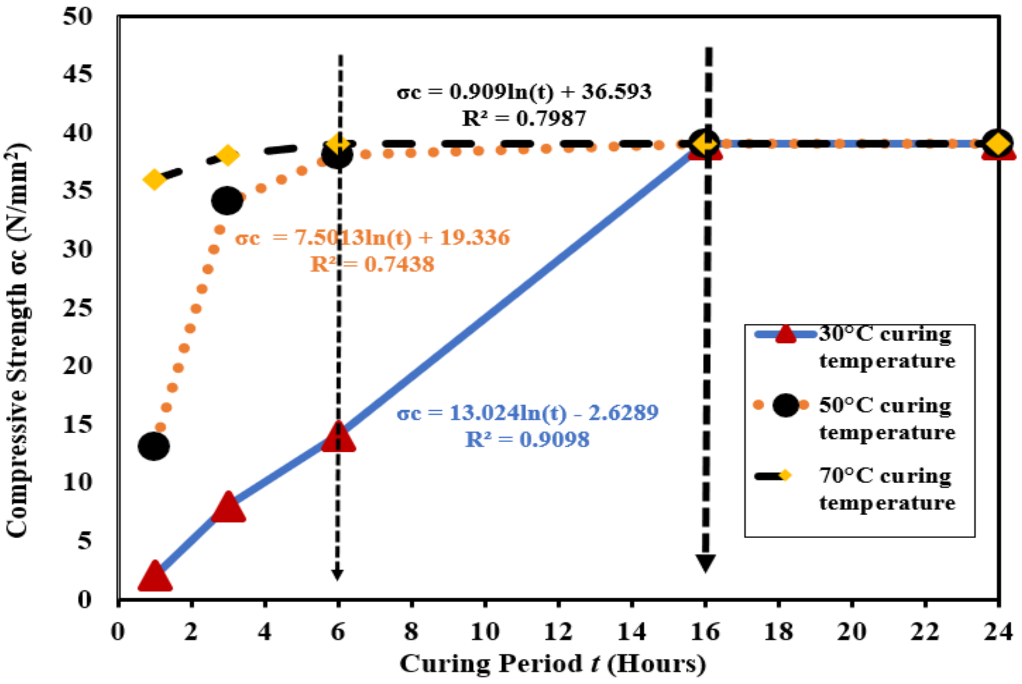


Figure 4.4: Compressive strength of PC without filler at varying curing period with different curing temperature of 30^{o}C, 50^{o}C and 70^{o}C.

PC exposed to curing temperature of 50^{o}C and 70^{o}C showed that a complete cross-linking occurred in it within the first 6 and 3 hours respectively. At an initial curing period of one hour, the compressive strength for both curing conditions were higher compared to the PC subjected to a curing temperature of 30^{o}C. This show that PC maturity is in concomitant to it exposure to high temperature, thus showing that early strength development of PC will occur when the PC is cured under high post curing temperature. Sufficient curing temperature and period result in desired maturity of PC. The maturity of PC is shown through the completeness of the cross-linking of polyester binder.

A strong relationship was exhibited between compressive strength and curing period at 30 ^{o}C, 50^{o}C and 50 ^{o}C curing temperature. All had regression, R^{2}, of more than 0.70. Therefore, the 70^{o}C curing temperature and curing period of 6 hours were chosen as the desired post-curing regime for PC due to the strong correlation demonstrated.

Additionally, the formula of compressive strength, σc, and curing period, t, for all PCs are as follows:

For curing temperature of 30^{o}C:

$\sigma_c = 13.024\ln(t) - 2.629$

$R^2 = 0.7987$

For curing temperature of 50^{o}C:

$\sigma_c = 7.501\ln(t) + 19.336$

$R^2 = 0.7438$

For curing temperature of 70^{o}C:

$\sigma_c = 0.909\ln(t) + 36.593$

$R^2 = 0.9098$

4.2.2 Optimum desired mix proportion of polymer concrete

In this study, the optimum mix proportion of PC was determined according to two conditions - flowability and compressive strength. These two variables were decided given that PC was employed with low binder content of 11%. Therefore, both variables are important to produce PC with micro-filler.

