DEVELOPMENT AND CHARACTERIZATION OF Al-3.7%Cu-1.4%Mg ALLOY/PERIWINKLE ASH (*Turritella communis*) PARTICULATE COMPOSITES

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Declaration

I hereby declare that, this research work titled "Development and Characterization of Al-3.7%Cu-1.4%Mg Alloy/Periwinkle Shell (*Turritella communis*) Ash Particulate Composites" was carried out by me, and the results of this research were obtained by tests carried out in the laboratory and all quotations are indicated by references.

Name of Student	Signature	Date

Certification

This research work titled "Development and Characterization of Al-3.7%Cu-1.4%Mg/Periwinkle (*Turritella communis)* Shell Ash Particulate Composites" by Nwabufoh M. Nebolisa with Registration Number M.Sc/Eng/01731/2010-2011 meets the regulations guiding the Award of Master degree in Metallurgical and Materials Engineering at Ahmadu Bello University, Zaria.

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Dedication

This research work is dedicated to Almighty God, whom by His Grace made this work a success and my Family for their prayers, supports and the encouragement towards my work, and also my friends who stood by me to see that this work was a success. Thank you so much.

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The completion of this research was made possible by the Grace of the Almighty God, may His name alone be glorified. My profound gratitude to my supervisors Prof. S.B. Hassan and Dr.G. B. Nyior for his guidance through the course of this work. Thank you.

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List of Abbreviation and Symbols

MMCs - Metal matrix composites

PWAp - Periwinkle shell Ash particulate

XRF - X-ray fluorescent

XRD - X-ray diffractometry

SEM - Scanning electron microscopy

EDS - Energy dispersive spectrometer

PMCs - Polymer matrix composites

Al-Cu-Mg - Aluminium-Copper-Magnesium

ABSTRACT

The development and characterization of Al-3.7%Cu-1.4%Mg/Periwinkle shell ash particulate composites using periwinkle ash as reinforcement has been carried out. The periwinkle ash was used in the production of MMCs ranging from 5 to 30% at an interval of 5% addition, using double stir casting method. The periwinkle shells were characterized using X-ray fluorescent (XRF) that revealed CaO, SiO_2, Al_2O_3, MgO, and TiO_2 as major compounds. The microstructural analyses of the composites produced were studied using X-ray Diffractometry (XRD), Scanning Electron Microscopy (SEM). The physical properties (density and porosity) and mechanical properties such as tensile strength, impact energy and hardness values of the composites developed were determined. The microstructure obtained revealed a dark ceramic (reinforcer) and white metallic phases. The results of the mechanical properties tests revealed that, the addition of periwinkle ash increased the hardness of the composites produced for all additions from 55.45 HRF at 0wt% to 87.75HRF at 30wt% progressively. While the yield strength and ultimate tensile strength of the composites produced increased from 115.56 N/mm^2 and 153.75 N/mm^2 at 0wt% to 151.60 N/mm^2 and 202.45 N/mm^2at 25wt% PWAp respectively. Thereafter, the yield strength and ultimate tensile strength dropped to 149.75 and 190.58 at 30wt% PWAp respectively. However, Impact energy of the composites produced decreased from 10J at 0wt% PWAp to 4.80J at 30wt% PWAp. These increases in strength and hardness values of the developed composites can be attributed to the proportion and uniform distribution of the hard and brittle ceramic phases in the ductile metal matrix. The density of the developed composites decreased with wt% additions of PWA particles. These results show that improved properties of Al-Cu-Mg alloy are achievable using PWA particles as reinforcement up to a maximum of 25wt%.

CHAPTER ONE

1.0 INTRODUCTION

1.1 BACKGROUND OF THE STUDY

The development of composite materials changed the world especially in engineering fields. It is because most of composite materials have been created to show improved combination of mechanical characteristics such as stiffness, toughness and high temperature strength that can not be met by conventional materials such as ceramic, metal alloys or polymers (Callister, 1999).

Many composites used today are at the leading edge of materials technology with performance and costs appropriate to ultra-demanding applications. Heterogeneous materials combining the best aspects of dissimilar constituents have been used by nature for millions of years (Jerry, 2002).

Metal matrix composites, possess some attractive properties, when compared with their plastic counterparts. These include: (i) strength retention at higher temperatures, (ii) higher transverse strength, (iii) better electrical conductivity, (iv) superior thermal conductivity and (v) higher corrosive resistance.

Potential uses of metal matrix composites (MMCs) are numerous in industries and they include such areas of application as aerospace (propellers, tails), defense (missile-shells), automotive (drive shafts, brake discs), sports goods (golf clubs and mountain bicycle frames) and marine (yacht fittings) (Ikechukwuka, 1997; Clyne, 2001). When compared with unreinforced matrix alloy, MMCs in general have superior mechanical properties, such as high strength, high stiffness, high wear resistance and very good high temperature properties. The driving force for research and development on particulate metal composites was a desire to produce a material with desirable features, characteristics of metal matrix composites without paying the high price of particulates (Clyne, 2001)

1.2 RESEARCH PROBLEM

It is generally agreed that improvement in mechanical properties of MMCs is created by reinforcement and also the properties are improved remarkably by introducing hard intermetallic compound into the aluminum matrix. The reinforcing materials are generally SiC, Al_2O_3, TiB_2, boron and graphite in order to achieve different properties. However, they are expensive as shown in Figure 1.2 and of high density (Aigbodion, 2010).

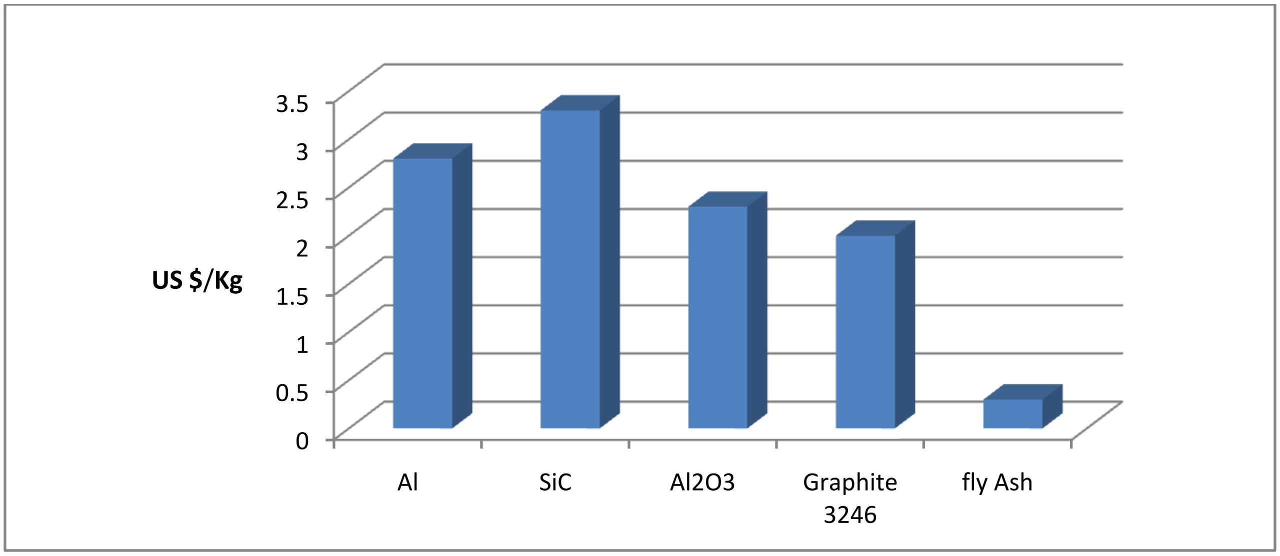


Figure 1.1: Prices of materials commonly used in making MMCs (Aigbodion, 2010).

The ever increasing demand for low cost reinforcement stimulated the interest towards production and utilization of by-products from industry as reinforcement due to their low densities, low cost, biodegradability and are naturally renewable at affordable cost. Aigbodion

(2007b) have used Kankara clay to reinforce Al-Si alloy, Aigbodion (2010) used bagasse ash as reinforcement in Al-Cu-Mg composite. Bienia *et. al.* (2003) used fly ash in reinforcement of aluminum matrix and Muali *et. al.* (1982) used vortex method in the production of aluminum alloy matrix composites reinforced with coconut shell char particles. They all reported good dispersion of the particles in the composites castings. It is in this light that the present research work has been undertaken with the objective to explore the use of periwinkle ash as a reinforcing material in Al-Cu-Mg alloy composites.

1.3 SIGNIFICANCE OF THE STUDY

Through this work, the use of periwinkle shell ash, an Agro waste as, a reinforcement to enhance the properties of Al-3.7%Cu-1.4%Mg alloy composite for automotive application (drake drums) will be achieved.

Furthermore, the work will enhance conversion of periwinkle shell as an agricultural waste into wealth and thereby reduce the environmental pollution caused by this waste and also influence positively the economy of the country.

1.4 AIM AND OBJECTIVES OF THE STUDY

The major aim of this research is to explore the possibility of using periwinkle shell ash as a reinforcing material in Al-3.7%Cu-1.4%Mg alloy composites while the objectives are;

i. To produce periwinkle shell particles in both unashed and ashed form.
ii. To determine the composition of periwinkle shell ash using XRD method.
iii. To determine mechanical and physical properties as well as microstructure of the Al-Cu-Mg/ PWAp composites produced.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 COMPOSITES

A composite is a material composed of two different materials bounded together with one serving as the matrix surrounding fibres or particles of the other. The fibers may be continuous throughout the matrix or short fibres and either aligned in all the same direction or randomly arranged (Mathew and Bill, 2002).

A composite is said to be a multifunctional material system that provide characteristics not obtainable from any discrete material. They are cohesive structures made by physically combining two or more compatible materials, different in composition and characteristics and sometimes in form (Kelly, 1975).

Suchetclan .V (1972) explained composite materials as heterogeneous materials consisting of two or more solid phases, which are in intimate contact with each other on a microscopic scale. They can also be considered as homogeneous materials on a microscope scale in the sense that any portion of it will have the same physical property. Matrix composites are classified into the following metal matrix composite, polymer matrix composite, ceramic matrix composite, carbon-carbon composite and hybrid composites.

In the past years, metal matrix composite have gained prominence especially aluminum metal matrix due to their low density. For instance, Aigbodion (2007a) explained the characteristics of this type of composite and emphasized the necessity of adapting advanced materials into commercial product. This would allow improving the material competitiveness in the automobile and aviation industries. Composites consist of one or more discontinuous phases embedded in a continuous phase. The discontinuous phases are usually harder and stronger than the continuous phases and are called the ‘reinforcement’ or ‘reinforcing material’, whereas the continuous phase is termed as the ‘matrix’. Properties of composite are strongly dependent on the properties of their constituent materials, their distribution and the interaction among them (Clyne, 2001). The composite properties may interact in a synergistic way resulting in improved or better properties.

2.2 COMPOSITE MATERIALS

A composite material is one, which is made of at least two materials working together to give material properties that are different to their own. Most composites consist of a bulk material (‘matrix’) and a reinforcement of some kind, added primarily to increase the strength and stiffness of the matrix (Asthana, 1998).

2.2.1 **Matrix Phases In Composite**

The matrix phase of particulate composites may be a metal, polymer, or ceramic. For particles-reinforced composites, the matrix phase serves several functions. First, it binds the particles together and acts as the medium by which an externally applied stress is transmitted and distributed to the particles; only a very small proportion of an applied load is sustained by the matrix phases. Furthermore, the matrix material should be ductile. In addition, the elastic modulus of the particle should be much higher than that of the matrix (Khanna, 2012). The second function of the matrix is to protect the individual particles from surface damage as a result of mechanical abrasion or chemical reactions with the environment. Such interactions may introduce surface flaws capable of forming cracks, which may lead to failure at low tensile stress levels. Finally, the matrix separates the particles and, by virtue of its relative softness and plasticity, prevents the propagation of brittle cracks from particle to particle, which could result in catastrophic failure (Khanna, 2012).

2.2.1.1 Metal matrix composites (MMCs)

MMCs are composed of metal or metal alloy base (called matrix) and a reinforcement (usually ceramic) material that is dispersed in that matrix .In the production of the composite the matrix and the reinforcement are mixed together (Metals Handbook, 1979).

In recent years, the development of metal matrix composite (MMCs) has been receiving worldwide attention on account of their superior strength and stiffness in addition to high wear resistance and creep resistance comparable to their corresponding wrought alloys. The ductile matrix permits the blunting of cracks and stress concentrations by plastic deformation and provides a material with improved fracture toughness. MMCs possess some attractive properties, when compared with organic matrices. These include the following:

(i) good strength at higher temperature;
(ii) higher transverse strength;
(iii) superior thermal conductivity; and
(iv) higher erosion resistance.

