EVALUATION OF HOT- GAS AND HEATED TOOL WELDMENTS OF POLYPROPYLENE/BONE PARTICULATE COMPOSITES

BY

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DECLARATION

I declare that the work in the dissertation entitled: “EVALUATION OF HOT- GAS AND HEATED TOOL WELDMENTS OF POLYPROPYLENE/BONE COMPOSITES”, has been carried out by me in the Department of Metallurgical Engineering under the supervision of Dr E.T. Dauda and Dr F. Asuke. The information derived from the literature has been duly acknowledgedin the text and a list of references provided. No part of this dissertation waspreviously presented for another degree or diploma at this or any other institution.

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CERTIFICATION

This dissertation entitled "Evaluation of Hot- Gas and Heated Tool Weldments of Polypropylene/Bone Composites" by Bashir Tsauri TUKURmeets the regulations governing the award of the degree of Master of Science (Metallurgical and Materials Engineering) ofAhmadu Bello University, Zaria, and is approved for its contribution toknowledge and literary presentation.

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DEDICATION

This work is dedicated to late Alhaji Mustapha Rabi'u and my lovely daughter, Aliya.

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I would like to first thank Almighty Allah for His guidance, protection and infinite mercies upon my life, steering me across turbulent educational waves throughout my stay in A.B.U, Zaria. Indeed to Allah be the glory.

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ABSTRACT

The evaluation of Hot-Gas and Heated-Tool weldments of Polypropylene/Bone composite was conducted. The composites were formulated by incorporating up to 30% by weight of calcined cow bone powder at an interval of 5% and -75μmsieved size was used as reinforcing phase during compounding process. The polypropylene materials (in unreinforced state) and various polypropylene/bone composites were welded, using hot-gas and heated-tool welding processes. Mechanical properties (tensile strength, flexural strength, impact strength and hardness) and physical properties (density, water absorption, degradability and morphology) of polypropylene and polypropylene/bone composite in both unwelded and welded conditions were examined. Results obtained showed increase in density (by 40% at 30% reinforcement); the amount of water absorbed increased as the time of immersion increased. Although the unreinforced polypropylene was saturated after 192 hrs of immersion in water, the reinforced composite's water uptake continued beyond 192 hrs in proportion of filler amount. Similarly, there were marked improvements in mechanical properties in the Unwelded Composite (UWC), which was attributed to the reinforcing ability of the bone. However, relatively lower values were recorded when welded samples were examined. More so, there were drops in tensile strength after 15% (40.91MPa) and 20% (41.54 MPa) in Heated Tool Weldments (HTW) and Hot Gas Weldments (HGW) respectively. On the basis of comparison, these values showed that at 15% reinforcement addition, HTW has strength value 16.70% lower than UWC of the same composition (15% bone), while in composite with 20% of reinforcement, the strength value of HGW was found to be 23.23% lower than UWC of the same composition. Furthermore, flexural strength and hardness witnessed increase as more of polypropylene was replaced by bone powder. Impact energy decreased and then increased; after 10% of reinforcement addition, all but UWC set of samples witnessed drop in their ability to absorb energy on impact as a result of bone additions. These behaviours have been explained in terms of strengthening effect and volume fraction of the reinforcement as well as the effect of welding processes.

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LIST OF ABBREVIATIONS

IWE	- International Welding Engineers
UWC	- Unwelded Composite
HTW	- Heated Tool Weldment
HGW	- Hot Gas Weldment
PP	- Polypropylene
w	- Weight of sample
ρ	-Density of sample
v	- Volume of sample

CHAPTER ONE

1.0 INTRODUCTION

The development of many technologies that make our existence so comfortable depends largely on the availability of suitable materials (Callister, 2007). However, most of these technologies require a material with unusual combination of properties (e.g. high specific strength, magnetic–transparent, conductive–transparent, catalytic–magnetic, huge yet invisible to human eye and so on), which indeed exceed the domain of our conventional metal alloys, ceramics, polymers, heat treatments etc (Luigi and Gianfranco, 2005;Hanemann and Vinga 2010).Nevertheless, the use of compositesas another class of engineering materials has proven to be vital and a promising candidate in the areas of these advanced technologies. Other answers to these contemporary developments include bio-technology, nanotechnology to mention a few.

Composites were developed to improve on the properties (strength to weight ratio, good corrosion resistance, thermal stability etc) of a monolithic material so that it could be used in sophisticated areas such as aviation (where high specific strength is desired), marine (where low weight and high corrosion resistance guaranty safety), sporting equipment (where less weight is appreciated), and many other applications which include high performance rocket-motor and pressure vessels (Harris, 1999).

Composites are made up of primarily two major individual materials referred to as constituent materials. These constituent materials are termed as matrix and reinforcement. At least one portion of each type is required. The matrix material surrounds and supports the reinforcement materials by maintaining their relative positions; while the reinforcements impart their special mechanical and physical properties to enhance the matrix properties. The net effect is thus an attainment of a material with a unique combination of properties not common to either the matrix or the reinforcement (Matthews and Rawlings, 2005; Callister 2007). The common matrices used include metals/alloys, ceramics and polymers while the reinforcement can be in form of fibre (short or continuous) or particulate reinforcement (Hull and Clyne, 1981).

Depending on the matrix and the reinforcement used in composite formulation, properties of the composite are indeed direct interpolation of its constituents' properties. As a consequence,thermoplastic composites display appreciable properties which are known to be inherent features of their matrices (Matthews and Rawlings, 2005). In line with this, thermoplastic reinforced composites enjoy high demand with increased interest to developing

technologies that transform these classes of composites into a form most suitable for practical applications (Yousefpour *et al*., 2004).

Fortunately, thermoplastic composites (owing to the inherent matrix's properties) offer some degrees of post manufacturing processing; hence they can be reheated and remoulded as infinitely as desired. To this effect, welding of thermoplastic composites becomes feasible as a significant number of this specie have since been welded in the course of repair or fabrication into desired configuration (**Buxton**, 2002).

Today, the use of welding to join thermoplastic composite structures is becoming more important since thermoplastic composite materials are increasingly being used to replace their metallic or thermoset composite counterparts. This is in line with their superior static and fatigue load bearing capacities in aerospace, automobile, and marine industries (Yousefpour *et al*., 2004). While the requirements of polymeric composites increase, so do the requirements for joining especially in structural application (Sercer and Raos, 2011). The use of traditional joining methods such as mechanical fastening, adhesives etc to join thermoplastic composites is difficult, labour and cost intensive (Matthews and Rawlings, 2005; Winkler, 2009); hence the development of welding techniques suitable for welding both un-reinforced and reinforced thermoplastic polymers. However, most of the techniques used are still at a state of intense research. Hence the present study (aimed at welding polypropylene/bone composite into butt joint) will determine the extent to which reinforcement be added in as much as welding (hot gas or heated tool) is desired along the lifecycle of the polypropylene/bone composite (in particular) and composites race in general.

1.1 Statement of the Problem

When Polymersare used as matrices (reinforced with fibres or particles), some properties are effectively tailored at the expense of others; to this effect, challenges during the life cycle of compositeare often encountered (Harris, 1999). Considering the fact that composites are fabricated into real engineering component using various joining processes such as welding (Sercer and Raos, 2011);however, weldability reduces as discontinuities (reinforcing phase) increase. For example, a highly reinforced polymer was found to be brittle and difficult to be welded; on the other hand, low reinforced polymer behaves synonymous to the parent material (**Buxton, 2002).**In line with this, polymer reinforcement reduces their fabricability especially by welding. Hence the present research is geared at providing the optimum level of bone particulate (powder) addition to polypropylene without appreciable compromises to its weldability and other properties of engineering interest.

1.2.Aim and Objectives

The aim of this work is to evaluate the possibility of welding polypropylene/bone composites using hot-gas and heated tool welding techniques. The specific objectives include the following:

- To procure cow's limb bone from nearby abattoir; subsequently, wash (degrease) the bone with detergent to good physical condition.
- To produce calcined bone powder of -75μm particle size.
- To use various amounts of calcined bone powder as reinforcement in the production of polypropylene/ bone composites.
- To use both hot- gas and heated tool welding techniques to join the various composites into butt welded structure.
- To evaluate the physical and mechanical integrity of the produced composites and also when they have been welded.