4.2.2.1 Flowability

Table 4.4 present the flow spread diameter of blended polymer (polyester resin incorporating only fillers) as obtained in the flowability test. The table presents the flow spread diameter of blended polymer incorporating 11% binder content of SHA and

$CaCO_3$ fillers, respectively. Generally, the overall flow spread diameter contracted when the filler content increased. From the tables, higher flow spread diameter indicates higher flow and filling abilities for all blended polymer and vice versa. The discussion at this point of investigation reflects that great flowability and filling ability give great workability of PC. The overall flowability of various blended polymers gradually decreased when filler content increased. However, the blended polymers incorporating SHA showed slight reduction in flow spread diameter. This was proved by their differences between flow spread diameter of blended polymer with and without filler content. The overall differences of flow spread diameter with and without filler were less than 15% for all blended polymers as illustrated in Table 4.4. Also, the blended polymers incorporating calcium carbonate had immense reduction in flow spread diameter as illustrated in Table 4.4. From the table, the differences of flow spread diameter (with and without filler) for the blended polymer mentioned was more than 15%. This situation gives insight that SHA is a great filler in polymer resin in terms of flowability and filling ability. Interestingly, due to the lightness and fineness of SHA, this ash can also provide greater workability. Thus, the SHA in this study became an actively-mobilized filler in PC compared to $CaCO_3$ fillers. Immense reduction of flow spread diameter for $CaCO_3$ blend polymer is due to high density as well as heaviness of calcium carbonate filler. The heaviness of filler led to inactive movement as compared to SHA. For instance, even though calcium carbonate filler had a comparable fineness to SHA (refer Figure 4.2 and Table 4.3), the mobilized character in this blended polymer was different. Similar findings were also observed by similar studies done by Hafizah (2015) and Bignozzi *et al.* (2000).

Table 4.5: Percentage difference of flow spread diameter of blended polymer (with and without filler) at different filler content

Filler content (%)	Polymer concrete incorporating SHA 11% Binder				Polymer concrete incorporating $CaCO_3$ 11% Binder			
	***FSD (mm)**	***% Diff**	**SD (mm)**	**COV (%)**	***FSD (mm)**	***% Diff**	**SD (mm)**	**COV (%)**
0	441.0	0.0	5.6	1.3	438.0	0.0	4.2	0.6
12	401.8	08.89	6.2	0.7	329.3	24.82	1.2	1.4
14	392.4	11.02	2.3	1.1	325.3	25.73	1.6	0.2
16	377.7	14.35	4.6	1.6	323.0	26.26	3.4	1.3

*FSD = Flow spread diameter, *% Diff = Percentage difference of FSD with and without filler, *SD = Standard deviation and COV = Coefficient of variation

The overall outcomes of flowability in this test were accepted because the standard deviation (SD) and coefficient of variation (COV) was less than 8 mm and 2.5%, respectively. Additionally, the SD and COV value of flow spread diameter is presented in Tables 4.4.

4.2.2.2 Compressive strength

Table 4.6 present the compressive strength of PC obtained from the cube compression test. The figure displays the compressive strength of PC incorporating 11% binder content of SHA and CaCO3 micro-fillers, respectively. From the figure, the compressive strength for all PCs increased as the filler content increased. This behaviour can be clearly seen in Tables 4.6. Figure 4.6 shows that PC-SHA (14% of filler content) has superior compressive strength as compared to PC-CaCO3. This is believed to be caused by the high fineness of fillers which has tremendously increased the surface area (Table 4.6). The highest compressive strength of PC-SHA was achieved with 14% filler content. The inference made was that the filler had satisfactorily filled the spaces between inert granular materials.

The density of SHA was 0.24 g/cm^3 (see Figure 4.17 and Table 4.9), and the filler was thus deemed to be a light filler. With this, the SHA could be added up to 14% filler content. This light micro-filler of SHA is able to 'actively-mobilize' its filling ability, and then increases the compressive strength of PC as discussed in sub-section 4.1.2.1. Higher filing ability of PC-SHA resulted in a higher compressive strength of PC. The desired compressive strength PC-SHA was achieved at 14% filler content, while the desired compressive strength for PC-CaCO3, was achieved at 12% filler content. These are shown in Table 4.5 where the desired compressive strength is highlighted (bolded and underlined to the number). At this point of investigation, the desired maximum compressive strength was obtained. The complete results of compressive strength in this test were accepted since the standard deviation (SD) and coefficient of variation (COV) was less than 3 mm and 10%, respectively as illustrated in Table 4.5.