However, the major disadvantage of metal matrix composites is their densities and consequently lower specific mechanical properties compared to polymer matrix composites. Another notable difficulty is the high- energy requirement for fabrication of such composites (Callister, 1999).

2.2.1.2 Polymer matrix composites (PMCs)

A very large number of polymeric materials, both thermosetting and thermoplastic, are used as matrix materials for the production of composites. Some of the major advantages and limitations of resin matrix are shown in Table 2.1.

Table 2.1: Advantages and limitations of resin matrix (Callister, 1999).

Advantages	Limitations
1. low densities	1. low transverse strength.
2. good corrosion resistance	2. low operational temperature limit
3.low thermal conductivities	
4.low electrical conductivities	

In general, the polymer matrix are selected on the basis of adhesive strength, heat resistance, chemical and moisture resistance etc. The resin must have mechanical strength commensurate with that of the reinforcement. It must be easy to use in the fabrication selected and also stand up to the service conditions.

2.2.1.3 Ceramic matrix composites (CMCs)

CMCs are inorganic, non-metallic materials made from compounds of a metal and non-metal. Ceramic materials may be crystalline or partly crystalline. They are formed by the action of heat and subsequent cooling. Clay was one of the earliest materials used to produce ceramics, but many different ceramic materials are now used in domestic, industrial and building products. Ceramics materials tend to be strong, stiff, brittle, chemically inert and non-conductors of heat and electricity, but their properties vary widely. For example, porcelain is widely used to make electrical insulators, but some ceramic compounds are superconductors (Clyne, 2001).

Ceramic fibers, such as alumina and SiC (Silicon Carbide) are advantageous in very high temperature applications and also where environmental attack is an issue. Since ceramics have poor properties in tension and shear, most applications as reinforcement are in the particulate form (for instance zinc and calcium phosphate).

2.2.1.4 Carbon–carbon composites

One of the most advanced and promising engineering material is the carbon fiber reinforced carbon-matrix composite, often termed a **carbon–carbon composite;** as the name implies, both reinforcement and matrix are carbon. These materials are relatively new and expensive and, therefore, are not currently being utilized extensively. Their desirable properties include high-tensile moduli and tensile strengths that are retained to temperatures in excess of 2000˚C, resistance to creep, and relatively large fracture toughness values. Furthermore, carbon– carbon composites have low coefficients of thermal expansion and relatively high thermal conductivities; these characteristics, coupled with high strengths, give rise to a relatively low susceptibility to thermal shock. Their major drawback is a propensity to high-temperature oxidation. The carbon–carbon composites are employed in rocket motors, as friction materials in aircraft and high-performance automobiles, for hot-pressing molds, in components for advanced turbine engines, and as ablative shields for re-entry vehicles. The primary reason that these composite materials are so expensive is the relatively complex processing techniques that are employed (Callister, 1999).

2.2.1.5 Hybrid composites

A relatively new fiber-reinforced composite is the **hybrid,** which is obtained by using two or more different kinds of fibers in a single matrix; hybrids have a better all-around combination of properties than composites containing only a single fiber type. A variety of fiber combinations and matrix materials are used, but in the most common system, both carbon and glass fibers are incorporated into a polymeric resin. The carbon fibers are strong and relatively stiff and provide a low-density reinforcement; however, they are expensive. Glass fibers are inexpensive and lack the stiffness of carbon. The glass–carbon hybrid is stronger and tougher, has a higher impact resistance, and may be produced at a lower cost than either of the comparable all-carbon or all-glass reinforced plastics. There are a number of ways in which the two different fibers may be combined, which will ultimately affect the overall properties (Callister, 1999).

2.2.2 Reinforcement

The purpose of the reinforcement in a composite material is to increase the mechanical properties of the neat resin system. All the different fibers used in composites have different properties and so affect the properties of the composite in different ways (Lowshenko et al, 1997). For most of the applications, the fibers need to be arranged into some form of sheet, known as a fabric, to make handling possible. There are two types of reinforcement material namely fiber and particulate. Based on the two materials, composites are classified into fiber reinforced composite and particle reinforced composites.

In order to achieve these properties, the selection depends on the type of reinforcement, its method of production and chemical compatibility with the matrix and the following aspects must be considered while selecting the reinforcement material.

(i) size – diameter and aspect ratio;
(ii) shape – chopped fiber, whisker, spherical or irregular particular, flake, etc;
(iii) surface morphology – smooth and rough;
(iv) poly – or single crystal;
(v) structural defects – voids, second phases; and
(vi) surface chemistry – e.g. sio_2 or c on sic or other residual films.

Even when a specific type has been selected, reinforcement inconsistency will persist because many of the aspect cited above in addition to contamination from processing equipment and feedstock may vary greatly (Arsenault, 1984).

2.2.2.1 Particle reinforced composites

Particle reinforced composites involve ceramic particles in a metal matrix, and are widely used for tips of cutting tools. Ceramic particles are hard but brittle and lack toughness; the metal is soft and ductile. Embedding the ceramic particles in the metal gives a material that is strong, hard and tough. Glass spheres are widely used with polymers to give a composite which is stronger and stiffer than the polymer alone (Mathew and Bill, 2002).

Microstructures of metal and ceramics composites, which show particles of one phase strewn in the other, are known as particle reinforced composites. Square, triangular and round shapes of reinforcement are known, but the dimensions of all their sides are observed to be more or less equal. The size and volume concentration of the dispersoid distinguishes it from dispersion hardened materials (Pandey *et al.*, 1991).

2.2.3 Interface

The interface is a bounding surface or zone where a discontinuity occurs, whether physical, mechanical, chemical etc. The matrix material must “wet” the particle. Coupling agents are frequently used to improve wet-ability. Well “wetted” particle increase the interface surfaces area. To obtain desirable properties in a composite, the applied load should be effectively transferred from the matrix to the particle via the interface. This means that the interface must be large and exhibit strong adhesion between particles and matrix. Failure at the interface (called debonding) may or may not be desirable (Valdez *et al.*, 2008).

2.3 METAL MATRIX COMPOSITES (MMCs)

2.3.1 Matrices used for MMCs

Metal matrices used for developing MMCs include aluminum and its alloys, titanium alloys, magnesium and its alloys, copper, cobalt, silver and nickel (Ikechukwuka, 1997). Metal matrices, aluminum and its alloys have been used extensively in the fabrication of the particulate MMCs due to their relative low cost, high strength-to-weight ratio, high toughness and excellent atmospheric corrosion resistance (Rajan *et al*, 2007).

MMC materials have a combination of different, superior properties to an unreinforced matrix which are; increased strength, higher elastic modulus, higher service temperature, improved wear resistance, high electrical and thermal conductivity, low coefficient of thermal expansion and high vacuum environmental resistance. These properties can be attained with the proper choice of matrix and reinforcement

Composite materials consist of matrix and reinforcement. Its main function is to transfer and distribute the load to the reinforcement or particles. This transfer of load depends on the bonding which depends on the type of matrix and reinforcement and the fabrication technique.

The matrix can be selected on the basis of oxidation and corrosion resistance or other properties (Taya and Arsenault, 1989). Generally Al, Ti, Mg, Ni, Cu, Pb, Fe, Ag, Zn, Sn and Si are used as the matrix material, but Al, Ti, Mg are used widely. It is because of their unique combination of good corrosion resistance, low density and excellent mechanical properties (Trumper, 1987). The unique thermal properties of aluminum composites such as metallic conductivity with coefficient of expansion that can be tailored down to zero, add to their prospects in aerospace and avionics. Titanium (Baxter, 1992) has been used in aero engines mainly for compressor blades and discs due to its higher elevated temperature resistance properly. Magnesium is the potential material to fabricate composite for making reciprocating components in motors and for pistons, gudgeon pins and spring caps (Lioyd, 1994). It is also used in aerospace due to its low coefficient of thermal expansion and high stiffness properties combined with low density. The choice of Silicon Carbide as the reinforcement in aluminum composite is primarily use for missile guidance system which is replacement to beryllium components because structural performance is better without special handling in fabrication demanded by latter's toxicity (Demeis, 1989). Recently aluminium-lithium alloy has been attracting the attention of researches due to its good wettability characteristics (Huda et al., 1993)

Magnesium and magnesium alloys are among the lightest materials for practical use as the matrix phase in metal matrix composites, when compared to other currently available structural materials. Magnesium is very attractive because of its unique combination of low density and excellent machinability. However, it has been reported by several authors (Kim et al., 1990, Hack et al., 1984) that though their low density (35% lower than that of Al) makes them competitive in terms of strength/density values. Magnesium alloys do not compare favorably with aluminium alloys in terms of absolute strength.

The reason for aluminium being a success over magnesium is said to be mainly due to the design flexibility, good wettability and strong bonding at the interface.

2.3.2 Reinforcements used in MMCs

The materials usually used as reinforcement in MMCs include carbides (SiC, TiC, B_4C), oxides (Al_2O_3, SiO_2), nitrides (SiN_4, AlN) and single element materials (C, Si) (Aigbodion, 2007a). These reinforcements are usually in form of continuous fibers, short fibers, and particles. A continuous fiber has an aspect (length-to-width) ratio> 1000, while a short fiber and a particle have aspect ratio of 10-1000 and 1-4, respectively(Clyne,2001).

Table 2.2: Typical Properties of discontinuous reinforcements for Alumiuium and Magnesium (Clyne, 2001).

Reinforcement	Al_2O_3	SiC particle	Al_2O_3 particle
Crystal structure	α –Al_2O_3	Hexagonal	Hexagonal
Density(gcm^{-3})	3.3	3.2	3.9
Average diameter(µm)	3.0	Variable	Variable
Length (µm)	150	-	-
Mohs hardness	7.0	9.7	9.0
Strength(Mpa)	2000	-	-
Young's Modulus(Gpa)	300	200-300	380

The production, processing and type of application of various reinforcements depend on the production technique for the composite materials (Aigbodion, 2010).

2.3.3 Application of MMCs

MMCs have a proven track record as successful "high-tech" materials in a range of applications. MMCs utilizations provide significant benefits including performance benefits (component lifetime, improved productivity), economic benefits (energy savings or lower maintenance cost) and environmental benefits (lower noise levels and fewer air-borne emissions). Engineering viability of MMCs in a number of applications has been well documented. MMCs having different types of reinforcements (whiskers, particles, short fibers and continuous fibers) have been produced both by solid state and liquid state processing (Surappa and Rohatgi, 1981, Ikechukwuka, 1997).

Brake rotors for German high-speed train ICE-1 and ICE-2 was developed by knorrBremse AG and made from a particulate reinforced aluminum alloy (AlSiMg+SiC particulates) supplied by Duralcan. Compared to conventional parts made out of cast iron with 120 kg/piece, the 76 kg of the MMCs rotor offers attractive weight saving potential. The braking system (discs, drums, calipers or back-plate) of the New Lupo from Volkswagen were made from particulate reinforced aluminum alloy supplied by Duralcan (Surappa and Rohatgi, 1981).

MMCs continuous fiber reinforced pushrods have been produced by Duralcan for racing engines. These pushrods weigh 40 % as much as steel, they are stronger and stiffer, and have high vibration damping. MMCs wires have also been developed by Duralcan for the core of electrical conductors (Surappa and Rohatgi, 1981). The unique properties of this type of conductor offer substantial performance benefits when compared to the currently used steel wire reinforced conductors.

2.3.4 Production of Metal Matrix Composites

Metal matrix composite materials can be produced by many different techniques. The focus of the selection of suitable process engineering is the desired kind, quantity and distribution of the reinforcement (particles and fibers). By altering the manufacturing method, the processing and the finishing, as well as the form of the reinforcement, it is possible to obtain different characteristic profiles, although the same composition and amounts of the components are involved. The production of a suitable precursor material, the processing to a construction unit or a semi-finished material (profile) and the finishing treatment must be separated. For cost effective reasons, prototypes with dimensions close to the final product and reforming procedures are used, which can be minimized by using mechanical finishing of the construction units (Ikechukwuka, 1997).

2.3.5 Casting Method used for MMCs

Stir-casting techniques are currently the simplest and commercial methods of production of MMCs. This approach involves mechanical mixing of the reinforcement particulate into a molten metal bath and transferring the mixture directly to a shaped mould prior to complete solidification. In this process, the crucial thing is to create good wetting between the particulate reinforcement and the molten metal. This process has major advantage that the production costs of MMCs are very low (Naresh, 2006).