1.3. Significance of the Research

This research is intended at providing a unique combination and wide range of both physical and mechanical properties of polypropylene (light, cheap and readily available thermoplastic) without much sacrifice to its light weight through reinforcement with bone (often considered as waste in most parts of Nigeria), which is light in weight, yet hard and strong (Rauf, 2014); hence providing improved specific properties. Subsequent welding and testing is intended at providing a guide to processability and fabricability of the composite. This has provided another yardstick not only to fabrication technique(s) but also an ease of repair in case of breakage or damage in the race of polymeric composites dynasty and specifically to polypropylene/bone composite.

1.4. Scope and Limitation of the Research

This work utilized the concept of fusion welding process to study the effect of animal bone particles addition on the weldability of polypropylene reinforced composite. Moreover, the research was however limited to:-

- The use of hot gas welding and heated tool welding techniques in welding polypropylene and polypropylene/bone composites.
- Welding both reinforced and unreinforced polypropylene into butt jointed structure.
- The use of calcined bone particles as reinforcement.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1Polymersand Polymer Composites

A polymer is generally a product of polycondensation, polymerization or polyaddition.The molecules in polymers are gigantic in comparison with hydrocarbon molecules. Due to their large molecular size,they are often termed as macromolecules. These long molecules are

composed of structural entities called repeated units or monomerwhich are successively repeated along the chain (Callister, 2007). The term monomer (a Greek word: meros meaning part and mono meaning single)refers to thesmall molecule from which a polymer is synthesized, hence the name polymer to imply polymer is composed of many parts. When combined with various agents to enhance or in any way alters the material properties of polymers the result is referred to as plastics (Rosen, 1971).

The word plastic is derived from the Greek word (plastikos) meaning capable of being shaped or moulded. Plastics are typically polymers of high molecular mass, and may contain other substances to improve performance and/or reduce costs. Monomers of plastic are either natural or synthetic organic compounds. Thus composite plastics refer to those types of plastics that result from bonding two or more homogeneous materials with different material properties to derive at a final product with certain desired material's properties (Callister, 2007).

2.1.1Classification of polymers

Polymeric materials or precisely polymers are often classified based on a number of parameters; chemistry of synthesis (condensation and addition polymers), response to temperature (thermosets and thermoplastics), size (molecular weight), shape, structure (linear, branched, crosslinked and network polymers), special properties (smart materials)etc (Rosen, 1971; Hull and Clyne, 1981; Callister, 2007).

2.2.1.Polypropylene

Polypropylene (PP), also known as polypropene, is a thermoplasticpolymer made by the chemical industry and used in a wide variety of applications, including packaging, textiles (e.g. ropes, thermal underwear and carpets), stationery, plastic parts and reusable containers of various types, laboratory equipment, loudspeakers, automotive components, and polymer banknotes. It is an addition polymer made from the monomer propylene(Calafut, 1998).
It is formed when Propylene (an olefin) is polymerized to a crystalline isotactic polymer called polypropylene. The properties of polypropylene are highly dependent on its structure which in

turn depends on its tacticity (the relative orientation of eachmethyl group (CH_3) to next methyl groups in neighbouring monomer). This can either be isotactic or syndiotactic. This parameter has a strong effect on the polymer's ability to form crystals(Calafut,1998).

Three main classes of polypropylene include homopolymer, random copolymer, and block copolymer. The very commoncomonomer being used is typically ethylene. Ethylene-propylene rubber or EPDM added to polypropylene homopolymer increases its low temperature impact strength. Randomly polymerized ethylene monomer added to polypropylene homopolymer however, decreases the polymer crystallinity and makes the polymer more transparent (Rosen, 1971; Callister, 2007).

2.2.2. Chemical and physical properties of polypropylene

Polypropylene in its commercial status is basicallyisotactic and of crystallinity between that of low-density polyethylene (LDPE) and high-density polyethylene (HDPE). It however, differs from polyethylene owing to its lower density, relatively higher transition temperature, higher melting point, higher thermal dimensional stability: and can be made electrically conductive (e.g. PP-EL; Homopolymer) through treatment with electrically conductive particles (Calafut, 1998; ERIKS TOOL, 2014).

The melting point of PP ranges between about 130 °C (for 30% crystallinity) to 175 °C (for perfectly isotactic (PP), while the melt flow rate (MFR) or melt flow index (MFI) is a measure of molecular weight of polypropylene. Polypropylene with higher MFR will fill the plastic mould more easily during the injection or blow-moulding production process. However, some physical properties, like impact strength decrease with increasing MFR(Calafut, 1998).

From the aforementioned properties of polypropylene, PP can be reinforced easily by incorporating suitable filler and already established as a potential candidate for various post manufacturing thermal treatment (Calafut, 1998) such as welding processes (Czigány and Kiss,

2011). However, as the amount filler increase, weldability decrease (**Buxton, 2002;** Sercer and Raos, 2011) - hence the need to investigate the optimum level of such additions without much effect on its weldability.

2.2.3.Polymer additives

Polymer additives refer to foreign substances added or introduced intentionally to change, modify or tailor polymers' properties so that desired properties for a given operation are obtained(Work *et al.*, 2004). They usually represent minor components of the mixture formed (usually less than 5%) yet significantly affect the net properties of a polymer (Matthews and Rawlings, 2005). Some additives may act by controlling molecular arrangement of the host polymer and thus having a net effect on the intrinsic properties (because they are function of molecular nature). Others may operate to alter some of their physiochemical behaviours. However, to satisfy engineering consumption, modification beyond molecular amendment has to be done in as much as sound and reliable mechanical, chemical, and physical properties are desired (Rosen, 1971; Callister, 2007).

A number of polymer additives have since been developed for a number of reasons, the commonest categories of these additives in polymers include (Callister, 2007):-

- Plasticizers to increase flexibility.

- Stabilizers and anti- oxidants to prevent polymer degradation.

- Filler materials: they are introduced to replace some volume of the weak and more expensive polymer, thereby improving properties and or reducing the cost of the final product.

- Surface actants to stabilize emulsion polymers.

- Chain transfer agents and other polymerization modifiers to control molecular weight.

- Crosslinkers used to modify polymer networks.

Other polymer additives include flame retardants, colourants etc (Rosen, 1971; Callister, 2007).

2.2.4. Bone particles

Bone or bone powder (as a polymer additive) falls into category of fillers. Nevertheless, bone is very vital to the present study; this is because, expected improvement in matrix's properties has direct bearing onthe presence of bone particles.

Bones are rigid organs that form part of the endoskeleton of vertebrates; its tissue composes ofprimarily dense connective tissue. They function to move, support, and protect various organs of the body, produce blood cells (red and white) and store minerals. Because bones come in a variety of shapes and have a complex internal and external structure they possess little weight(Rauf, 2014).

Bone in particular, is a relatively hardand light inweight and often considered as natural composite. It consists of hard inorganic crystal(hydoxylapatite or osteones) in a matrix of a tough organic constituent called collagen (Hull and Clyne, 1981). It has relatively high compressive strengthbut poor tensile strength.Althoughcollagen is tough, its presence however, does not toughen bone; as such bone isessentially brittle(Rauf, 2014).

Two types of bone can be identified microscopically according to the pattern of collagen forming the osteoid (collagenous support tissue): woven bone which is characterized with haphazard organization of collagen fibres and is mechanically weak and lamellar bone which has a regular parallel alignment of collagen into sheets (lamellae) and is mechanically strong (Hull and Clyne, 1981; Rauf, 2014).

Limb bone or long bone fall into a class of compact bone which is characterized by a shaft, that is much greater in length than width. They are comprised of (mostly) dense but contain lesser amounts of bone marrow, which is located within the core of the shaft and spongy bone(Rauf, 2014). This is however, eliminated or altered in the course of calcinations; and thus leaving a strong and carbonated apatite (Figueiredo *et al.*, 2010).

When bone is introduced into a given matrix, considering the aforementioned nature and properties of bone, it is expected that, based on the principle of interpolation in properties between matrix and reinforcement; there will be an improvement in the overall properties of the final composite. Moreover, strength per cost is optimised (Callister, 2007).

2.2.5. Composite materials

Composite as applied to engineering materials refers to a product obtained as a result of combinationbetween two or more materials. In other words, a composite is a material whose constituents are chemically distinct from one another at microscopic level and they can be recognized bydistinct and recognizable interface between them (Matthews and Rawlings, 2005). They are however combined so that new material of tailored properties in line with intended application is realized (Harris, 1999). In view of this, many materials are indeed composites owing to the fact that they contain at least two constituents of varied characteristics. In many

cases, there exist a strong and stiff component (reinforcement) embedded in a softer constituent forming the matrix (Hull and Clyne, 1981).