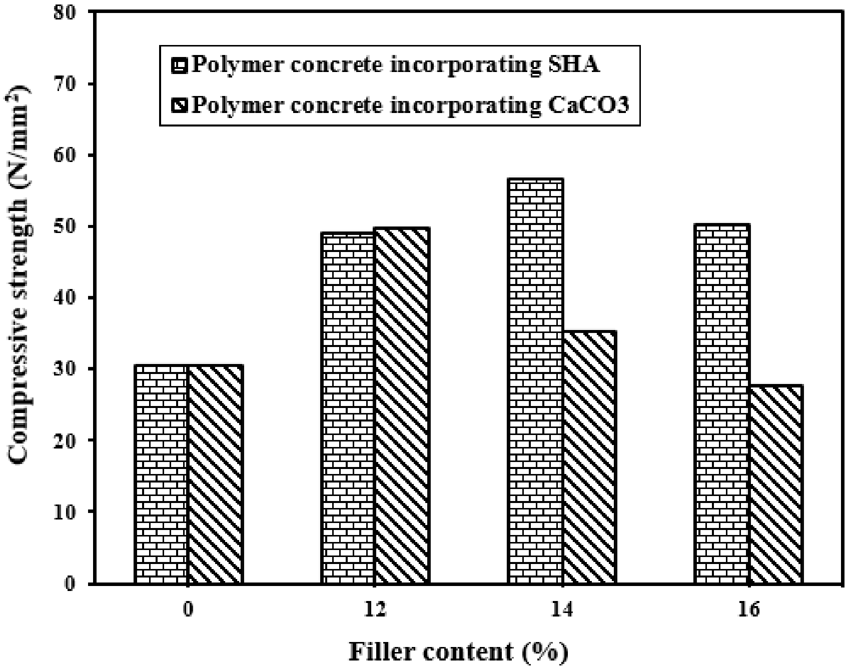


Figure 4.5. Compressive strength of Polymer Concrete containing SHA and $CaCO_3$ at 11% binder content.

Table 4.6: Percentage different of compressive strength, σc of PC (with and without filler) at different filler content

Filler content (%)	Polymer concrete incorporating SHA 11% Binder				Polymer concrete incorporating $CaCO_3$ 11% Binder			
	σc (N/mm^3)	***% Diff**	**SD (mm)**	**COV (%)**	**σc (N/mm^3)**	***% Diff**	**SD (mm)**	**COV (%)**
0	30.40	0.0	2.3	6.6	30.40	0.0	2.3	8.2
12	49.03	61.3	2.7	4.9	**49.80**	63.82	2.4	3.5
14	**56.60**	86.2	2.4	6.4	35.20	15.79	2.0	6.3
16	50.10	64.80	2.6	8.1	27.78	8.62	2.1	5.8

4.3 Physical Properties of Polymer Concrete

Physical properties of PC were obtained under non-destructive tests; namely density and water absorption.

4.3.1 Density of polymer concrete

Density is explained as the weight for every unit volume of a material together with the void innate in the tested material. Figures 4.6 showed the density of PC incorporating SHA and $CaCO_3$ micro-fillers, respectively.

PC-SHA had approximately similar density with the control PC, with and without filler, in fact. PC-$CaCO_3$ had higher density than control PC. In this study, two factors affected the density of PC: density of filler incorporated and fineness of filler. The higher the density of filler, the higher the PC density and vice versa. Meanwhile, finer filler produces denser PC. However, it was noted that all PC-SHA had lower density than normal cement concrete (2311 kg/m^3).