2.4 ALUMINIUM ALLOYS

Aluminium alloys are alloys in which aluminium (Al) is the predominant metal. The typical alloying elements are copper, magnesium, manganese, silicon, tin and zinc. There are two principal classifications, namely casting alloys and wrought alloys, both of which are further subdivided into the categories heat-treatable and non-heat-treatable. About 85% of aluminium is used for wrought products, for example rolled plate, foils and extrusions. Cast aluminium alloys yield cost-effective products due to the low melting point, although they generally have lower tensile strengths than wrought alloys. The most important cast aluminium alloy system is Al–Si, where the high levels of silicon (4.0–13%) contribute to give good casting characteristics. Aluminium alloys are widely used in engineering structures and components where light weight or corrosion resistance is required (polmer, 1995)

Alloys composed mostly of aluminium have been very important in aerospace manufacturing since the introduction of metal-skinned aircraft. Aluminium-magnesium alloys are both lighter than other aluminium alloys and much less flammable than alloys that contain a very high percentage of magnesium (Ahmad and Zaki, 2003).

Aluminium alloy surfaces will develop a white, protective layer of aluminium oxide if left unprotected by anodizing and/or correct painting procedures. In a wet environment, galvanic corrosion can occur when an aluminium alloy is placed in electrical contact with other metals with more negative corrosion potentials than aluminium, and an electrolyte is present that allows ion exchange. Referred to as dissimilar-metal corrosion, this process can occur as exfoliation or as intergranular corrosion. Aluminium alloys can be improperly heat treated. This causes internal element separation, and the metal then corrodes from the inside out. Aircraft mechanics deal daily with aluminium alloy corrosion (polmer, 1995).

Aluminium alloy compositions are registered with The Aluminum Association. Many organizations publish more specific standards for the manufacture of aluminium alloy, including the Society of Automotive Engineers standards organization, specifically its aerospace standards subgroups (Schwarz *et al.*, 2004) and ASTM International

2.4.1 Engineering use and Aluminium alloys properties

Aluminium alloys with a wide range of properties are used in engineering structures. Alloy systems are classified by a number system (ANSI) or by names indicating their main alloying constituents (DIN and ISO). Selecting the right alloy for a given application entails considerations of its tensile strength, density, ductility, formability, workability, weldability, and corrosion resistance, to name a few. A brief historical overview of alloys and manufacturing technologies (Sanders, 2001) Aluminium alloys are used extensively in aircraft due to their high strength-to-weight ratio. On the other hand, pure aluminium metal is much too soft for such uses, and it does not have the high tensile strength that is needed for airplanes and helicopters

2.4.2 Aluminium alloys versus types of steel

Aluminium alloys typically have an elastic modulus of about 70 GPa, which is about one-third of the elastic modulus of most kinds of steel and steel alloys. Therefore, for a given load, a component or unit made of an aluminium alloy will experience a greater deformation in the elastic regime than a steel part of identical size and shape. Though there are aluminium alloys with somewhat-higher tensile strengths than the commonly used kinds of steel, simply replacing a steel part with an aluminium alloy might lead to problems.

With completely new metal products, the design choices are often governed by the choice of manufacturing technology. Extrusions are particularly important in this regard, owing to the ease with which aluminium alloys, particularly the Al–Mg–Si series, can be extruded to form complex profiles.

In general, stiffer and lighter designs can be achieved with aluminium alloys than is feasible with steels. For instance, consider the bending of a thin-walled tube: the second moment of area is inversely related to the stress in the tube wall, i.e. stresses are lower for larger values. The second moment of area is proportional to the cube of the radius times the wall thickness, thus increasing the radius (and weight) by 26% will lead to a halving of the wall stress. For this reason, bicycle frames made of aluminium alloys make use of larger tube diameters than steel or titanium in order to yield the desired stiffness and strength. In automotive engineering, cars made of aluminium alloys employ space frames made of extruded profiles to ensure rigidity. This represents a radical change from the common approach for current steel car design, which depends on the body shells for stiffness, known as unibody design (Ahmad and Zaki 2003).

Aluminium alloys are widely used in automotive engines, particularly in cylinder blocks and crankcases due to the weight savings that are possible. Since aluminium alloys are susceptible to warping at elevated temperatures, the cooling system of such engines is critical. Manufacturing techniques and metallurgical advancements have also been instrumental for the successful application in automotive engines. In the 1960s, the aluminium cylinder heads of the Corvair earned a reputation for failure and stripping of threads, which is not seen in current aluminium cylinder heads (Kaufman and John, 2000).

An important structural limitation of aluminium alloys is their lower fatigue strength compared to steel. In controlled laboratory conditions, steels display a fatigue limit, which is the stress amplitude below which no failures occur – the metal does not continue to weaken with extended stress cycles. Aluminium alloys do not have this lower fatigue limit and will continue to weaken with continued stress cycles. Aluminium alloys are therefore sparsely used in parts that require high fatigue strength in the high cycle regime (more than 10^7 stress cycles) (Naresh, 2006).

2.4.3 Heat sensitivity considerations

Often, the metal's sensitivity to heat must also be considered. Even a relatively routine workshop procedure involving heating is complicated by the fact that aluminium, unlike steel, will melt without first glowing red. Forming operations where a blow torch is used can reverse or remove heat treating, therefore is not advised whatsoever. No visual signs reveal how the material is internally damaged. Much like welding heat treated, high strength link chain, all strength is now lost by heat of the torch. The chain is dangerous and must be discarded (Surappa and Rohatgi, 1981).

Aluminium also is subject to internal stresses and strains when it is overheated; the tendency of the metal to creep under these stresses tends to result in delayed distortions. For example, the warping or cracking of overheated aluminium automobile cylinder heads is commonly observed, sometimes years later, as is the tendency of improperly welded aluminium bicycle frames to gradually twist out of alignment from the stresses of the welding process. Thus, the aerospace industry avoids heat altogether by joining parts with rivets of like metal composition, other fasteners, or adhesives (Vogel *et al.*, 1986).

Stresses in overheated aluminium can be relieved by heat-treating the parts in an oven and gradually cooling it—in effect annealing the stresses. Yet these parts may still become distorted, so that heat-treating of welded bicycle frames, for instance, can result in a significant fraction becoming misaligned. If the misalignment is not too severe, the cooled parts may be bent into alignment. If the frame is properly designed for rigidity that bending will require enormous force.

Aluminum's intolerance to high temperatures has not precluded its use in rocketry; even for use in constructing combustion chambers where gases can reach 3500 K. The Agena upper stage engine used a regeneratively cooled aluminium design for some parts of the nozzle, including the thermally critical throat region; in fact the extremely high thermal conductivity of aluminium prevented the throat from reaching the melting point even under massive heat flux, resulting in a reliable, lightweight component (Hack *et al.*, 1984).

2.4.4 Household wiring

Aluminium wire has high conductivity and relatively low price compared with copper in the 1960s, aluminium was introduced at that time for household electrical wiring in North America, even though many fixtures had not been designed to accept aluminium wire. But the new use brought some problems (Ahmad and Zaki, 2003):

- The greater coefficient of thermal expansion of aluminium causes the wire to expand and contract relative to the dissimilar metal screw connection, eventually loosening the connection.
- Pure aluminium has a tendency to creep under steady sustained pressure (to a greater degree as the temperature rises), again loosening the connection.
- Galvanic corrosion from the dissimilar metals increases the electrical resistance of the connection.
- All of this resulted in overheated and loose connections, and this in turn resulted in some fires. Builders then became wary of using the wire, and many jurisdictions outlawed its use in very small sizes, in new construction. Yet newer fixtures eventually were introduced with connections designed to avoid loosening and overheating. At first they were marked "Al/Cu", but they now bear a "CO/ALR" coding.
- Another way to forestall the heating problem is to crimp the aluminium wire to a short "pigtail" of copper wire. A properly done high-pressure crimp by the proper tool is tight enough to reduce any thermal expansion of the aluminium. Today, new alloys, designs, and methods are used for aluminium wiring in combination with aluminium terminations.

2.4.5 Wrought alloys

The International Alloy Designation System is the most widely accepted naming scheme for wrought alloys. Each alloy is given a four-digit number, where the first digit indicates the major alloying elements (Ahmad and Zaki 2003).

- 1000 series are essentially pure aluminium with a minimum 99% aluminium content by weight and can be work hardened.
- 2000 series are alloyed with copper, can be precipitation hardened to strengths comparable to steel. Formerly referred to as duralumin, they were once the most common aerospace alloys, but were susceptible to stress corrosion cracking and are increasingly replaced by 7000 series in new designs.
- 3000 series are alloyed with manganese, and can be work hardened.
- 4000 series are alloyed with silicon. They are also known as silumin.
- 5000 series are alloyed with magnesium.
- 6000 series are alloyed with magnesium and silicon. They are easy to machine, are weldable, and can be precipitation hardened, but not to the high strengths that 2000 and 7000 can reach. 6061 alloy is one of the most commonly used general-purpose aluminium alloy.
- 7000 series are alloyed with zinc, and can be precipitation hardened to the highest strengths of any aluminium alloy (tensile strength up to 700 MPa for the 7068 alloy).
- 8000 series are alloyed with other elements which are not covered by other series. Aluminium-lithium alloys

2.5 APPLICATIONS OF AL ALLOY

2.5.1 Aerospace alloys

Scandium–aluminium alloy (Al-Sc alloy)

Plate 2.1: Parts of Mig–29 made from Al–Sc alloy

The addition of scandium to aluminium creates nanoscale Al_3Sc precipitates which limit the excessive grain growth that occurs in the heat-affected zone of welded aluminium components. This has two beneficial effects: the precipitated Al_3Sc forms smaller crystals than are formed in other aluminium alloys (Kaufman and John, 2000) and the width of precipitate-free zones that normally exist at the grain boundaries of age-hardenable aluminium alloys is reduced (Ahmad, 2003). Scandium is also a potent grain refiner in cast aluminium alloys, and atom for atom, the most potent strengthener in aluminium, both as a result of grain refinement and precipitation strengthening. However, titanium alloys, which are stronger but heavier, are cheaper and much more widely used (Schwarz *et al.*, 2004)

The main application of metallic scandium by weight is in aluminium-scandium alloys for minor aerospace industry components. These alloys contain between 0.1% and 0.5% (by weight) of scandium. They were used in the Russian military aircraft Mig 21 and Mig 29 (Ahmad and Zaki 2003).

Some items of sports equipment, which rely on high performance materials, have been made with scandium-aluminium alloys, including baseball bats, (Bjerklie and Steve, 2006) lacrosse sticks, as well as bicycle[14] frames and components, and tent poles. U.S. gunmaker Smith & Wesson produces revolvers with frames composed of scandium alloy and cylinders of titanium (Shevell and Richard, 1989)

2.5.2 List of aerospace aluminium alloys

The following aluminium alloys are commonly used in aircraft and other aerospace structures (Shevell and Richard, 1989)

- 7068 aluminium, 7075 aluminium, 6061 aluminium
- Note that the term aircraft aluminium or aerospace aluminium usually refers to 7075(Wagner and Penny, 2009).
- 6063 aluminium alloys are heat treatable with moderately high strength, excellent corrosion resistance and good extrudability. They are regularly used as architectural and structural members (Kaufman and John, 2000).

The following list of aluminium alloys are currently produced used:

- 2195 aluminium – Al-Li alloy, used in Space Shuttle Super Lightweight external tank,[20] and the Space X Falcon 9 and Falcon 1e second stage launch vehicles (Bjelde *et al.*, 2007)

- 2219 aluminium – Al-Cu alloy, used in the original Space Shuttle Standard Weight external tank
- 5059 aluminium – Used in experimental rocket cryogenic tanks

2.5.3 Marine alloys

These alloys are used for boat building and shipbuilding, and other marine and salt-water sensitive shore applications (Stephen, 1993)

5052 aluminium alloy, 5059 aluminium alloy, 5083 aluminium alloy

5086 aluminium alloy, 6061 aluminium alloy, 6063 aluminium alloy

4043, 5183, 6005A, 6082 also used in marine constructions and off shore applications.

2.5.4 Automotive alloys

6111 aluminium and 2008 aluminium alloy are extensively used for external automotive body panels, with 5083 and 5754 used for inner body panels. Hoods have been manufactured from 2036, 6016, and 6111 alloys. Truck and trailer body panels have used 5456 aluminum.

Automobile frames often use 5182 aluminium or 5754 aluminium formed sheets, 6061 or 6063 extrusions.

Wheels have been cast from A356.0 aluminium or formed 5xxx sheet (Kaufman and John, 2000).