Depending on the nature of composite forming blocks, composites are produced by casting (solidification from the melted state), moulding, chemical polymerization etc (Harris,1999).Most composites are however, produced using polymer matrix material often called a resin(Rosen 1971). There are many different categories of polymers available depending upon the starting raw materials. There are also several othersub-categories, each with numerous variations viz: polyester, vinyl ester, epoxy, phenolic, polyamide, polypropylene and others; while the reinforcing materials are often fibresbut particle substances are also used (Matthews and Rawlings, 2005).

2.2.6. Classification of Composites

Composites have been classified on numerousbases and in line with the parameters that suite such classifications. For example biodegradable and non-biodegradable exist (when environment is of concern), bio-composite and synthetic composite is another class that considers origin, fibrous and laminate composites and so on.

However, a more generalised classification is on either the basis of reinforcement or the matrix used. This may be due to the significance of these two composite forming blocks.

2.2.7. Classification based on reinforcement

Since reinforcement is accomplished either by the use of fibre or particulates (particles): under this classification of composites, fibre-reinforced composites and particulate reinforced composites are often distinguished.

(i) Fibre reinforced: – this class of composites use fibrous materials to mechanically enhance the strength and elasticity of composites. They are characterized by fibre length which is much greater than cross-sectional dimension (aspect ratio); and thus determine the critical length (Matthews and Rawlings, 2005).

Since the matrix is tough but relatively weak, reinforcement with fibre (continuous or discontinuous) will enhance andstrengthen matrix's properties. Hence, fibre reinforced

composites are characterized by enhanced properties in the light of strength, elasticity etc. However, the properties achieved through this reinforcement are functions of inherent properties of both thefibre and the matrix, their volume relative to one another, the fibre length and orientation within the matrix as well the nature of theirinterface (Harris,1999).

(ii) Particulate reinforced: -Particulate reinforced composites on the other hand, consist of particles of various sizes and shape often dispersed within the matrix. The particles dimensions are approximately the same in all directions. The reinforcement can be achieved by the use of large-particle or dispersion-strengthened (very small) or particle-reinforced composites. The distinctionbetween these is based upon mechanism of reinforcement.Properties are tailored in line with intended application. It has an advantage of being cheap and rarely damaged during processing (Matthews and Rawlings, 2005).

2.2.8. Classificationbased on matrix

On the basis of matrix, three classes of composite materials are often distinguished: viz,Metal Matrix Composites (MMCs), Ceramics Matrix Composites (CMCs), and Polymer Matrix Composites (PMCs).

(i) Metal Matrix Composites (MMCs)

Metal Matrix Composites (MMCs) are the class of composite in which the monolithic material to which reinforcement is added is metal or metal alloy(s).This is to say, the matrix in MMCs are basically metallic; and thus reinforcement is introduce to improve and or tailor their

physical,chemical and mechanical properties in line with intended use- hence a wide range of this category is obtainable (Hull and Clyne, 1981; Harris, 1999).

The commonest metals usedas matrix in composites formulation include aluminium, magnesium and titanium. These metals are often combined (alloyed) with other elements in order to improve their physical and mechanical properties,e.g. aluminium reinforced with ceramic particles or short fibres, titanium containing large-diameter fibres etc. Final properties of this class of composites arestrongly controlled by thermal (heat) and mechanical treatments which indeed dictate the microstructure (Harris, 1999).

(ii) Ceramic Matrix Composites (CMCs)

Here, the monolithic material (matrix) is principally ceramic material. Ceramic matrix composites are built primarily for toughness but not for strength.This is because ceramics are so brittle that little increase in toughness may be advantageous. Organic matrix/ceramic aggregate composites include asphalt concrete, mastic asphalt, mastic roller hybrid, dental composite and chobhamarmour (a special type of composite armour used in military applications).Ceramic fibres may be added to ceramics to improve toughness(Matthews and Rawlings, 2005).

(iii) Polymer Matrix Composites (PMCs)

These are class of composite in which the major constituent is polymeric(Work*et al*, 2004); more concisely, the matrix is a polymer material. They can however, be of thermoset or thermoplastic. While the former cannot be heated once it set, the latter can unlimitedly be reheated and remoulded "as much as desired" (Callister, 2007). Also as a class of composites, they can be reinforced with both fibre and particulate.

PMCs are very popular due to their low cost and simple fabrication methods, and arecharacterized byhigh specific strengths and stiffness, high fracture resistance abrasion resistance, impact resistance, corrosion resistance,fatigue resistances and at low cost (Matthews and Rawlings, 2005; ERIKS TOOL, 2014).

2.2.9. Design considerations in composites

Since the fundamental principle of composite formulation is based on the ability to tailor favourable properties into an enhanced and desired ones and minimising the effect of deficiencies of the constituents; for example, when polymeric material (tough and light) is reinforced with glass fibre (hard and brittle) – the resulting composite will be "relatively" strong, tough and light. The inherent deficiencies, i.e. low strength (in the polymer matrix) and

brittleness (in the fibre)are thus eliminated or they play almost zero effect in the composite formed (Harris, 1999).

When choosing a matrix for a given composite, a designer has to bear in mind that introduction of reinforcement into a matrix alter all its properties and cause subsequent changes in microstructure. One property that drives special attention in composites design, unlike thecommon engineering design where approximation to isotropy may be assumed; composite formulations however, result to huge anisotropy (directional properties) in strength, stiffness and many other properties (Hull and Clyne, 1981).To this effect, all parameters of interest need to be examined before endorsing a given composite suitable for a given application (Matthews and Rawlings, 2005).

To start with,although matrix is chosen on the basis of desired goal (cost reduction or properties improvement), however, the primary role of the matrix shall never be negotiated. This is to say, since the matrix has to surround, protect (from mechanical damage) and maintain the relative position of reinforcement in the important stressed directions, good wettability of matrix with respect to reinforcement under consideration be assured (Harris, 1999).

Secondly, load bearing capability of reinforcement has to be ascertained. This is achieved by proper choice of the reinforcement. Moreover, volume fraction, shape, sizeand size distribution, orientation, distribution or agglomeration, particle specific surface area, surface chemistry etc are dictators to reinforcing ability of the reinforcing phase (Matthews and Rawlings 2005; Hanemann and Vinga, 2010).

Last to be considered here is the interaction between matrix and reinforcement. This is becauseefficiency of load transfer is directly related to the quality of the matrix/reinforcement bond- the interface.This parameterbecomes much more significant when thermal treatments areinvolved along manufacturing route (Harris, 1999). As the interface must be strong enough to bear the load transfer between the phases involved; its strengthmusthowevernot be so high that toughening mechanismsare inhibited (Matthews and Rawlings 2005).

2.2.10. Properties and testing of composites

Before a composite can be considered usable, it has to meet some properties soundness when tested. The properties arecategorized as physical, mechanical, electrical, thermal, chemical, and optical properties and so on (John, 1992).

Specific gravity, porosity etc are termed as physical properties. Thermal properties include thermal conductivity, thermal expansion, heat resistance etc. Mechanical properties tell more

about response to external loading (force) and include tensile strength, compressive strength, impact strength etc (Kambour and Robertson, 1972; John, 1992).

Electrical properties include electric strength, flash over, volume and surface resistivity, power factor. Chemical properties are resistance to water absorption, chemicalattack, enzymatic and microbial attacks, pHetc. As such materials are tested to reveal their properties, which by extension help at predicting and preventing failure (John, 1992; Callister, 2007).

2.2.11. Applications ofpolymer composites

Due to their relatively low cost, ease of manufacture, versatility and other unique properties, composite are used in an enormous and expanding range of products. From paper clips to spaceships, composites have already displaced many conventional materials suchas metals and alloys inboth sensitive and insensitive applications (Harris, 1999).

Below are some few examples of composite applications:

i. **Aviation and marine**

Polymer reinforced composite provide answer to the question of aviation sector as their target has been to obtain material of highest specific strength. The less weight and high strength of composites is highly utilized in aviation sector. In marine, apart from high strength to weight ratio, excellent corrosion resistance of these composites is to greatly extend a thirst of marine industry. Statistics shows that more than 60% of metals and alloys have been replaced with composite in thosefields (Yousefpour *et al.,* 2004).

ii. **Food storage and health**

Due to relatively chemical inertness of polymer composites, they are employed in food storage, especially those that will not be further processed. Drugs are also stored in this class of materials.