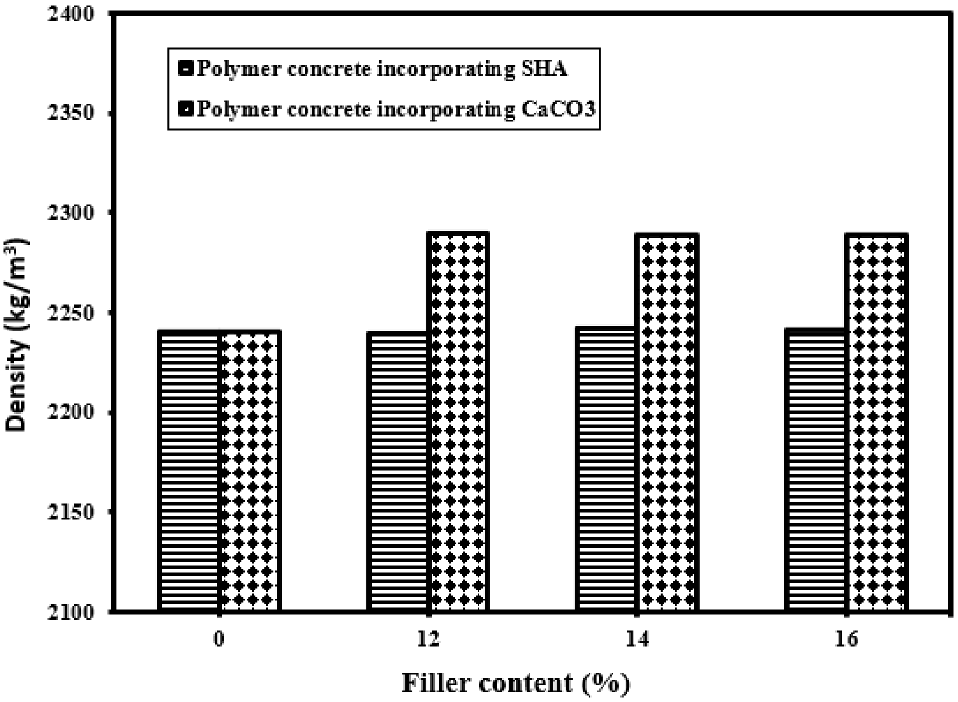


Figure 4.6 Average density of PC incorporating fine micro filler

4.3.2 Water absorption of pc

The water absorption of PC was investigated to understand the effect of pore formation on water absorption based on filler content as shown in Figures 4.7 to 4.10. From the results, it was found that the density of PC increased due to increase in water absorption of PC, and this also occurred due to the increase effect of the filler content. Water absorption of PC incorporating SHA micro-filler at 14% and 16% increased up to 7 days and remained constant thereafter at 14 days to 28 days. Nevertheless, it demonstrated a strong correlation between fine fillers and the water absorption capability of PC. The result showed that the PC-SHA has a lower water absorption compared to the PC-$CaCO_3$.