2.5.5 Al-Cu-Mg Alloy

Al-Cu-Mg Alloy is an age-hardenable aluminum alloy with copper and magnesium as the main alloying elements. It also contains other elements such as iron and nickel to produce a variety of strengthening phases in the alloy which enhance the stability of the alloy at high temperatures. It was developed for manufacturing components exposed to high temperature in automobile and aerospace industries (Metals handbook, 1979). It has been used as a matrix material for fabricating MMCs (Ikechukwuka, 1997).

Al-Cu-Mg Alloy derives its strength from a combination of precipitation and dispersion hardening (Ikechukwuka, 1997). It exhibits three aging sequences, with the stable phases being S (Al_2CuMg), θ (Al_2Cu) and (X). It also contains intermetallic phases such as AlCuNi, $(CuFe)Al_3$ and Al_9FeNi which arise from solid- state reactions between aluminum and alloying elements in Al-Cu-Mg Alloy as shown in Figure 2.1(Aigbodion, 2010). The Cu-rich phases reduce the amount of copper available for solid solution strengthening of the alloy. However, the Al_9FeNi phase forms preferentially over the copper-rich phases when iron and nickel are in the ratio of about 1:1. The presence of Al_9FeNi phases in the alloy helps to control grain size, the amount of copper available for solid solution strengthening, and impede movement of dissolutions (Ikechukwuka, 1997).

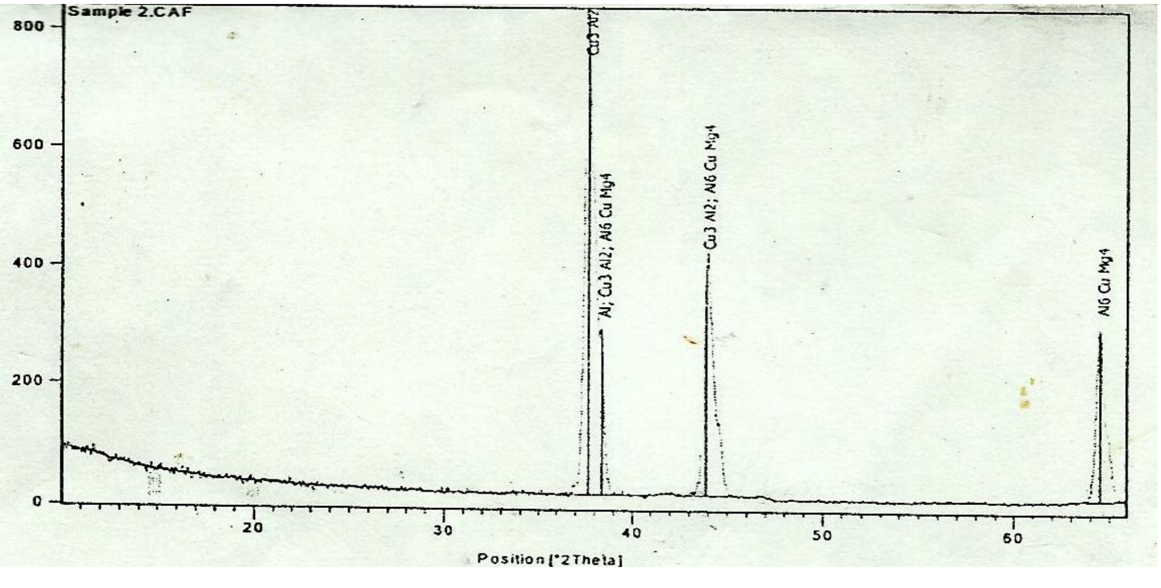


Figure 2.1: XRD pattern of the Aluminium alloy (Aigbodion, 2010)

2.5 PERIWINKLE SHELL

Periwinkle (*Turritella communis*) is a specie of sea fish which is dark and with a banded shell. The shell look fragile but they are usually difficult to break. Plate 2.1 shows a periwinkle shell

Plate 2.2: A periwinkle shell

The shell is broadly ovate, thick, sharply pointed except when eroded. The shell contains 6 to 7 whorls with some fine threads and wrinkles. The color is variable from grayish to gray-brown often with dark spiral bands. The inside of the shell has a chocolate-brown color. The width of the shell ranges from 10 to 12 mm at maturity. With an average size of 16–38 mm, shell height can reach up to 30 mm, 43 mm or 52 mm.

Periwinkle is commonly found on rocky shores in the higher and middle intertidal zone of Calabar, South-South of Nigeria. It sometimes lives in small tide pools .It may also be found in muddy habitats and can reach depths of 20,000 mm.The periwinkle in this part of Nigerian is considered a delicacy in that region because the meat is high in protein, omega 3 and low in fat and also used as bait for catching small fish. The hard shells, which are regarded as wastes ordinarily posed environmental nuisance in terms of its unpleasant odour and unsightly appearance in open-dump sites located at strategic places, are now being considered as reinforcement for Al alloy in replacement for expensive, unaffordable reinforcement.

CHAPTER THREE

3.0 MATERIALS AND METHODS

3.1 MATERIALS

The materials used were pure aluminum wire obtained from Cutix Nigeria, Limited, Oba Anambra state. Copper and Magnesium were purchased from a chemical shop at Onitsha. Plate 3.1 shows periwinkle shells.

Plate 3.1 the periwinkle shells

3.2 EQUIPMENTS

Table 3.1: Equipment used for analysis

S/No	Type of Equipment	Location of the Equipment	Manufacturer
1	Rockwell hardness machine (model 38506)	Metallurgical and materials Eng. Lab. ABU	

2	Impact testing machine	Mechanical Engineering Lab. ABU	Hoopman Ind Ltd, UK
3	Crucible furnace	Metallurgical and materials Eng. Foundry workshop ABU	Locally made furnace
4	Electrical resistance furnace	IDC , Zaria, Kaduna state	Search technology china
5	X-ray Fluorescent machine (XRF)	Chemistry depart. Lab. Unilag. Lagos state	Light house Ind. Ltd, UK
6	Grinding & Polishing machine	Metallurgical and materials Eng. Lab. ABU	John Halt Ltd, Uk
7	Digital weighing machine	Metallurgical and materials Eng. Lab. ABU	Bruce Ind., Ltd, UK
8	Automatic sieving machine	Metallurgical and materials Eng. Lab. ABU	Search tech. Ind., UK
9	X-ray diffraction machine (XRD)	Department of Chemical and Metallurgical Eng. University of Witwatersrand Johannesburg, SA	Philips Ind., Ltd UK
10	Scanning electron microscopy/Energy dispersive spectrometer (EDS)	Department of Chemical and Metallurgical Eng. University of Witwatersrand Johannesburg, SA	Oxford INCA™ Ind., Ltd UK

3.3 METHODOLOGY

3.3.1 Collection and Preparation of periwinkle shells

The periwinkle (*Turritella communis*) shells used in this work were obtained from Watt market (Ndidem Iso) in Calabar, South-South, Nigeria. Plate 3.2 shows crushed periwinkle shell. The collected shells were ground to fine powder.

Plate 3.2 Crushed Periwinkle Shells

3.3.2 Periwinkle ashing and Size analysis

The fine powder of the periwinkle shells was placed inside an electric control furnace and heated to a temperature of 1,200 ˚C for five hours to obtain a black color ash which is the periwinkle shell ash (PWAp).

The particle size analysis of the periwinkle shell ash particles was carried out in accordance with BS1377: 1990 (Hassan and Aigbodion, 2010). 100 g of the periwinkle shell ash particles were placed unto a set of sieves arranged in descending order of fineness and shaken for 15 minutes which is the recommended time to achieve complete classification. The weight retained on 150 µm was used in the research (Prasad, 2006). Plate 3.3 shows the periwinkle shells powder and periwinkle shell ash.

(a) Periwinkle Shells powder (b) Periwinkle Shells ash

Plate3.3: Periwinkle shell powder and Periwinkle shell ash.

3.3.3 Determination of Chemical Composition of Periwinkle Ash

Mini Pal compact energy dispersive X-ray spectrometer (XRF) was used for elemental analysis of the periwinkle shell ash. The system is controlled by a PC running the dedicated Mini Pal analytical software (Prasad, 2006). Plate 3.4 shows XRF machine used.

Plate 3.4: XRF machine

3.3.4 Casting of the Composites

3.3.4.1 Melts of the Alloy

The metal matrix composites that were used in this research were produced using double stir-casting method at the foundry workshop of Metallurgical and Materials Engineering Department, Ahmadu Bello University, Zaria, Nigeria. The samples were produced by keeping the percentage of copper (3.7 %) and magnesium (1.4 %) constant and varying the reinforcing material (periwinkle shell ash) particles in the range 5-30 wt% with interval of 5 wt%. The pure aluminum cable wire with the required quantity of 3.7 %Cu (ligand 50 %Cu-50 %Al) was charged into the crucible furnace. The furnace was heated to 720 ˚C for 2 hours and then 1.4 %Mg was added. The periwinkle shell ash particles (PWAp) were preheated to 1000 ˚C for 1hour before adding to melt in order to improve the wettability and harmonize the temperature (Aigbodion 2010). Plate 3.5 shows cast samples of Al-Cu-Mg/PWAp composites

Plate3.5: Cast samples of Al-Cu-Mg/PWAp casting.

3.3.4.2 Composite Production

The furnace temperature was first raised to 720 ˚C to melt the alloy completely and then cooled to the liquidus point (580 ˚C) to keep the melt in a semi-solid state. At this stage the preheated periwinkle shell ash particles were added and mixed manually. After manual mixing was done, the composite slurry was re-heated to a temperature of 720 ˚C and then manual mixing was carried out again for about 20 minutes. In the final mixing process, and then pour into a preheated sand mould with diameter 18 mm × 300 mm length was used to prepare cast bars.

3.4 X-RAY DIFFRACTION ANALYSIS

XRD analysis was done to detect the presence of different phases in the developed composite (one sample). XRD analysis was carried out using a Philips X-ray diffractometer. The X-ray diffractograms are taken using Cu Kα radiation at scan speed of 3^0/ min at the Department of Chemical and Metallurgical Eng. University of Witwatersrand Johannesburg, South Africa. The samples were rotated at precisely one-half of the angular speed of the receiving slit, so that a constant angle between the incident and reflected beams is maintained. The receiving slit is mounted in front of the counter tube arm, and behind it is usually fixed a scatter slit to ensure that the counter receives radiation only from the portion of the specimen illuminated by the primary beam. The intensity diffracted at the various angle was recorded automatically on a chart and the appropriate (θ) and (d) values were then obtained (Rajan, *et al*, 2007; Ejiofor and Reddy, 1997). Plate 3.6: shows XRD machine used.

Plate3.6: XRD machine

3.5 MICROSTRUCTURAL EXAMINATION

Four (4) samples of 10 mm each was cut off from the produced Al-Cu-Mg/PWA particulate composites and ground on grades of SiC papers (grit size 400 – 600). The rough grit paper used was 400 grit sizes and the finest surface was obtained using 600 grit size SiC paper. The surface of the sample was then mechanically polished to obtain a shining smooth minor-like surface. The microstructural constitutes of the samples were studied using a Scanning electron microscope JEOL JSM-5900LV equipped with an oxford InCA^{Tm} Energy Dispersive Spectroscopy (EDS) system at Department of Chemical and Metallurgical Eng. University of Witwatersrand Johannesburg, South Africa. The polished samples were firmly held on the sample holder using a double-sided carbon tape before putting them inside the sample chamber. The SEM was operated at an accelerating voltage of 5 to 20kV. The image was recorded (Rajan *et al*, 2007). Plate 3.7: shows SEM/EDS machine used.