Another interesting application of composites is in medical transplant for prosthetic purposes- such as orthopedic fracture fixation plates, femoral stems for hip replacements, jaw remodeling. They also possess a slot in external orthotic as supports in case of limb deformity and many implants such as heart valve, artificial lung etc, have since been actualized out of this material(Harris, 1999).

iii. **Automobiles**

Automobiles industry is one of the highest consumers of composite materials. It uses composites in almost every point of their fabrication. Shape memory and smart composites

are highly employed where high precision is needed to replace costly alloys. Another benefit derived from this usage is an increased fuel economy of the automobile(Elmarakbi, 2013).

iv. **Construction**

Composites materials owing to their special properties are extensively used in the area of construction especially in multi storey buildings, where only unavoidable weight is allowed. To this effect, structure must be sound in the light of specific strength and other properties- hence the use of composites. Reinforced polymers have since become basic tools of construction engineers especially where seimic properties are vital parameters (Callister, 2007).

Other applications of composites include sport, security and defence, high performance rocket-motor, pressure vessel etc (Harris, 1998).

2.3.Welding of Plastics and Plastic Composites

Ideally, a designer prefers to produce a monolithic structure (structure without joint); however, due to limitations imposed by material, manufacturing process, cost etc,the use of joint become inevitable(Matthews and Rawlings, 2005). For example, as small as mobile phone is, there exist over 100 joints (Buxton, 2002).

Mechanical fasteners and adhesives are widely employed to join plastic composites. While the former generate stress riser in the event of drilling and galvanic corrosion(more often when metallic phase(s) is involved),mismatch ofcoefficient of thermal expansion, and damage of reinforcing phase(inducedby drilling), the later may be ruined by poor surface preparation. Moreover, specific properties are often compromised owing to (in most cases) addition of unavoidable weight (Yousefpour *et al*., 2004; Matthews and Rawlings, 2005).

In general, joining of thermoplastic materials is achieved by mechanical fastening, adhesive bonding, solvent or consolidation bonding and fusion bonding or welding. Fusion bonding or welding hasgreat potential for the joining, assembly, and repair of thermoplastic compositecomponents and also offers many advantages over other joining techniques (Buxton, 2002; Sercer and Raos, 2011).

Welding is a union of two or more materials by fusion, diffusion or surface alloying by the use of energy with or without the application of pressure, with or without the use of filler material. However, due the relative low fluidity (Melt Flow Rate) and melting temperatures in relation to metals, the use of filler material to consolidate joint is often unavoidable (Buxton, 2002; Winkler, 2009).

The advantages of plastic welding run from fabrication, post fabrication and even repair. Hence,welding of detail parts can be done to obtain assemblies and or repair with weight reduction, cost savingand can (sometimes) result to a joint that is even stronger than the parent material, provided strict adherences are assured both in line with the material (especially when filler material is needed) and the technique guidelines (Yousefpour *et al.,* 2004; Winkler, 2009).

The types of welding techniques/processes used are functions of both the material and joint to be formed; the likes of heated tool welding, hot gas welding, friction welding, induction welding etc, are widely used to achievefusion and diffusion in thermoplastics, while surface alloying is typically accomplished by the use of adhesives (Yousefpour *et al.,* 2004).

2.3.1.Fusion bonding

Fusion bonding or welding of polymer, involves primarilyheating the polymers at the interface to a temperature above their glass transition temperature(usually to viscous state), thus physicallycausing polymer chains to inter-diffuse and cooling the polymer for joint consolidation (Yousefpour *et al.,* 2004).

Fusion bonding technology is originated from the thermoplastic polymer industry; but the endorsement of thermoplastic matrix composites as suitable candidates for primary aircraft structures gave this technology an automatic slot, while researches to establish its optimum utilization is becoming a "lingua franca" (Costa *et al.,* 2012). Moreover, temperature, time and pressure are the key parameters that tell the success or otherwise of a particular welding technique (Winkler, 2009).

Fusion welding techniques are classified on the basis of the principle in which the heat is generated(**Buxton**, 2002). They include:-

- Thermal welding (techniques employing an external heat source).
- Friction welding (techniques where heat is generated by mechanical movement).
- Electromagnetic welding (techniques which directly employ electromagnetism).

The above classification of fusion-bonding is further sub-classified to about twenty already established polymers/polymer composites welding techniques. Figure 2.1 shows such classifications (Yousefpour *et al.,* 2004).

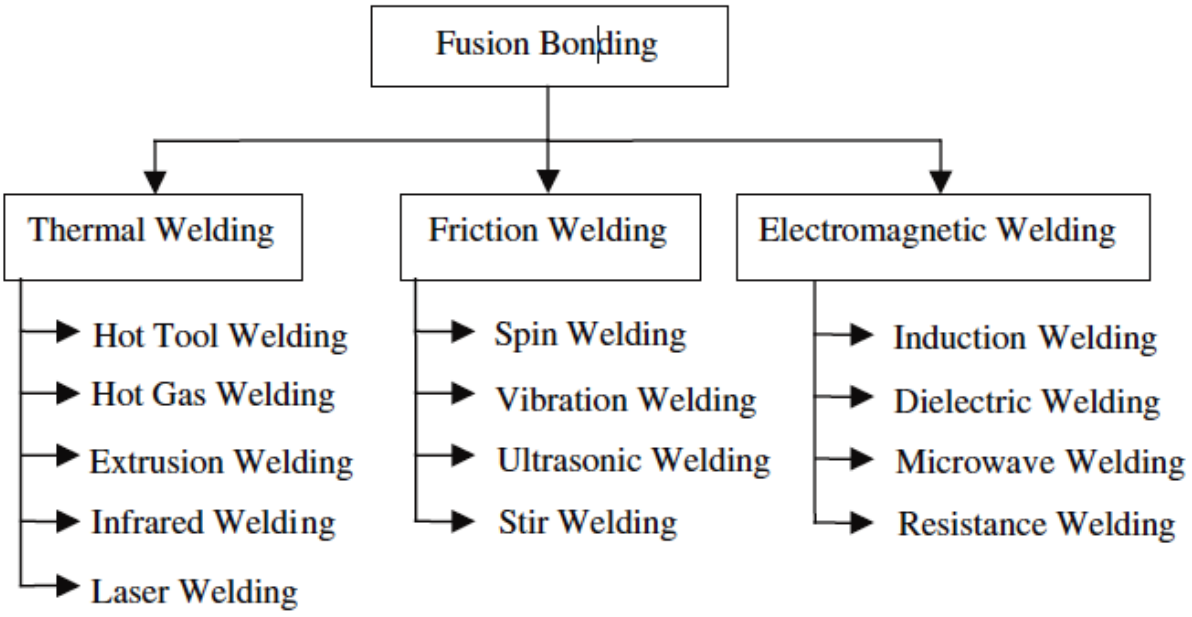


Figure 2.1:Classification of fusion bonding (Yousefpour *et al,* 2004).

2.3.2.Thermal welding

In thermal welding, the union of members to be joined is achieved by application of heat from external source (Winkler, 2009). It is a technique involving two-stage processes,an external heating stage and a forging stage. An external heat source applies heat directly to the individual bonding surfaces and melt the matrix; then, the heat source is removed and the bondingsurfaces are brought in contact under the forging pressure.

Thermal welding technique is divided into hot-tool (plate or bar) welding, infrared welding, hot gaswelding, extrusion welding, and laser welding (Yousefpour *et al.*, 2004).

A) Hot-tool welding

In hot-tool welding, the parts to be joined are heated by the use of suitable heating element to a state sufficiently enough to cause their union. A heated plate, rod etc (depending on the joint configuration) is placed between the surfaces to be joined until melt is realized at the interfaces. As shown in Figure 2.2, the hot-tool is then withdrawn and thesurfaces are brought together in the presence of applied pressure until thethermoplastic polymer solidifies(Yousefpour *et al.*, 2004).

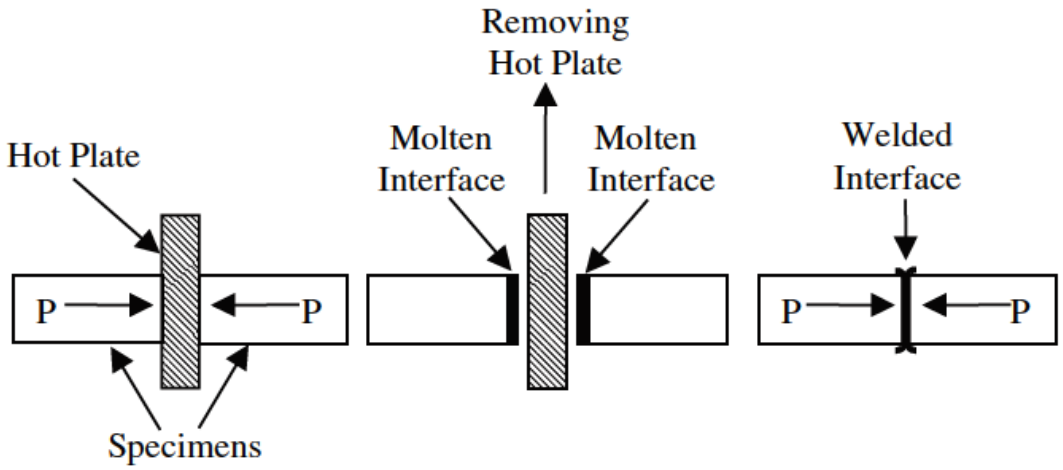


Figure 2.2:Schematic of the hot-tool welding. (Yousefpour *et al,* 2004).