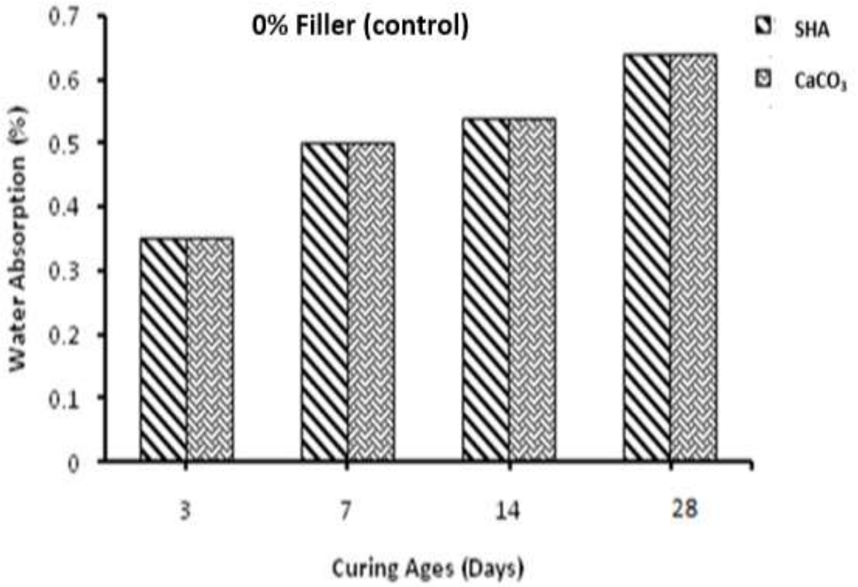


Figure 4.7: Water Absorption of PCs with 0% filler content

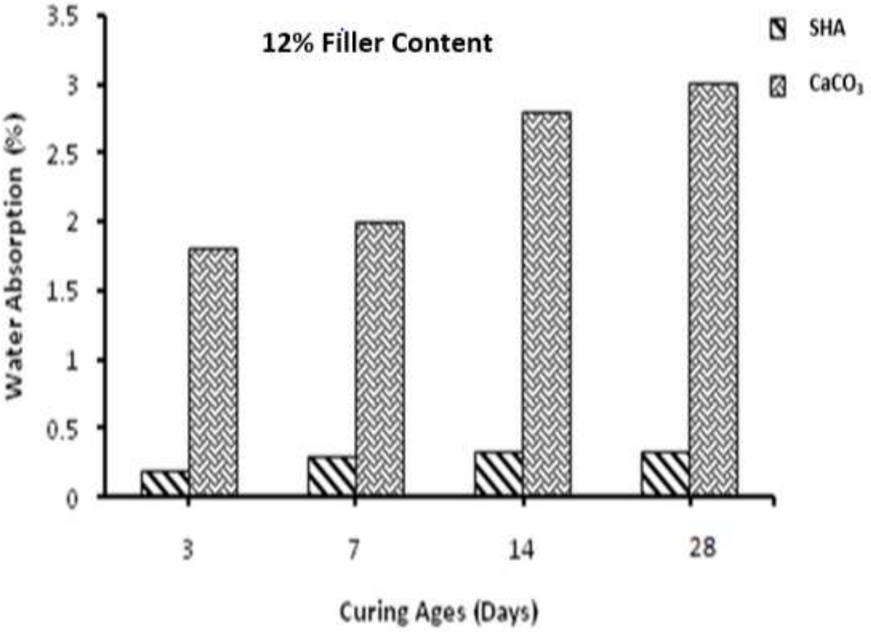


Figure 4.8: Water Absorption of PCs at 12% Filler Content

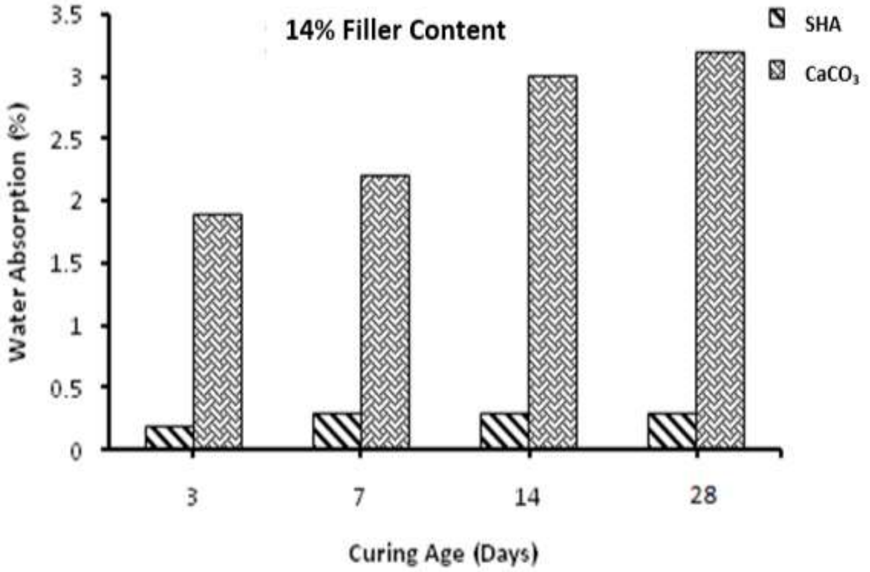


Figure 4.9: Water Absorption of PCs at 14% Filler Content

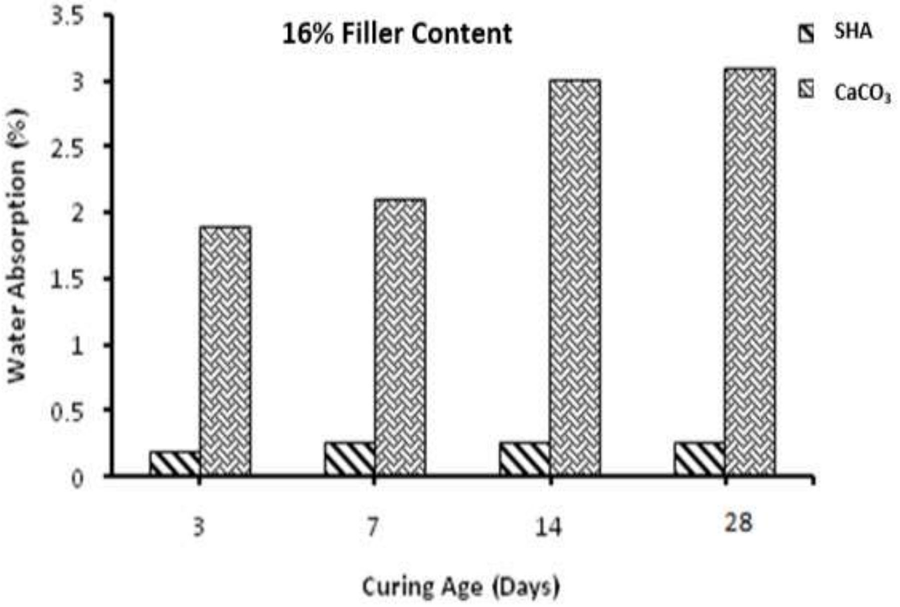


Figure 4.10: Water Absorption of PCs at 16% Filler Content

Incorporating SHA filler in PC significantly reduces the percentage of water absorption. It was found from the study that percentage of water absorption on PC incorporating SHA filler content had obviously reduced more than unfilled PC. It was revealed from the test that water absorption had decreased significantly when the SHA filler content was increased. This happened because the SHA micro-filler had filled all gaps between aggregates effectively and displaces air pockets (Figure 4.8 to 4.10). Another reason was that the SHA particle itself has small water absorption property due to its pozzolanic characteristic.

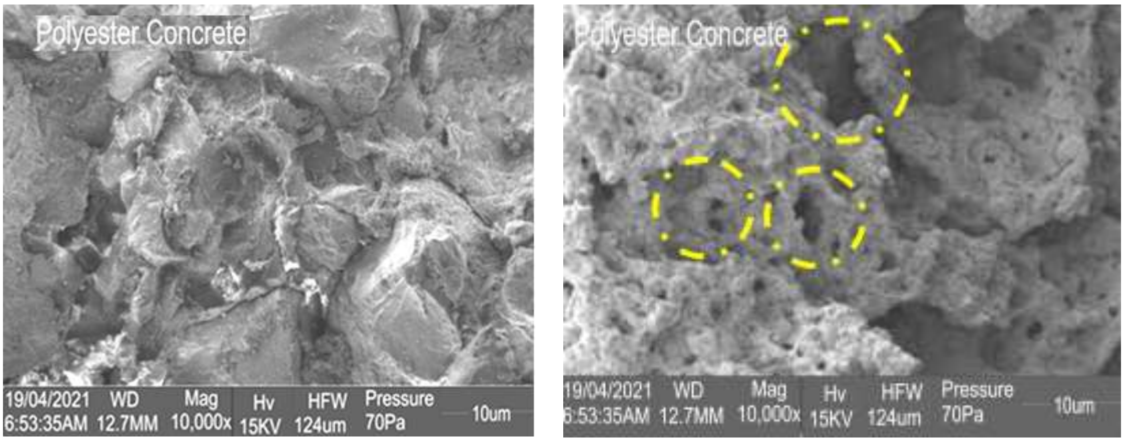
From Table 4.6, concrete quality of PC-SHA can be classified as satisfactory as the absorption rate falls below 1.0 while the PC-CCW could be classified as unsatisfactory as it falls above 1.5 referring to Khalid *et al.*, 2015 assessment of water absorption of PC table.

Table 4.7: Assessment of water absorption of PC (Khalid *et al.*, 2015)

Absorption (%)	Absorption rating	Concrete quality
<1.0	Low	Good
1.0 to 1.5	Average	Average
>1.5	High	Poor

4.4 Morphology Image

The PC's morphology image was captured under 10000 times magnifications as shown in Figure 4.11. PC-SHA was expected to have high compressive strength like PC-$CaCO_3$, evident through the densely packed structure. However, Figure 4.12(a) showed that the SHA filler was dispersed very well and the densely packed structure was obvious but the $CaCO_3$ filler in the PC- $CaCO_3$ had reduced performance when compared to PC-SHA due to the poor workability due to it particle size, thus leading to poor bonding between the materials and few voids experience in the PC as shown in Figure 4.11(b) (marked with yellow circle in the figure). In conclusion, SHA had demonstrated its high potential in becoming PC micro-filler since it could give high compressive strength and low porosity after significant physical modification.