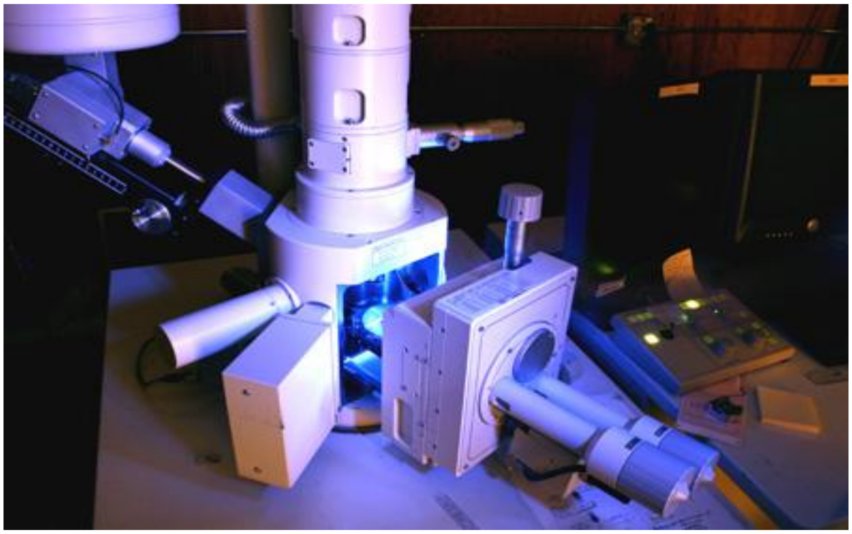


Plate 3.7: SEM/EDS machine

3.6 DETERMINATION OF PHYSICAL AND MECHANICAL PROPERTIES

3.6.1 Determination of Density

A clean seven (7) samples of Al-Cu-Mg/ periwinkle shell ash particle composite was weighed accurately in air using a laboratory balance and then suspended in water. The weight of the sample when suspended in water was determined, the volume of the sample was determined from the effect of displacement of water (Archimedean principle). The density was calculated using the following expression (Hassan and Aigbodion, 2010). Plate 3.8: shows density sample of the composite produced.

$$Density = \frac{\text{Mass}}{Volume} \text{ ------------------------------------ } (3.1)$$

Plate 3.8: shows density samples

3.6.2 Determination of Apparent Porosity

The seven (7) samples for apparent porosity (P_a) were kept in the oven at 110˚ C for 3 hours to obtain constant weight D. The specimen was then suspended in distilled water and boiled on a hot plate for 30 minutes. After boiling, while still in hot water, the water was displaced with cold water and the weight S was measured on a digital balance hinged on the tripod stand. The test specimen was removed from the water, and extra water was wiped off from the surface by lightly blotting the specimen with wet towel and the weight W of the soaked specimen suspended in air was measured. The apparent porosity of the specimen was determined using equation 3.2: (Prasad and Krishna, 2011). Plate 3.9: shows electrical resistance furnace used to determine apparent porosity of the composite produced.

$$\text{Apparent porosity} = \frac{W-D}{W-S} \times 100\ (\%) \qquad (3.2)$$

where P_a – apparent porosity;
D – constant weight;
W - weight of dried sample; and
S – weight of saturated sample.

Plate 3.9: Electrical resistance furnace

3.6.3 Determination of Hardness

The hardness values of the seven (7) samples were determined (ASTM E18-79) using the Rockwell hardness test machine located in the department of metallurgical and Materials Engineering at Ahmadu Bello University, Zaria. The sample with a 1/16 inch indenter made of steel ball. 10 mm sample was cut off using a hacksaw, ground and polished until it was smooth and flat. A Rockwell scale F, an indenter of a minor load 10 kg (fixed) was applied and a total load of 60 kg was used, the average hardness value was taken and recorded from three readings (Hassan and Aigbodion, 2010, Aigbodion, 2007). Plate 3.10 shows the Rockwell hardness machine.

Plate3.10: Rockwell hardness machine

3.6.4 Determination of Tensile Properties

The Tensile strength of the seven (7) samples were determined using Housefield tensometer tensile test machine at the department of Mechanical Engineering, Ahmadu Bello University, Zaria. The standard tensile test sample of a dumbbell shape with a diameter of 6 mm and a length 38 mm was used for the research. An initial load of 5 kN was applied on the samples, this load was increased until failure occurred. The results were read and recorded (Hassan and Aigbodion, 2010). Plate 3.11 shows the Housefield tensometer tensile machine.

Plate 3.11: Tensometer tensile machine

3.6.5 Determination of Impact Energy

The Impact energy of the seven samples were determined using the Charpy impact machine at the Department of Mechanical Engineering, Ahmadu Bello University, Zaria. Each sample was machined to a diameter of 8 mm, v-notched to 0.5 mm depth from the middle with a total length of 46 mm. This sample was gripped on the machine and a load was applied to cause breakage. The impact energy recorded gives an idea of the energy required to break the notched specimen. Plate 3.12 shows Charpy impact machine used (Aigbodion, 2007).

Plate3.12: Charpy impact machine.

CHAPTER FOUR

4.0 RESULTS

4.1 CHEMICAL COMPOSITION OF PERIWINKLE SHELL ASH BY XRF ANALYSIS

The chemical composition of Periwinkle shell ash is given in Table 4.1. The result of XRD, the Optical microscopy and SEM/EDS of the produced composites are shown in Figure 4.1 and Micrographs 4.1 to 4.4 respectively. The density and porosity of Al-3.7%Cu-1.4%Mg/PWAp containing 0 - 30% PWAp additions are shown in Figure 4.2 - 4.3 respectively. The mechanical properties of the produced composites are shown in Figures 4.4 to 4.7.

Table 4.1: Chemical composition of periwinkle shell ash particles

Constituent	%
SiO_2	32.84
Al_2O_3	10.20
Fe_2O_3	7.02
CaO	40.84
MgO	1.47
SO_3	0.26
K_2O	0.14
Na_2O	0.24
P_2O	0.01
Mn_2O_3	0.78
TiO_2	1.07

4.1.2: XRD Analysis of Al-Cu-Mg/PWAp Composite

Figure 4.1: shown XRD analysis of Al-3.7%Cu-1.4%Mg/PWA with 25 wt% periwinkle shell ash addition

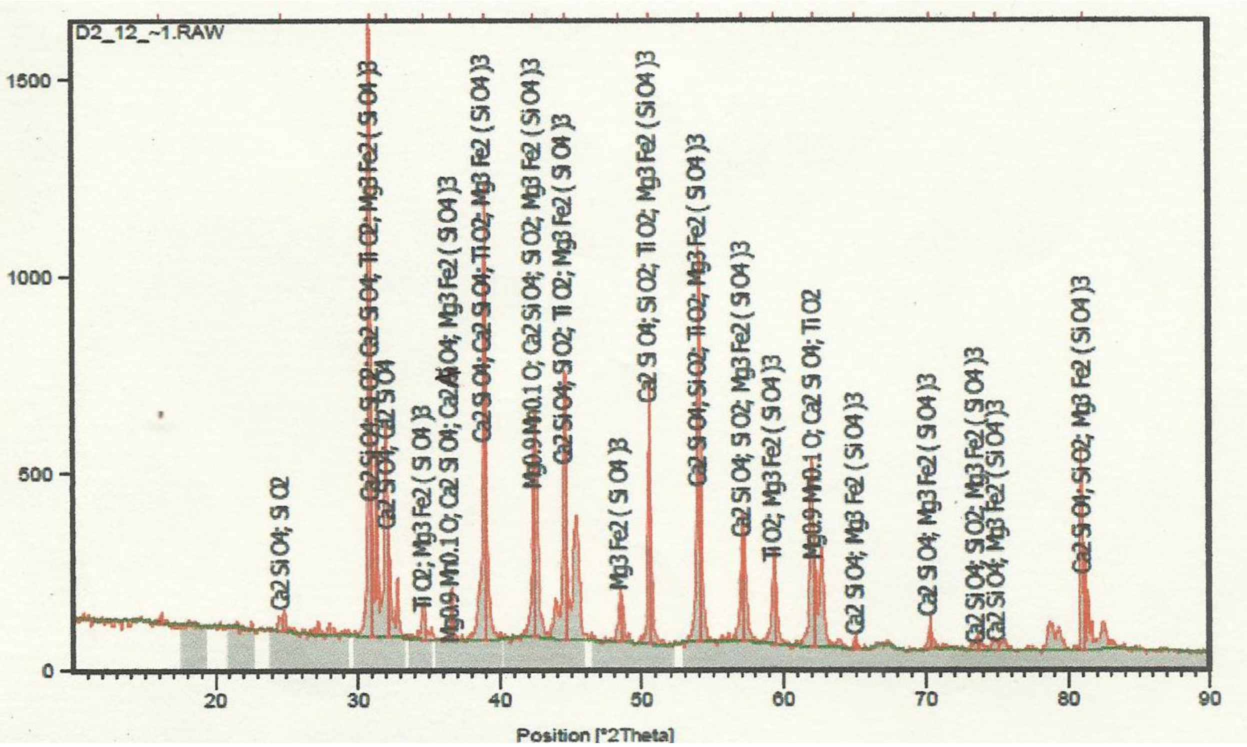
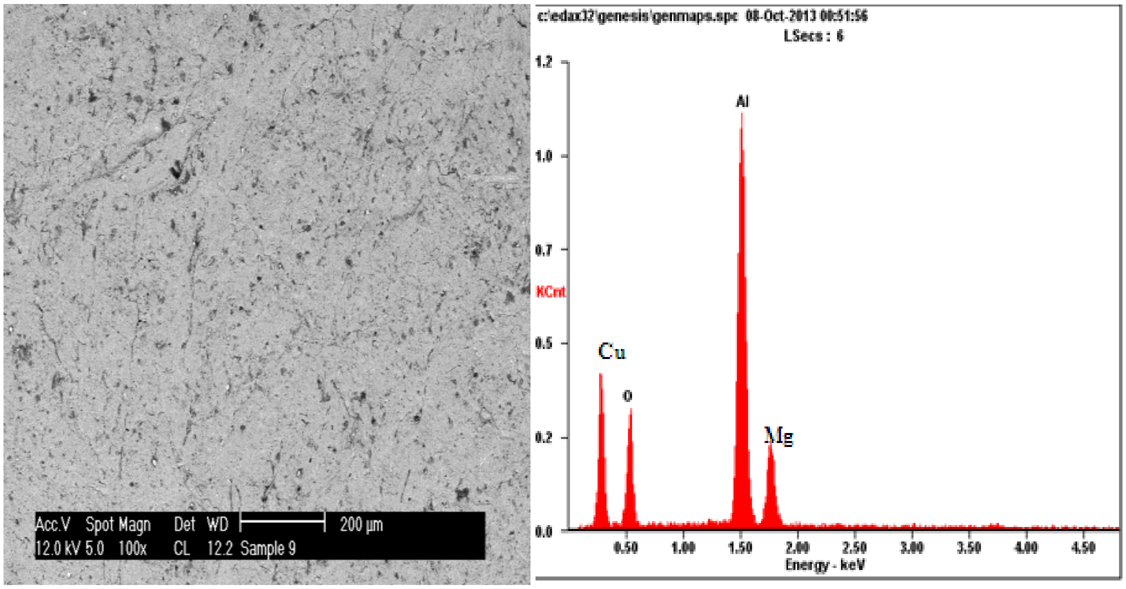


Figure 4.1: XRD pattern of Al-3.7%Cu-1.4%Mg/PWA with 25wt% periwinkle shell ash.

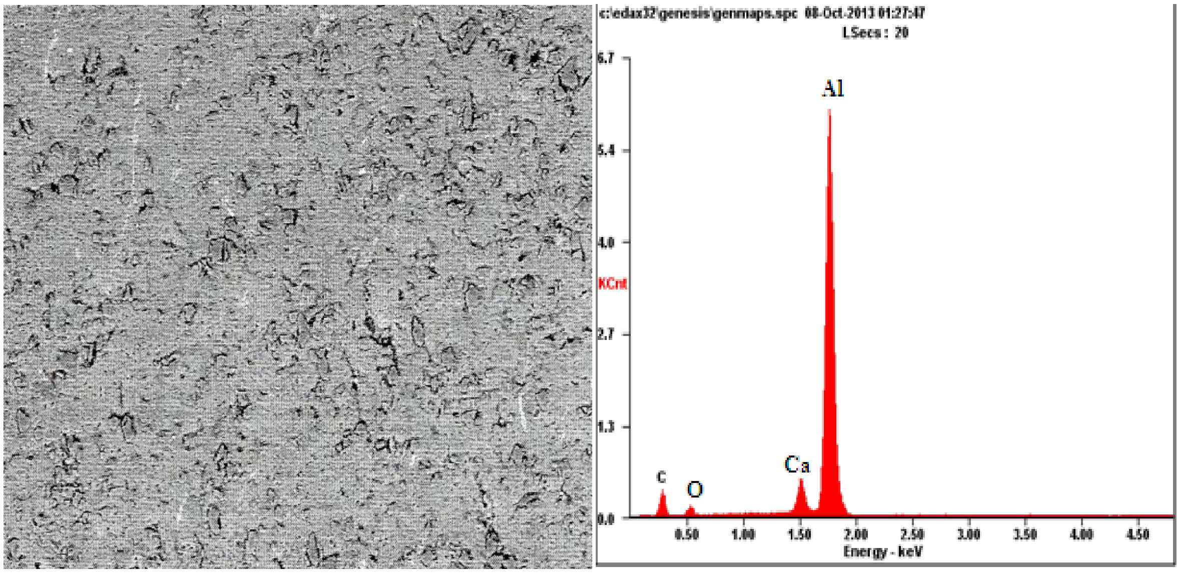
PHASES	FREQUENCY
$Ca_2SiO_4.SiO_2.Mg_3Fe_2(SiO_4)_3$	3
$Ca_2SiO_4.Mg_3Fe_2(SiO_4)_3$	3
$Ca_2SiO_4.SiO2.TiO_2.Mg_3Fe_2(SiO_4)_3$	3
$Mg_{0.9}.Mn_{0.10}.\ Ca_2SiO_4.Ca_2SiO2.Mg_3Fe_2(SiO_4)_3$	2
$TiO_2.Mg_3Fe_2(SiO_4)_3$	2
$Ca_2SiO_4.Ca_2SiO2.TiO_2.Mg_3Fe_2(SiO_4)_3$	1
$Ca_2SiO_4.Ca_2SiO_4$	1
$Mg_3Fe_2(SiO_4)_3$	1
$Ca_2SiO_4.Ca_2SiO_2$	1
$Mg_{0.9}.Mn_{0.10}.\ Ca_2SiO_4.TiO_2$	1