The heat can be applied directly or indirectly onto the workpiece. In direct heating, the heating element is situated between the joint interfaces as shown in Figure 2.3. On the other hand, in indirect process, heat is transferred to the joint interface through a joint component or by used ofnon adhesive coating ofPolytetrafluoroethylene(PTFE). Owing to poor thermal conductivity associated with plastic materials, indirect heating is only suitable for welding thin films (Winkler, 2009).

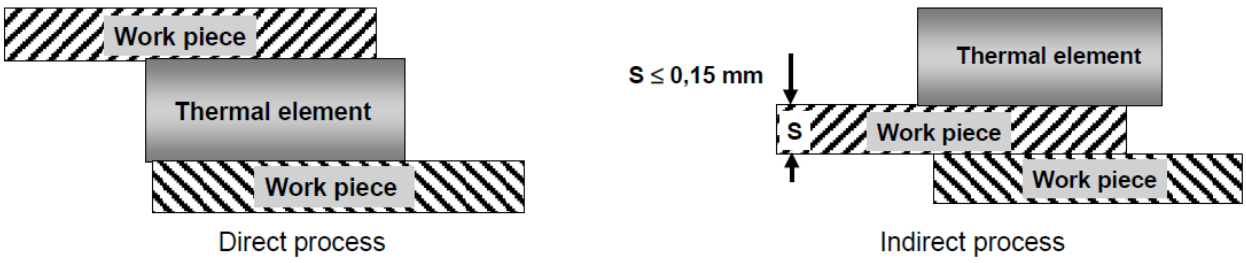


Figure 2.3: Schematic diagram showing different method of heating in hot tool welding (Winkler,2009).

The tools used to achieve heating are of different configurations, but commonly used are in form of plate or bar, hence the terms hot plate and hot bar welding techniques. Both hot plate and hot

bar welding techniques are commonly used process to join plastic based materials in almost all joint configurations (CES EduPack, 2005). While the former can weld relatively thicker plastic with weld strengths approximately equal to that of the parent material, the latter is limited to very thin films having a thickness of less than 0.5mm. In both cases, welding speed depends on section thickness and decreases as thickness increases. They can also be performed on composite components using higher welding forces. Moreover, the pieces of equipment used are available in numerous sizes, thus allowing for both factory and on-site welding (Yousefpour *et al.*, 2004). The final properties of joint in both hot plate and hot bar welding are functions of temperature, time, pressure and material's property (thickness,melting point etc) (Winkler, 2009).

B) Hot gas welding

In hot gas welding of thermoplastics, the bonding surfaces are melted with a hot air/gas stream,then thermoplastic filler rod (the same as the material being joined) is introduced with a slight pressure into a groove cut between two sheets. Upon solidification, the joint is established (Yousefpour *et al*, 2004). Except in few cases, the process is basically manual and somehow similar to oxyfuel welding of metal (Winkler, 2009). However, the basic difference here is, while oxyfuel involves combustion of suitable fuel (gas) to melt a metal,a stream of gas (typically air) passes over an electrically heated element and emerges from a nozzle to melt the polymer (CES EduPack, 2005).

As shown in Figure 2.4, the stream of heated gas is directed towards the joint between two thermoplastic parts, where it melts or softens both the polymer and a filler rod.

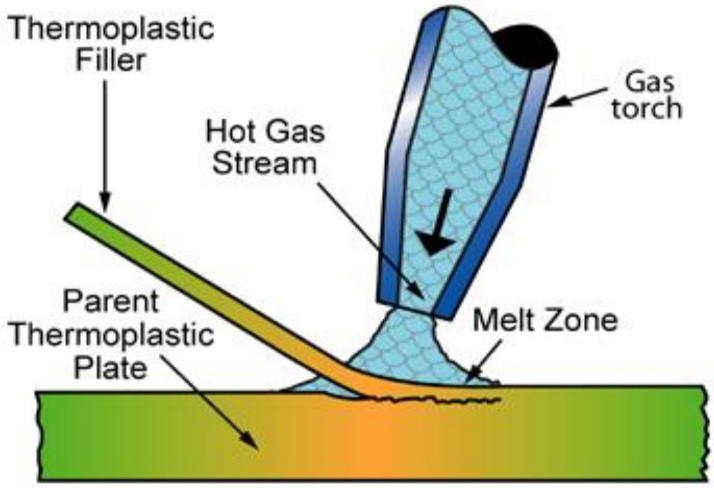


Figure 2.4:Schematic diagram of hot gas welding process(Adopted from grantadesign software 2005)

Hot gas welding find significant application in joining polymers like polyethylene, PVC, Polypropylene, ABS etc, and also find potentials in their blend/composite material (CES EduPack, 2005). Moreover equipment is portable and inexpensive; thus making the process suitable in both installation and repair even on site. However, the technique may not be suitable for lap shear joints (Yousefpour *et al.*, 2004).

C) Infrared welding

The union is achieved by exposing substrates' interfaces to an intense infrared radiation or an electrically-heated ceramic platen for melting or softening to occur. The substrate is subsequently pressed (after removing the heat source) to the end of solidification. This is to say, infrared welding is a non-contact welding technique (Costa*et al.*, 2012).

Infrared technique has an advantage of being a very fast joining process and can even be automated (Yousefpour *et al.*, 2004). As shown in Figure 2.5., the heating, removal and subsequent solidification of polymer parts is an event of seconds (about 5 sec per weld), but is limited due to high tooling cost (CES EduPack, 2005).

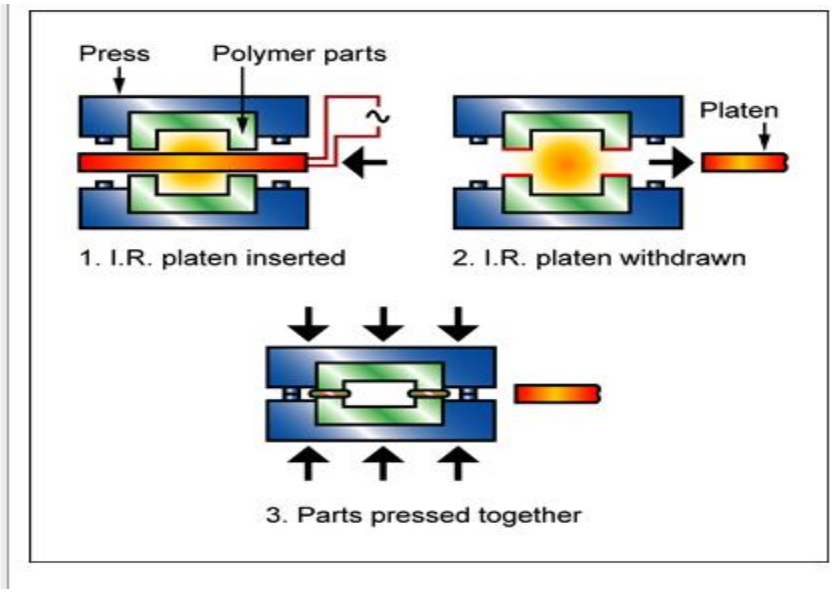
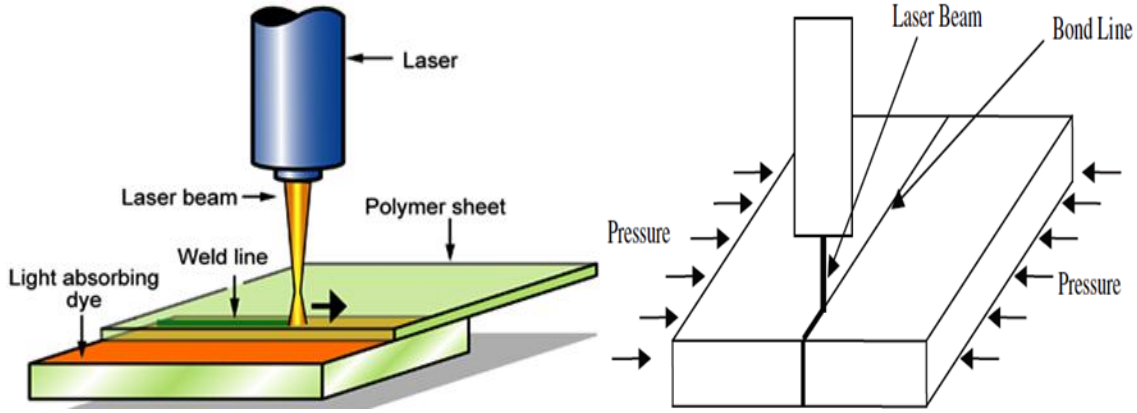