(a) (b)

Plate X: Micro graph of PC incorporating micro-filler (a) PC-SHA (b) PC-$CaCO_3$

4.5 Summary of Findings

The following inference are made on the characterization of raw polymer binder and filler and properties of polymer concrete.

4.5.1 Characterizations of polymer binder

i. 0.6% of inhibitor additive of methyl methacrylate (MMA) was able to prolong the duration of fresh working life of polyester resin for about 15 minutes in ambient temperature.

ii. Inhibitor additive of MMA did not interrupt the curing process, even though the fresh working life of polyester binder had been prolonged. Once the polymers had been cross-linked, it would provide sufficient hardness.

4.5.2 Characterizations of filler

i. The average density for SHA and $CaCO_3$ were about $0.24 g/cm^3$ and $0.76 g/cm^3$ respectively. SHA was the lighter material.

ii. SHA and CCW can be classified as fine micro-filler. The fineness of SHA was significantly improved after physical modification. SHA was finer than $CaCO_3$.

iv. SHA could withstand high temperature and it was therefore inferred that the post-curing process would not affect SHA’s characteristics.

4.5.3 Properties of polymer concrete

This sub-section concludes the findings on the subject matter as follows:

i. Incorporating filler in PC significantly affected the apparent density of PC, where the apparent density of PC increased with lower filler content. The two factors affecting apparent density of PC are density and fineness of filler.

ii. PC-SHA had superior compressive strength to PC- $CaCO_3$.

iii. Increasing the filler content of SHA in PC shows significant increase in its compressive strength. Highest compressive strength of PC-SHA was achieved at 16% filler content.

iv. PC-SHA has a lower water absorption rate than PC- $CaCO_3$. PC-SHA also has a lower water absorption rate than plain cement concrete.

v. The PC-SHA is rated as ‘good concrete’ as shown in Table 4.6.

CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

The conclusion drawn from this study to fulfill the research objectives are as follows:

Methyl methacrylate (MMA) can be used as effective inhibitor additive in polyester resin in ambient temperature. Adding up to 0.6% of MMA effectively prolonged the working life without interrupting the curing process of polyester resin.

SHA has great filler characteristics after being ground. The modified SHA was called ground SHA and classified as fine filler. The filler could withstand thermal degradation between the range of 800 and 100^0C. SHA has a lighter density ($0.24g/cm^3$) than $CaCO_3$ ($0.76g/cm^{3}$) filler. SHA has a specific gravity of 2.32 while $CaCO_3$has a specific gravity of 2.70. SHA efficiency as a fine micro-filler in PC is promising.

High strength PC was produced with low binder content of about 11% polyester resin with sufficient workability. PC-SHA achieved optimum compressive strength at 16% filler content, while PC-$CaCO_3$ has a poor compressive strength at 12, 14 and 16% filler contents. Poor internal curing of PC- $CaCO_3$ was observed after destructive test. Investigation at this stage prove that PC-SHA cures faster than PC- $CaCO_3$.

5.2 Recommendations

The following are the recommendations for future studies:

i. Polymer inhibitor additive of MMA could be increased at higher percentage up to 1% until meeting the optimum polymer formulation with respect to working life.

ii. Long term durability study of PC-SHA under various aggressive chemical environments can be a potential research outcome to be sought after.

iii. Evaluation of the influence of fine and coarse micro-filler (Natural/agricultural byproduct and Manmade/industrial base micro-filler) on bonding behaviour of polymer concrete to normal concrete substrate

5.3 Contribution to Contribution

The study revealed the characteristics of PC-SHA with optimum mix proportion at low binder (11%) and different filler contents (12%, 14%, & 16%). PC-SHA gave the highest compressive strength (56.6 N/mm^2) at 14% filler contents and least water absorption of 0.26% at 16% filler contents after 28 days as compared to compressive strength valve of PC-CaCo$_3$ (49.8 N/mm^2) and water absorption of 3.1% at 16% filler contents after 28 days.