4.1.3 SEM/EDS Analysis of Composites Produced

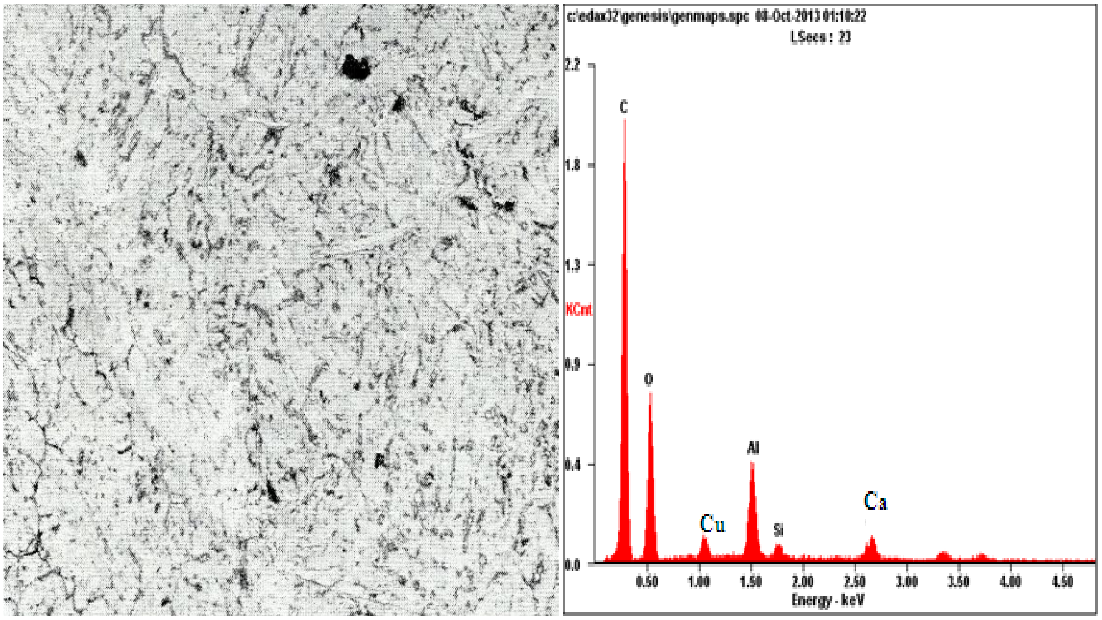
The SEM/EDS analyses of the produced composites are shown in micrograph 4.1 to 4.4.



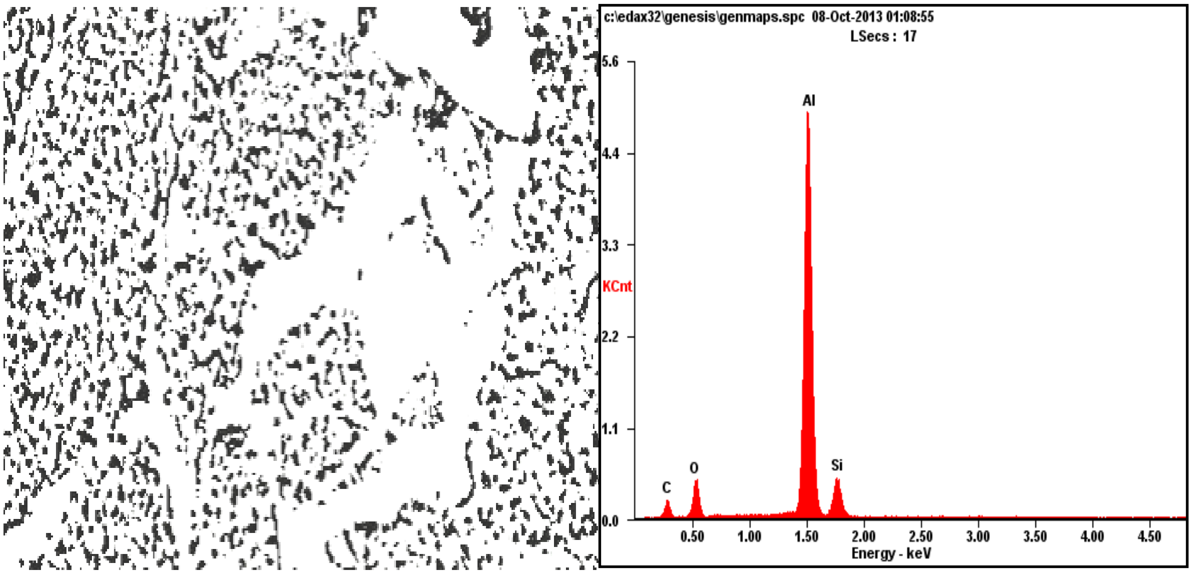
Micrograph 4.1: SEM/EDS of the unreinforced Al-3.7%Cu-1.4%Mg/EDS. Showing the Al-Cu-Mg Alloy and a network of $CuAl_2$ compound (dark) in the matrix of Al (white).



Micrograph 4.2: SEM/EDS of the reinforced Al-3.7%Cu-1.4%Mg with 10wt% PWAp. Showing a network of (Al-Mg and $CuAl_2$) compounds and PWAp at the grain boundaries in the matrix of Al (white).



Micrograph 4.3: SEM/EDS of the reinforced Al-3.7%Cu-1.4%Mg with 25wt% of PWAp. Showing a network of Al-Cu and Al-Mg compounds (dark) and with distribution of PWAp in the grain boundaries in the Al matrix.



Micrograph 4.4: SEM/EDS of the reinforced Al-3.7%Cu-1.4%Mg with 30wt% PWAp. The structure reveals little spacing between the arm and distribution of carbonized periwinkle ash as well as $CuAl_2$ and Al–Mg phases in the grain boundaries (dark) in Al matrix (white).

4.1.4 Physical Properties of the Composites Produced

The density and apparent porosity of Al-3.7%Cu-1.4%Mg/PWAp containing 0 – 30 wt% PWAp additions are shown in Figure 4.2 - 4.3 respectively.

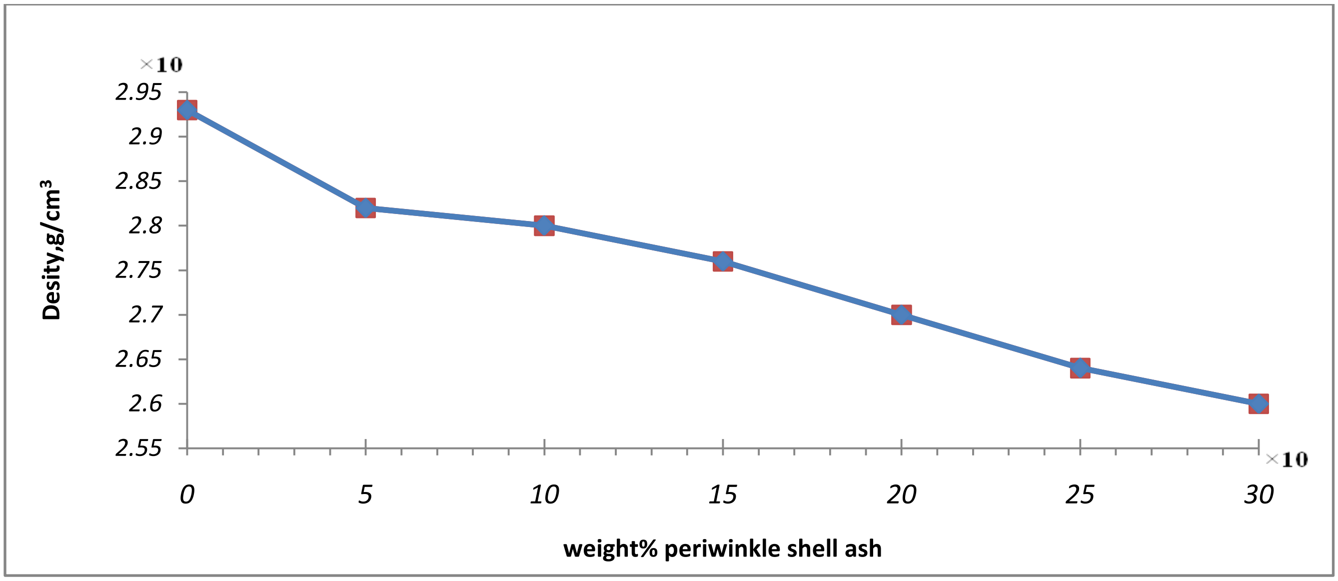


Figure 4.2: Variation of Density of Al-Cu-Mg/PWAp with wt % PWAp additions

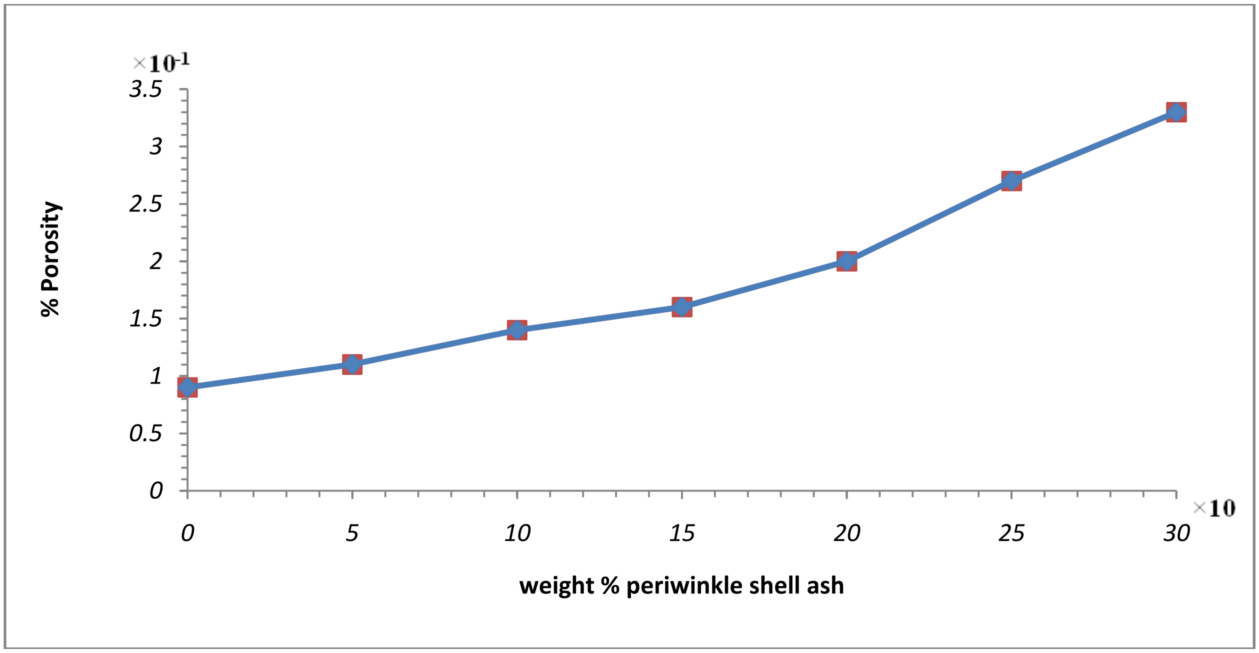


Figure 4.3: Variation of Porosity of Al-Cu-Mg/PWAp with wt % PWAp additions

4.1.5 Mechanical properties of the composite produced

The mechanical properties of Al-3.7%Cu-1.4%Mg/PWAp containing 0 – 30 wt% PWAp additions are shown in Figures 4.4 to 4.7 respectively.