Figure 2.5:A schematic diagram of infrared welding process

(Source: Grantadesign, 2005)

D) Laser welding

Principally, laser welding is known to be a domain of metal fabrication; as metals undergo defined phase transformation upon heating (with vapourization being the worst case), such transformation does not exist in polymer.This may be due to plastics' poor response to intense heating. Plastic upon exposure to laser, get quickly decomposed (burnt) by laser beams even at a low laser power levels (Yousefpour *et al.*, 2004).This together with other plastic behaviours(softening, glass transition etc) limits the use of laser in welding thermoplastic material. However, with proper modification and controlled radiation,the concentrated heat of laserbeams finds application in welding thermoplastic polymer and even thermoplastic composite parts (CES EduPack, 2005).



A B

Figure 2.6: A schematic diagram showing laser welding of thermoplastic materials a) with absorbing surface b) with non-absorbing surface.

(Adopted from grantadesign, 2005 and Yousefpour *et al.*, 2004)

The main requirements of this technique are that, one part of the jointshould be able to transmit the laser beam while the other part should absorb the beam. In the event of non-absorbing surface, the interface is made absorptive by proper coating or pigments addition. As shown in Figure 2.6, the beam passes through the first part and gets absorbed by the other part or the coating, thus generating heat high enough to soften the interfaces. At this point, the two parts are pressedtogether while they solidify to a permanent joint (Buxton, 2002).

Examples of materials that can be joined using laser welding in both reinforced and unreinforced stateincludePolypropylene, Polycarbonate, Acrylic, Nylon, ABS etc. However, the use of laser welding technique is limited since less-expensive alternative welding techniques are availablein most cases (Yousefpour *et al.*, 2004).

2.3.3.Friction welding

In friction welding, the union resulting from melting of the joint interfaces is achieved by friction or vibration. This is to say,the heat that causes the union is generated at the interface between two plastic surfaces as product of their relative mechanical movements (Buxton, 2002). In other words, two thermoplastic surfaces are rubbedtogether until friction generated by spinning motion

develops sufficient heat for fusion of the twosurfaces; while the applied pressure ensure their unity (Winkler, 2009).

The concept behind friction welding is a generation of heat at a joint interface from frictional work under the application of pressure, followed by cooling and consolidation of the polymer. Because of the fact that thermoplastics are poor heat conductors, frictional heat required for (especially for spinwelding) is produced almost immediately while the temperature of the material immediately below the surface of the weld remains unchanged thereby resulting in good joint strength and appearance (Yousefpour *et al.*, 2004).

Several methods (linearvibration welding, spin welding, ultrasonic welding, and friction stir welding) are available to generate heat at the joint interface from frictional work. Moreover, the short joining periods makes this technique unique and applicable to joining both similar and dissimilar materials, e.g. polymer to metals(CES EduPack, 2005).

2.3.4.Electromagnetic welding

This concept of fusion welding utilizes the principles of inductive heatingto a temperature, high enough to cause fusion in ajoint area (Buxton, 2002).The technique consists of moulding opaque powders or insertssuch as iron oxide, stainless steel, ceramic, ferrite and graphite, into thepolymer matrix between the parts to be joined. This bonding materialis placed at the interface of the twoplastic parts, which are then brieflyexposed to an oscillating electromagnetic field. Upon application of a high frequency alternating current (in the case of inductive heating) through a set of conductive work coils, an electromagnetic field is created and in a matter of seconds,the magnetic materials at the weld interface get hot enough to attain fusion temperature and thusthe parts to be joined are fused together (Yousefpour *et al.*, 2004).

If however, polymers with a high dielectric loss factor coefficient are involved, their tendency of converting some of the field energy to heat upon changing of thefield polarity is utilized. Here an intense magnetic field (in MHz frequency region) is applied to the polymer by two electrodesthat are pressed against both sides of the parts to be joined. The combined effect of heat and pressure causes consolidation and emergence of joint(Yousefpour *et al.*, 2004; CES EduPack, 2005).

Various variants of electromagnetic welding techniques are distinguished, they include induction welding,dielectric welding, microwave welding, and resistance welding.

2.4.Review of the Previous Literatures

Czigány and Kiss (2011), studied "Friction Stir Welding (FSW) of fibre reinforced Polymer Composites", they established the effectiveness of Friction Stir Welding in polymeric composites welding. They succeeded in welding 10 mm polypropylene sheet containing up to 30 wt% chopped glass fibre. From the result of their research, it was established that the strength at the welding seam is comparable to the strength of the unreinforced polypropylene (exceeding the strength of unreinforced welded seam by 27%). When compared with the strength of the bulk reinforced composite, the strength value is about 64% of strength of unwelded reinforced polypropylene. However, they attributed this difference to the differential Melt Flow Rate of the two substrates.

Magnus (2012), having identified the limitations of adhesive joining and mechanical fastening processes in the light of joint design, increased weight, increased thickness and stress among other things. He attempted to establish a joining technique for metal to polymer with unlimited design flexibility and high fabrication rate as compared to the aforementioned processes. He investigated the "Feasibility Study of Metal to Polymer Hybrid Joining",andclaimed that Ultrasonic metal-polymer welding seemed to be the most promising method for joining metal to polymer hybrid structures. This technique was employed by him to successfully join metal and fibre reinforced polymers. He attributed the successes recorded to uniform mixing (as observed from morphology study) between the metallic and polymeric partner, which in turn promotes intermolecular contact and mechanical interlocking in the weld zone. He further concluded that high joints strength can be achieved with this joining method at relatively low energy input, and at very short welding times as compared to other welding methods.

Oliveira *et al.* (2010)in a research titled "Preliminary study on the feasibility of friction spot welding in poly methyl methacrylate, PMMA", established a weld of strength values comparable to other available welding techniques, while joining times are equal or shorter. Light optical microscopy and Vickers micro hardness measurements showed the presence of heat affected zone and a thin consolidated stir zone, where physical-chemical transformations related to thermo-mechanical processing led to changes in local mechanical strength.

Barron and Buggy (2000), in their attempt to develop a biomimetic artificial joint; they studied the durability of Carbon Fibre Reinforced (CFR) Polyetheretherketone- Polyurethane adhesive joint in aqueous environment. They bonded CFR to PEEK and established that both failure/degradation is a function of discontinuities due to ingression of water by wicking and diffusion processes.

Johnston of Dukane Corporation examined the suitability of vibration welding in welding amorphous resins such as ABS/PC, PVC, PMMA; semi-crystalline such as HDPE, PP; composites materials such as fibre and particle reinforced. Based on experience along with

experimental result obtained, vibration welding was established to be a promising method in welding wood plastic composite to achieve assembly. This is because, cycle times for assembly are very short and curing time not required.Added to these advantages, is the fact that no mechanical fasteners or expensive glues are required.

Amanat *et al.* (2010) in their effort to develop a biocompatible polymer of reduced permeability so that it survives the chemicallyaggressive *in vivo* environment and thus competing with the existing active implanted devices and at reduced cost; they established in their report titled "Welding methods for joining thermoplastic polymers for the hermeticenclosure of medical devices" laser welding to be the most suitable for the realization of hermetic joint. This is because it requires shorter processing time and the issue of Heat Affected Zone (HAZ) can be neglected.

In a research titled "Machine Crushed Bone as a Partial Replacement of Coarse Aggregates in Lightweight Concrete", Javed *et al.* (2012) produced a lightweight concrete of superior properties to engineering eye, when they replaced up to 50% of normal aggregate with machine crushed animal bone.

Rauf (2014), in his paper "A Review on Mechanical Properties of Animal Bone", presented a report on the unique qualities of bone and thus, the strengthening potentials as filler in composite formulation.