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APPENDICES

APPENDIX A

MIX DESIGN OF DRY AND WET MIXES FOR CUBE COMPRESSION, DENSITY AND WATER ABSORPTION TESTS

APPENDIX A1: DRY MIX DESIGN

i. COARSE AGGREGATES AT 30% CONTENT

Volume of mould = 0.1m x 0.1m x 0.1m = 0.001m^3

Specific Gravity (S.G) for course aggregate = 2.5

- Mass = S.G X volume of mould

2.5 X 0.001 = 0.0025 X 30%

= 0.00075 X 1000

= 0.75kg

ii. FINE AGGREGATES

Volume of mould = 0.1m X 0.1m X 0.1m = 0.001m^3

Specific gravity (S.G) for fine aggregates = 2.64

Mass = S.G X volume of mould

- AT 46% CONTENT

2.64 X 0.001 = 0.00264 X 46%

= 0.0012144 X 1000 = 1.2144kg

- AT 44% CONTENT

0.00264 X 44%

= 0.0011616 X 1000 = 1.1616kg

- AT 42% CONTENT

0.00264 X 42%

= 0.0011088 X 1000 = 1.1088kg

APPENDIX A2: WET MIX DESIGN

i. POLYESTER RESIN AT 11% CONTENT

Volume of mould = 0.1m X 0.1m X 0.1m = 0.001m^3

Specific Gravity (S.G) for polyester resin = 11.1

Mass = S.G X Volume of mould

- ➢ 11.1 X 0.001 = 0.0111 X 11%

0.001221 X 1000 = 1.221kg

ii. COBALT NAPHTHENATE (CoNp) AT 0.5% RESIN MASS

- ➢ 0.5% x 1221 = 6.105kg or 6.105ml

iii. METHYL ETHYL KETONE PEROXIDE (MEKP) AT 1% RESIN MASS

- ➢ 1% X 1221 = 12.21g or 12.21ml

iv. METHYL METHACRYLATE (MMA) AT 0.6% POLYESTER RESIN MASS

- ➢ 0.6% X 1221 = 7.33g or 7.33ml

APPENDIX B

Table1: Working life of Polyester Resin

Inhibitor content (%)	0	0.1	0.2	0.3	0.4	0.6	0.8	1.00
Time (minutes)	5:18	6:73	7:39	8:41	9:45	11:28	12:05	15:01

Table 2: Specific gravity of fillers

Type of Filler	**Specific Gravity**
SHA	2.32
$CaCO_3$	2.70

Table 3: Average Density of PC

	Polymer concrete with fillers		
Filler content (%)	**SHA (kg/m^3)**	**$CaCO_3$ (kg/m^3)**	**Plain Cement Concrete (kg/m^3)**
0	2240	2240	2311
12	2239	2290	
14	2242	2289	
16	2241	2289	

Table 4: Percentage Difference of Average Density of PC

	PC-SHA		**PC-$CaCO_3$**	
Filler content (%)	Density (kg/m^3)	% Diff	Density (kg/m^3)	% Diff
0	2195	0.00	2195	0.00
12	2005	8.65	2065	5.92
14	1966	10.43	2022	7.88
16	1902	13.35	1972	10.2

% Diff = percentage difference related to 0% filler content

Table 5: Water absorption of PCs

	Filler content													
	0% filler		**12%**				**14%**				**16%**			
			SHA		**$CaCO_3$**		**SHA**		**$CaCO_3$**		**SHA**		**$CaCO_3$**	
Days	**Density (kg/m^3)**	**Rate (%)**	**Density (kg/m^3)**	**Rate (%)**	**Density (kg/m^3)**	**Rate (%)**	**Density (kg/m^3)**	**Rate (%)**	**Density (kg/m^3)**	**Rate (%)**	**Density (kg/m^3)**	**Rate (%)**	**Density (kg/m^3)**	**Rate (%)**
0	2020	0	2032	0	2089	0	2008	0	2026	0	1901	0	1974	0
3	2027	0.35	2036	0.2	2127	1.8	2012	0.2	2064	1.9	1905	0.2	2012	1.9
7	2030	0.5	2038	0.3	2131	2	2014	0.3	2070	2.2	1906	0.26	2016	2.1
14	2031	0.54	2039	0.34	2148	2.8	2014	0.3	2087	3	1907	0.26	2033	3
28	2033	0.64	2039	0.34	2151	3	2014	0.3	2090	3.2	1907	0.26	2036	3.1

Rate (%) = rate of water absorption