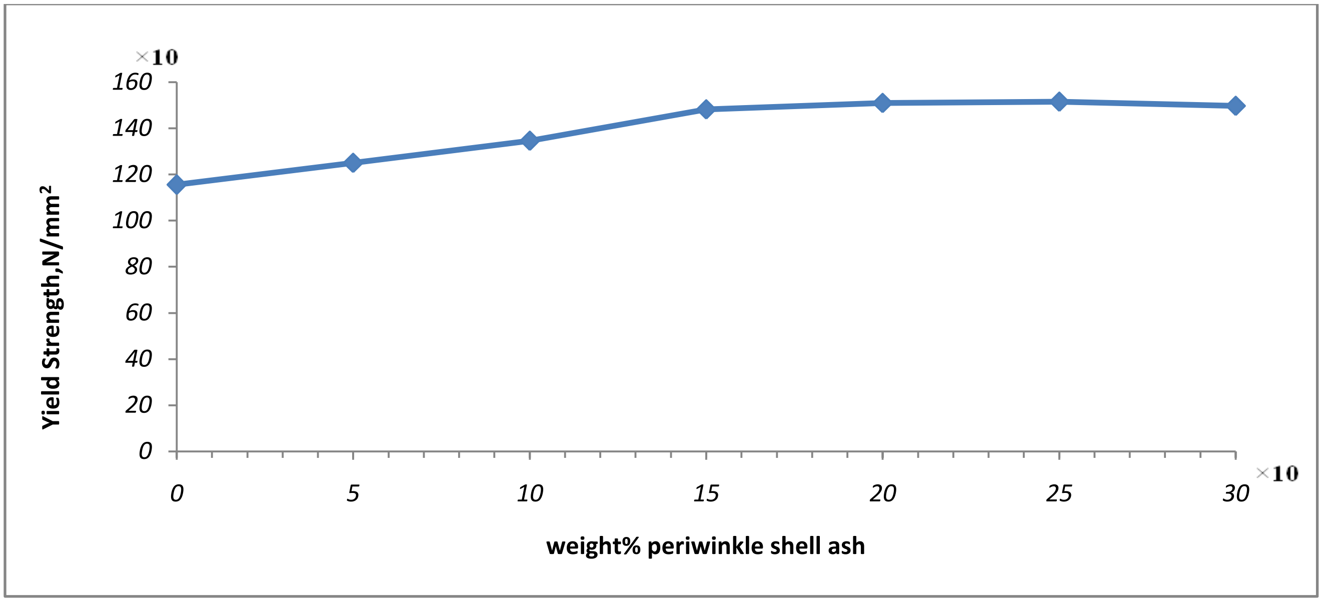


Figure 4.4: Variation of Yield strength of Al-Cu-Mg/ PWAp composites.

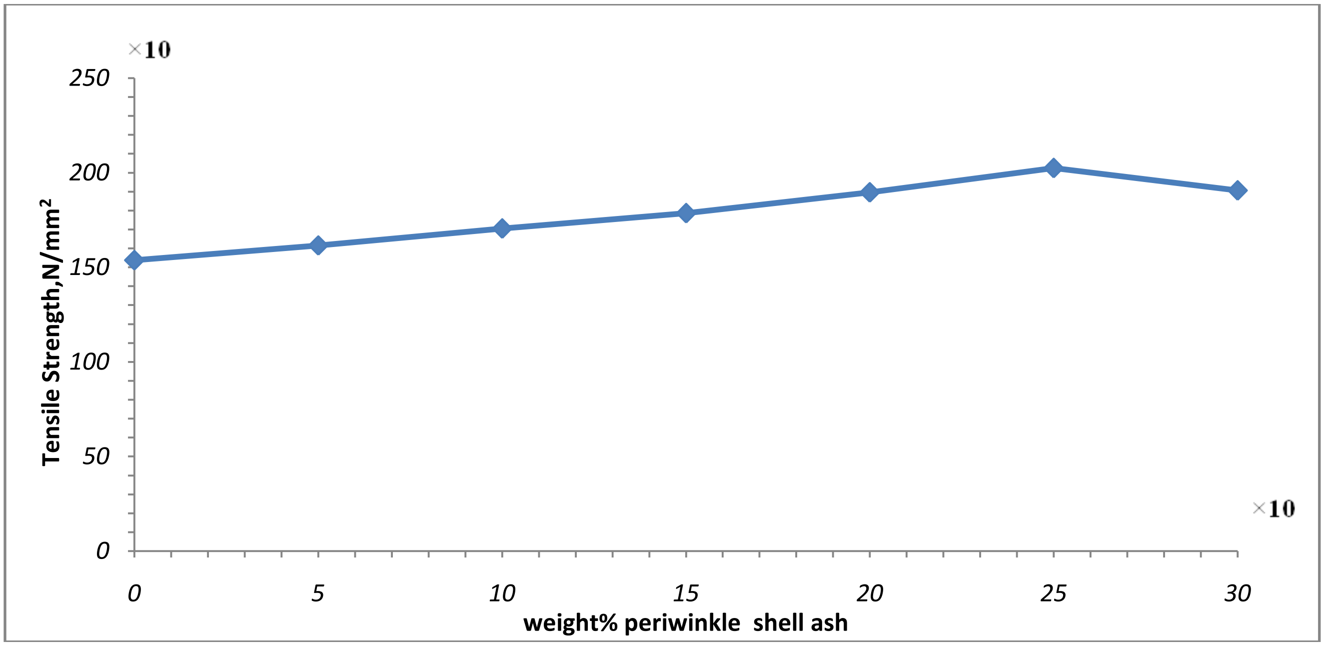


Figure 4.5: Variation of Tensile strength of Al-Cu-Mg/ PWAp composites.

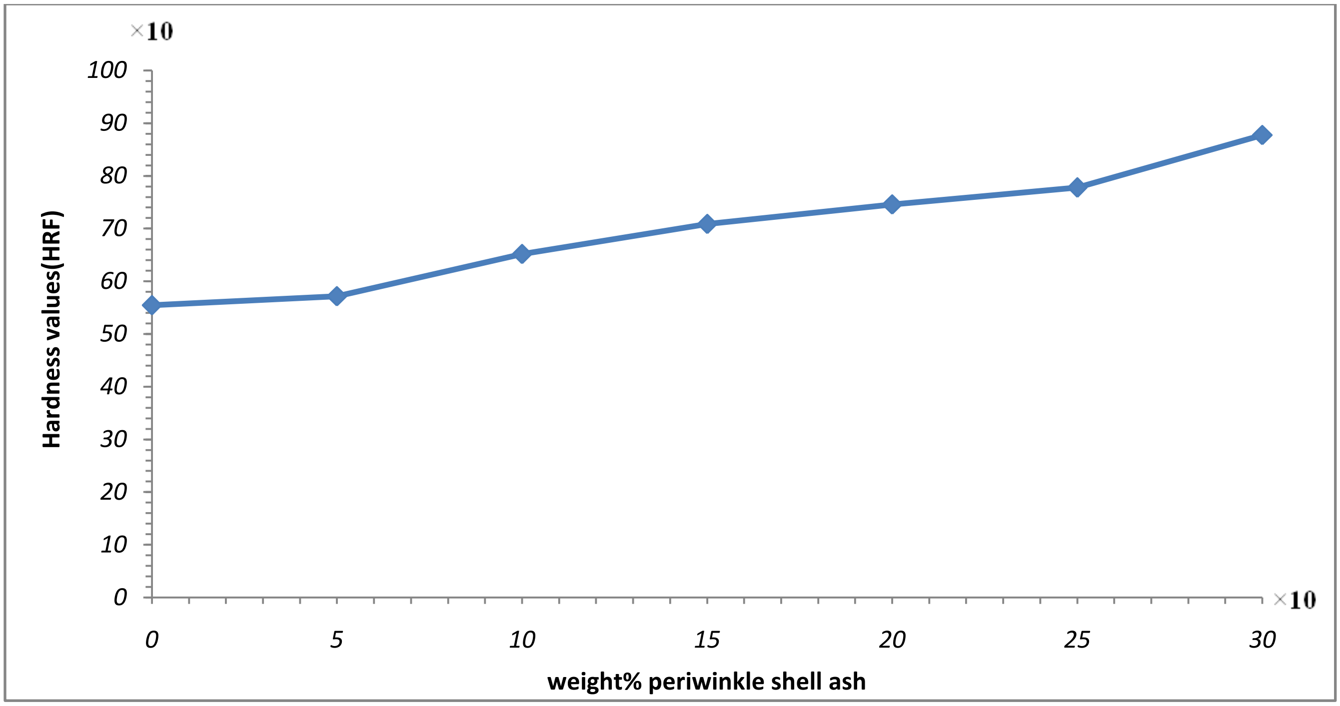


Figure4.6: Variation of hardness values of Al-Cu-Mg/ PWAp composites.

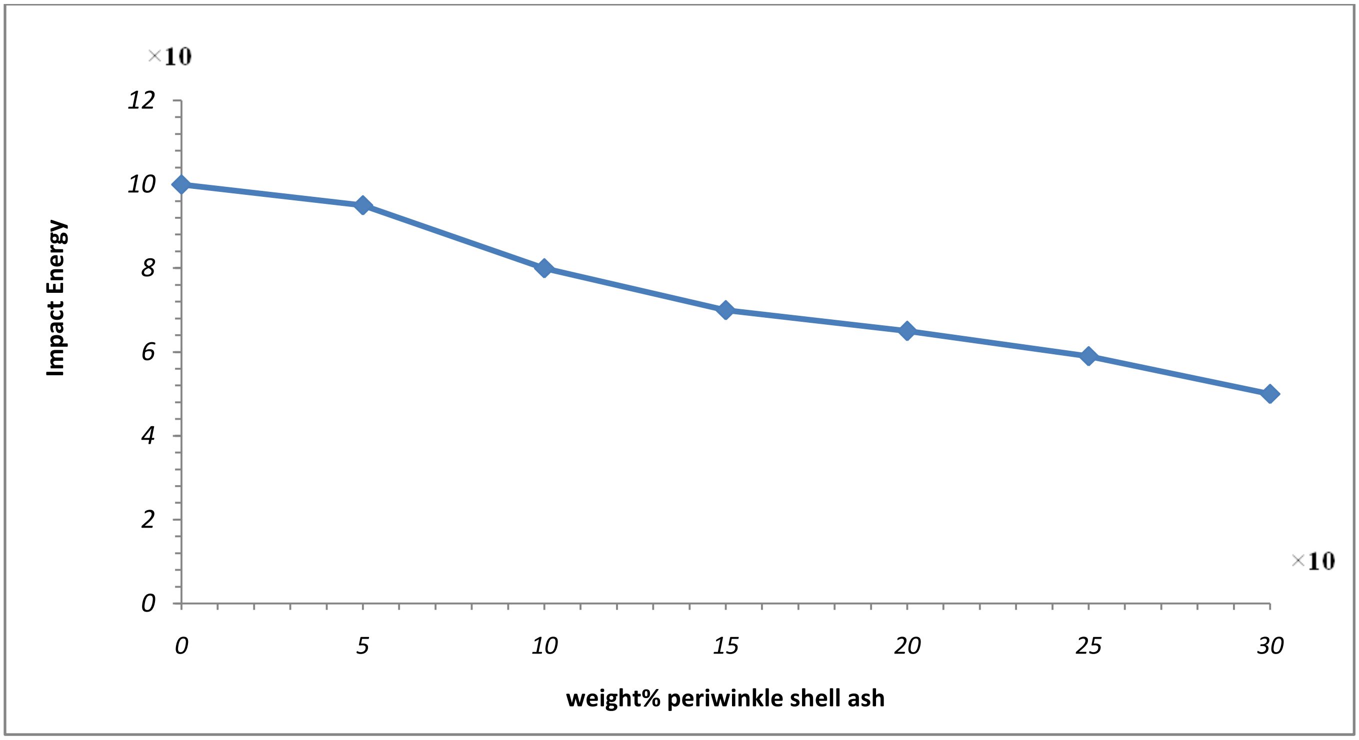


Figure 4.7: Variation of the impact energy of Al-Cu-Mg/ PWAp composites

CHAPTER FIVE

5.0 DISCUSSION

5.1 CHEMICAL ANALYSIS OF THE PERIWINKLE SHELL ASH BY X-RAY FLUORESCENT

The determined chemical composition of the periwinkle shell ash determined by XRF is given in Table 4.1. The analysis confirmed that SiO_2, Al_2O_3, Fe_2O_3, and CaO were found to be major constituents of the ash. Silicon dioxide, iron oxide, alumina and titanium oxide are known to be among the hardest substances. Some other oxides viz. K_2O Na_2O and P_2O were also found to be present. The presence of hard compounds like SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO and TiO_2 suggested that, the periwinkle shell ash can be use as particulate reinforcement in various metal matrixes. This result of XRF is in agreement with the result of XRD obtained. Therefore, the present work suggests the possibility of periwinkle shell ash as particulate in metal matrix composites since the chemical composition has similarity with the XRF analysis of rice husk ash, fly ash and bagasse ash currently used in metal matrix composite (Prasad, 2006, Hassan and Aigbodion, 2010, Rajan *et al.,* 2007).