Other outstanding contributions to the domain of polymers/polymer composite and its welding processes include the works of Aigbodion*et al.* (2010),Buxton (2002), Yousefpour *et al.* (2004) and Costa *et al.* (2012). They made an intensive review in both the already established and emerging techniques for joining (welding) of polymer and polymer composites.Although particle reinforced polymers are most friendly in both material and processing cost, less emphasis is paid to particle reinforced polymers welding. Hence, research geared at realizing the full potential of this emerging material cannot be over emphasized.

CHAPTER THREE

3.0 MATERIALS AND METHODS

This chapter described the details of materials, pieces of equipment and experimental method for the evaluation of hot-gas and heated-tool weldments of polypropylene/bone composite.

3.1. Materials

The materials used in this research work include:-

i. Polypropylene pellet (matrix)

ii. Cow's limb bone

iii. Stains remover

v. Aluminium foil

vi. Tap water

vii. Lubricant (premium engine oil SAE 20W/50).

3.2. Equipment

The equipment used in this research work include:-

i. Vibratory Set of sieves

ii. hacksaw, Instron Universal Tester (model 3369)

iii. Charpy Impact (model 412) Machine

iv. Measuring cylinder

v. Thermometer

vi. Marking out tools

vii. Hammer mill

viii. Ball mill

ix. Two roll mill (compounder)

x. Crucible

xi. Muffle Furnace

xii. Drying oven

xiii. A pair of thongs

xiv. Sartorius MC1 digital weighing balance (of 0.0001g accuracy)

xv. Polishing machine

xvi. Scanning Electron Microscope (Phenom ProX)

xvii. Plastic Welding kit.

3.3Experimental Methods

The methodology used for this study is outlined stepwise; from bone procurement to compounding, welding and finally composite testing. Below is the summary of each event as undertaken during the course of the research.

3.3.1. Bone procurement and treatment: Fresh cow bones (limb bones) were collected from abattoir at Akure, Ondo State, washed and cleaned to a good physical condition. The bones were then crushed in a hammer mill. Due to oiliness of crushed bone, the bone was degreased prior to grinding in ball mill. Both crushing and grinding operations took place at Federal University of Technology, Akure (FUTA).

The powdered bone was subsequently heated (calcined) in a muffle furnace at about 600^0C for 45 minutesto drive off volatile matters. The heating was done at a very low concentration of oxygen. This was achieved by heating the bone in a closed crucible. This is because, it was reported by Figueiredo *et al.*(2010)that heating bones in an oxygen-rich atmosphere gives bone ash, which is both chemically and physically quite differentfrom calcined bone and thus, may not possess the properties needed for effective reinforcement.

3.3.2.Bone degreasing: The bone was degreased so as to get rid of any trace of oil or blood that may inhibit its proper bonding with the matrix (polypropylene). The reinforcement (bone) was degreased using water soluble stain remover (diluted methanol). The process utilized the differential weight between the bone and the associating oily substances (gravity separation), hence by pouring hot water (mixed with stain remover) and allowing it to settle, the two phases got separated by decanting out the oily water. This process was repeated using hot water and later cold water without stain remover several times until clean (shallow) surface is observed at the surface of the water. The powder was later dried in a drying oven at 105 ^{0}C.

3.3.3.Sieving of bone: The bone powder was sieved to achieve the needed particle size. An Octagon Vibratory set of sieves was used. 950g of the powdered bone (after secondary grinding) was charged in the first sieve, put on and allowed to vibrate for 15 minutes. After sieving, the undersize of 75µm were used as reinforcing phase.

3.3.4. Compounding of mix: The matrix and reinforcement were compounded or mixed usingtwo roll mills at Nigerian Institute of Leather and Science Technology (NILEST), Zaria. This was done to ensure intimate mixing of composite forming constituents. The machine (two roll mill) was switched on and set to a preheat temperature of 180 degree Celsius for an hour. The matrix, polypropylene (Grade HPIG110 sourced from INDORAMA/Eleme Petrochemical Company Limited, Rivers State) was then introduced when the two rolls were just closed by regulating the gap between them. After two minutes, a measured amount of filler (bone particles) was introduced. The mixture was left for about three minutes for effective homogenization. The mixture was then ejected in a rectangular mould and pressed to shape usinghot press.

3.3.5.Cutting: The Mix, after cooling, was cut to obtain required sizes for various tests using hacksaw and marking out tools. The cut samples were divided into two groups, the first was used as control, while the other was tested after welding.

3.4Welding of Composite

The choice of welding process was done with the aid of material/process selection software (CES EduPack, 2005), and in line with the requirements of International Welding Engineers (IWE) guidelines for plastics welding. The welding processes undertaken in this research were hot gas and heated tool welding technique.

Having cut the composite into desired pieces, the composites were fusion welded using hot gas and heated tool welding processes. Here, the union of the pieces was achieved by heating the surfaces to be joined such that they flowed and fused together.

3.4.1.Hot gas welding: It involved directing a stream of hot air at the interfaces to be joined, while the substrates were joined through the use of filler material. Here, a single V-weld was considered. Each sample (suitable for a given test) was cut at halfway its length. A bevelled edge was created in line with IWE guidelines for plastics joining.

A very thin section of each composition was cut with a snip and used as a filler material. This is to ensure that the filler got melted at infinitely higher rate when compared with the interfaces being joined. As such, when the hot air (from air gun) at about 250 ^{0}C (so that the temperature at the substrates' interface is approximately 175^{0}C) is blown onto the substrate, the melted filler penetrated and filled the created bevelled edges, and upon cooling, the weld was produced. Each weldment was visually examined and the defect free ones were tested for the properties desired.

3.4.2. Heated tool welding: The cut samples (as in the case of hot gas process) were welded. The substrate's thickness was 3.3 mm, hence surface preparation was not emphasized. The temperature of the heated tool was monitored with a thermometer and at about 175^{0}C, a heated tool was then placed between the parent materials being joined. The tool was later withdrawn and the two substrates were brought to contact under slight pressure. After joint consolidation, visual examination was again used to screen out welds with visible defects.

3.5. Testing of Composites

Somephysical and mechanical properties of the composites before and after welding were determined in accordance with standards. These properties included density, water absorption, tensile and flexural strength,percentage elongation, impact energy, hardness and surface morphology.

On the basis of comparison, the effect of reinforcing phase (bone addition) on weldability of polypropylene/bone composite was established by comparing the polymer's properties in unreinforced, reinforced and welded conditions. Both welded and unwelded composite were subjected to the same set of tests. The behaviours of welded seam were later compared with the bulk composite. Moreover, the welding seams of both reinforced and unreinforced polypropylene were compared. This was to ascertain the net effect of reinforcement.

3.5.1. Densitydetermination:A suitable sample of a known mass,**w** was cut out from the bulk composite. Itwasimmersedin a measuring cylinder, half filled with water and the increase in volume, **v**, recorded, gave the volume of the sample (Archimedes' principle). The density of each sample **(ρ**) was obtained using the following equation:

$$\rho = \frac{w}{v}(g/cm^3) \quad \text{……. ……..……....3.1}$$

3.5.2.Water absorption Test: Water absorption test was performed using ASTM D-7031 -04 reported by Ghasemi and Behzad (2009) as a guide.Sample of dimensions25mm x 10 mm x 3.3mmwas cut,dried at 105 ^{0}C in an oven, weighed as$\mathbf{w_i}$and placed in aclosed container(kept at room temperature) containing water for 336 hrs. After every successive 24 hrs, each sample wasdried (by wiping) and weighed as $\mathbf{w_f}$. The percentage weight gained (corresponding to the amount of water absorbed) was calculated and recorded for each sample, and hence the percentage water absorbed was calculated using the following formula:

$$\%\text{weight gained} = \frac{Wf - Wi}{Wi} \times 100\% \quad \text{.....3.2}$$

3.5.3. Tensile Test: Tensile test was carried out using Instron 3369 universal tensile tester. A "Dog-bone"standard sample as specified by ASTM (test method D638) was prepared. The increased width of the specimen in the gripping zones was to ensure that the specimen breaks at a failure zone between the grips (Brent, 2008). Each sample was subjected to test by loading to its maximum load carrying capacity. The test involved gripping the test sample with lower (stationary) grip, while the upper (movable) grip was lowered to grip the upper part of the specimen. A word of command for tensile test moved the upper grip up and thus pulling the specimen to breakage in accordance with the set strain rate (crosshead speed of 20 mm/ minute). The strength value was read directly from the computer attached to the machine.