5.2 X-RAY DIFFRACTION ANALYSIS (XRD)

The XRD pattern of the Al-3.7%Cu-1.4%Mg alloy reinforced with 25 wt% periwinkle shell ash composite produced by double stir casting method is shown in Figure 4.1. From the above Figures it was observed that, the major diffraction peaks of the reinforced alloy are 31˚, 39˚, 42˚, 54˚ and 81˚. The large peaks found are of $CaSiO_4.SiO_2.Mg_3Fe(SiO_2)_3$; $Ca_2SiO_4.Mg_3Fe_2(SiO_4)_3$; $Ca_2SiO_4.SiO2.TiO_2.Mg_3Fe_2(SiO_4)_3$.where as small peaks indicate the presences $Ca_2SiO_4.Ca_2SiO_4$; $Mg_3Fe_2(SiO_4)_{3;}$ $Ca_2SiO_4.Ca_2SiO_2$ etc. In these diffractograms, one can evidently deduce the crystalline phases of the aluminium alloy from that of the composite material. The diagram and confirm the presence of PWA particles in the composites.

5.3 MICROSTRUCTURAL ANALYSIS

The microstructure of the unreinforced alloy is shown in micrograph 4.1. The structure contains essentially a network of the θ phase ($CuAl_2$) and plates of Al-Cu-Mg compound in α- Aluminum matrix.

Micrographs 4.2- 4.4, show the microstructures of the alloy reinforced with periwinkle shell ash. The structure reveals a network of the $CuAl_2$ compound as well as the Al-Mg compound with the periwinkle particulates at the grain boundaries. The microstructures reveal the distribution of the PWAp particles in the grain boundaries and the presence of precipitates of $CuAl_2$ and the Al-Mg compound in the Al matrix.

In the composite examined, no effects of unfavorable phenomena were observed, such as the sedimentation or flowing out of the reinforcing phase, as well as the formation of particle agglomerates or gas blisters. The composite matrix alloy had a eutectic structure, typical of cast materials (Metals Handbook, 1979; Valdez et al, 2008). This showed that there was good interfacial bounding between the periwinkle shell ash particles and Al-Cu-Mg matrix. The good interfacial bounding may be obtained from the magnesium in the matrix which helps in enhancing wettability of the ceramic phase in metal matrix (see Micrograph 4.2-4.4).

The unreinforced alloy reveals the presence of α-Al, Cu and Mg as evident from the EDS spectra (see Micrograph 4.1). From the EDS analysis of the composites material (see micrographs 4.2-4.4) there are indication of some possible chemical reaction between aluminum melt and periwinkle shell ash particles which led to the released of Si, Ca, Al and Cu e.t.c in the composites, since the periwinkle shell ash particles consist of SiO_2, CaO andAl_2O as the major constituents, while the minor ones are Na_2O, SO_3 and P_2O (see Table 4.1).These structures are in agreement with phases studied by other researchers (Naresh, 2006 and Rohatgi, 1994).

Finally, as mentioned above, no particle fragmentation occurs in the aluminium alloy melt, giving rise to a clean surface, which can form an intimate contact with the aluminium alloy melt and obtain a perfect metallurgical bonding after solidification without impurities or gas presenting at particle-matrix interface.

5.4 DENSITY

The result of the density measurements of Al-3.7%Cu-1.4%Mg with Wt% addition of periwinkle shell ash is shown in figure 4.2 and Table A1 (Appendix A).

The result show that the density of the unreinforced alloy is 2.93g/cm^3 and the density of Al-Cu-Mg/PWAp composites decreased as periwinkle shell ash additions increases in the aluminium alloy. The density of the reinforced periwinkle shell ash particulate composites decreased from 2.82g/cm^3 at 5wt% of PWAp addition to 2.60g/cm^3 at 30wt% of PWAp addition. This is due to fact that periwinkle shell ash particles are less denser than aluminum alloy and also it showed that composites with light weight auto components can be made with periwinkle shell ash. This is in agreement with the earlier work of Hassan and Aigbodion and Rajan *et. al*, (Hassan and Aigbodion, 2010; Rajan *et. al*, 2007)

5.5 APPARENT POROSITY

The apparent porosity values of the reinforced MMCS with periwinkle shell ash particles slightly increased with percentage periwinkle shell ash addition is shown in figure 4.3 and Table A2 (Appendix A). The porosity of the reinforced periwinkle shell ash particulate composites increased from 0.11% at 5wt% of PWAp addition to 0.33% at 30wt% of PWAp addition. The obtained values were lower than that obtained by Zhou and Xu. The lower values of apparent porosity obtained in this study was a result of the use of a covering fluxes combine with double stirring method during casting (Zhou and Xu, 1997; Hassan and Aigbodion, 2010).

5.6 MECHANICAL PROPERTIES

5.6.1 Yield and Tensile Strength

From the results of the tensile test, it reveals that the yield strength and Ultimate tensile strength of the base metal alloy was improved on addition of the reinforcement (PWAp) are shown in Figures 4.4 - 4.5 and Table A3 – A4 (Appendix A). This is due to fact increase in the percentage of PWAp allow it to increase in the reaction to produce more calcium silicate resulting in higher strength of the composite produced.

From Figures 4.3 and 4.4, the yield strength and ultimate tensile strength of composites increased with increasing percentage of periwinkle shell ash addition to maximum value of 151.60 and 202.45 N/mm^2 for yield and tensile strength respectively at 25 wt% PWAp addition and then decreased to 190.58 and 149.75 N/mm^2 for yield and tensile strength at 30wt% PWAp addition respectively. The slightly decrease in the strengths at 30 % periwinkle shell ash addition is attributed to the agglomeration and segregation of periwinkle shell ash particles in the microstructure (see micrograph 4.4). With increasing weight fraction of periwinkle shell ash, more loads are transferred to the reinforcement which also resulted in a higher tensile strength. This behavior is in agreement with the work carried out by Aigbodion and Hassan (Hassan and Aigbodion, 2010). This result may be attributed to several factors including the good particle-matrix interfacial bonding, the fine reinforcement particle size, the strengthening effect of the periwinkle shell ash on the aluminium alloy matrix.

5.6.2 Hardness values

Hardness values of the produced composites with %wt periwinkle shell ash are shown in the Figure 4.6 and Table A5 (Appendix A), the hardness values of the developed composites increased as the %wt periwinkle shell ash addition increases in the alloy. The hardness value of composites increased from 55.45 for the unreinforced composite and the presence of the hard ceramic phase in the ductile matrix has resulted into the increase in the hardness of the composite from 57.15 to 87.75 for 5 wt% and 30 wt% periwinkle shell ash particles respectively.these increments were attributed to increase in weight percentage of hard and brittle phase of the periwinkle shell ash particles in the aluminium alloy. This hardness values of the periwinkle shell ash particles are obtained from the phases presences of $TiO_2.Mg_3Fe_2$ $(SiO_4)_3$, $Ca_2SiO_4.SiO_2.TiO_2.Mg_3Fe_2$ $(SiO_4)_3$ and Mg_3Fe_2 $(SiO_4)_3$ of the chemical made up of the particles. also the presence of periwinkle shell ash particles in the alloy increases the dislocation density at the particle-matrix interfaces. This is as a result of differences in coefficient of thermal expansion (CTE) between the hard brittle reinforced particles and soft and ductile metal matrix which results to elastic and plastic incompatibility between the matrix and the reinforcement. Hence, there is an improvement in the hardness values (Hassan and Aigbodion, 2010, Prasad, 2006, Rajan, 2007).

The strengthening effect of periwinkle shell ash particles is noticeable, with the hardness values and tensile strength values of the composites. To understand the above improvement, it is desirable to discuss the strengthening mechanism in details. Generally, the mechanical properties of metallic materials are significantly affected by various factors, such as grain-boundaries, sub-structures, solid solutions, second phases, and so on. For the present materials, contributions from sub-structures and solid solutions effects can be ignorable. Based on the structural information obtained above (Figs. 4.1 - 4.3), the grain refinement of the matrix and the reinforcement contributed by breadfruit seed hull ash particles are the main reasons for the improvement of the hardness and tensile strength illustrated above (Hassan and Aigbodion, 2010).

5.4.3 Impact energy

The results of impact energy with weight fraction of periwinkle ash are shown in Figure 4.7 and Table A6 (Appendix A). From the figure above, the impact energy of the sample, decreased as the percent periwinkle addition increased in the alloy. The brittle nature of the reinforcing materials (PWAp) plays a significant role in reducing the impact energy of the resultant composite (Hassan and Aigbodion, 2010,Rajan, 2007).

CHAPTER SIX

6.0 CONCLUSIONS AND RECOMMENDATIONS

6.1 CONCLUSIONS

From the results and discussions the following conclusions can be made:

1. Al-Cu-Mg alloy/PWAp composites were synthesized successfully by using double stir casting method.

2. Periwinkle ash can be used as a reinforcer in metal matrix composites

3. The density of Al-Cu-Mg alloy/PWAp composites decreased with increases in the periwinkle shell ash content. This means that composites of light weight can be produced with periwinkle shell ash.

4. Incorporation of periwinkle shell ash particles in aluminum matrix has led to the production of low cost aluminum composites with improved hardness and strength.

5. The developed Al-Cu-Mg/PWAp exhibits superior mechanical properties to the Al-Cu-Mg alloy.

6.2 RECOMMENDATION

Based on the analysis and discussion of the results obtained in this research work, the followings recommendation can be made:

1. It is recommended that matrix–particles interface reactions be investigated using high-resolution transmission electron microscopy (HRTEM).
2. Other mechanical properties of this material such as fatigue and creep should be investigated.

6.3 CONTRIBUTIONS TO KNOWLEDGE

From the study, the followings contributions to knowledge can be made:

i. Periwinkle shell ash particle (PWAp) can be considered as an alternative to reinforcement of MMCs instead of the costly and high density reinforcer e.g. SiC, Al_2O_3.

ii. Periwinkle shell ash particulate improved the mechanical properties of Al-Cu-Mg alloy (e.g. tensile strength $150N/mm^2$ to $200N/mm^2$).

iii. Considerable experimental data on periwinkle shell ash that was generated from this research which could be utilized by other researchers.

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APPENDIX A

Table A1: Density of Al-Cu-Mg Alloy with percentage periwinkle shell ash

Wt% PWAp	Weight (grams)	Volume (cm^3)	Density (g/cm^3)
0	20.49	7.00	2.93
5	20.55	7.29	2.82
10	20.44	7.30	2.80
15	20.75	7.52	2.76
20	20.87	7.73	2.70
25	21.15	8.01	2.64
30	20.57	7.91	2.60

Table A2: Porosity of Al-Cu-Mg Alloy with percentage periwinkle shell ash

Wt% PWAp	W_1 grams	W_2 grams	W_3 grams	% Porosity
0	10.154	1.567	10.162	0.09
5	10.188	1.924	10.197	0.11
10	10.395	2.549	10.406	0.14
15	10.251	3.387	10.262	0.16
20	10.450	5.959	10.459	0.20
25	11.207	4.178	11.226	0.27
30	10.567	4.526	10.587	0.33

Table A3: Yield Strength of Al-Cu-Mg Alloy with percentage periwinkle shell ash

Wt% PWAp	Cast- composite (N/mm^2)
0	115.56
5	125.05
10	134.60
15	148.20
20	151.00
25	151.60
30	149.75

Table A4: Tensile Strength of Al-Cu-Mg Alloy with percentage periwinkle shell ash

Wt% PWAp	Cast- composite (N/mm^2)
0	153.75
5	161.58
10	170.45
15	178.60
20	189.58
25	202.45
30	190.58

Table A5: Hardness Values of Al-Cu-Mg Alloy with percentage periwinkle shell ash

Wt% PWAp	Cast- composite (HRF)
0	55.45
5	57.15
10	65.15
15	70.80
20	74.55
25	77.75
30	87.75

Table A6: Impact Energy of Al-Cu-Mg Alloy with percentage periwinkle shell ash

Wt% PWAp	Cast- composite
0	10.00
5	9.50J
10	8.00J
15	7.00J
20	6.50J
25	5.90J
30	5.00J