Moreover, other tensile properties (elongation and strain) werealso obtained from this test asread directly from the computer.

3.5.4.Flexural/Bend Test: A sample of dimensions 120 mm x 10 mm x 3.3 mm was prepared; a span of 60 mm was marked and placed between two supports, and a load was applied at the centre as specified by ASTM D790.

Here three point bend test accessories were attached to the Instron machine. The simply supported (by lower arm)composite beam was loaded with a bending force by punch (upper arm) of the machine. The test was continued until the specimen failed. The test result was digitally captured from the computer set.

3.5.5.Hardness Test: Hardness test is aimed at establishing resistance to deformation by indentation. A shore D durometer was used to determine durometer hardness value,in accordance with ASTM D2240.The minimum thickness of 6 mm was achieved by placing two layers of the composite (each 3.3 mm thick), and arrived at anapproximately 6.6 mm thickness. Three samples per composition were tested; they were indented for aboutfive seconds (5s) with durometer indentor. A total of five indentations were made (on both welded and unwelded areas) and an average was computed.

3.5.6.Impact test: For this test, Charpy impact testing machine 412 was used with 15 joules pendulum.A V-notch was created(45 degrees) into a sub-size sample with dimensions55mm x 10mm×3.3 mm at halfway its length (i.e. 27.5mm). The notch was created for stress concentration during the impact test. The sample was then placed on the machine, and the pendulum was allowed to hit the specimen as it swings under gravity. The impact energy was obtained by reading energy loss of the pendulum as a result of hitting the sample directly from the machine.

3.5.7.Scanning Electron Microscopy (SEM): A flat sample was prepared so that electrons were reflected uniformly when they struck on the sample. Samples were made conductive by coating themwith cobalt solution.

This test was carried outusing Phenom proX Scanning Electron Microscope.An electron beam was focused on the condenser lenses to a spot as it passed through pairs of scanning

coils/deflector plates in the electron column. The electron beam got deflected (in the final lens) into the ***x***and ***y*** axes so that it scanned in a raster fashion over a rectangular area of the sample surface.

The energy exchange between the electron beam and the sample yielded a reflection of high-energy electrons(detected by specialized detectors). The beam current absorbed by the specimen was detected, used to create imagesand with the aid of electronic amplifiers, specimen morphology was established. The image was captured from the computer attached to the Scanning Electron Microscope and presented under SEM results.

3.5.8.Soil Burial Test: Soil burial test was conducted using similar procedure as reported by Khalil *et al.*(2010) and Potts *et al.*(1972) as a guide. Samples of dimensions40 mm x 10 mm x 3.3 mm were prepared; each sample was weighed as $\mathbf{w_i}$ and later buried completely to a depth of 10 cm in a plastic bucket filled with natural soil.The soil was kept moist by sprinkling water at regular intervals. The filled bucket (with the samples buried) was kept under prevailing room temperature. After two weeks (336 hrs), the samples were removed, cleaned and weighed as $\mathbf{w_f}$.The weight loss (percentage) for each sample was computed and presented under results and discussions.

CHAPTER FOUR

4.0 RESULTS AND DISCUSSIONS

4.1 Introduction

This chapter presents the results of the physical and mechanical tests conducted in the course of this research.For ease of results presentation and referencing, the composite prior to welding (unwelded) will be referred to as UWC while Heated Tool and Hot Gaswelded composite will be respectively referred to as HTW and HGW, unless otherwise stated. When referring to the matrix (polypropylene), PP will be used. All scientific symbols and constants retain their conventional description.

4.2Density

Figure 4.1 shows the variation of composite's density with increasing reinforcement percentage.

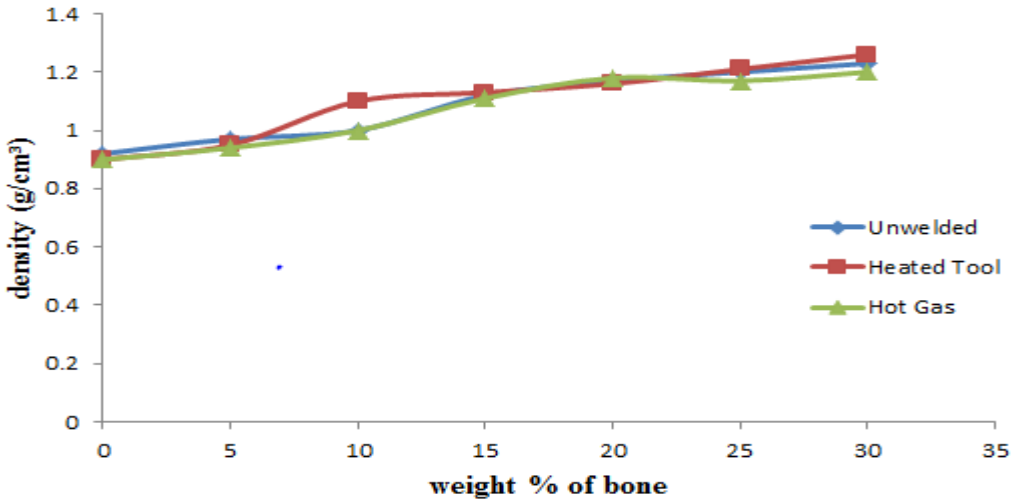


Figure 4.1: Variation of composites' densities with increasing percentage of reinforcement

The density of the composite rises with an increase in weight % of bone (Figure 4.1), owing to the fact that bone is denser than PP. Although there was general increase in densities of composites with increasing bone fraction; however, there were no significant differences among UWC, HTC and HGW samples. The increase in densities observed as PP is being replaced by bone powder is in line with the trend reported by Samir (2012).

4.3Water Absorption Figures 4.2 (a to g) show the variations of water absorbed by composites with time of immersion of various composite formulations.

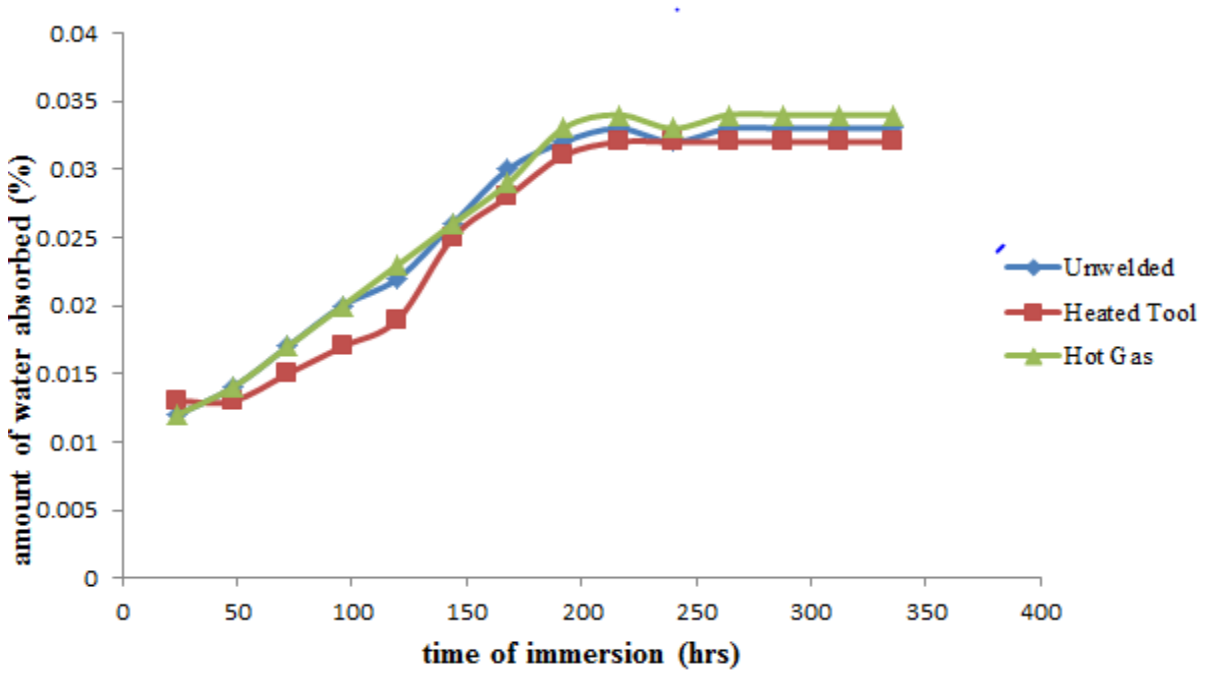


Figure 4.2a: Variation of water absorbed with time of immersion of PP/0% bone composite

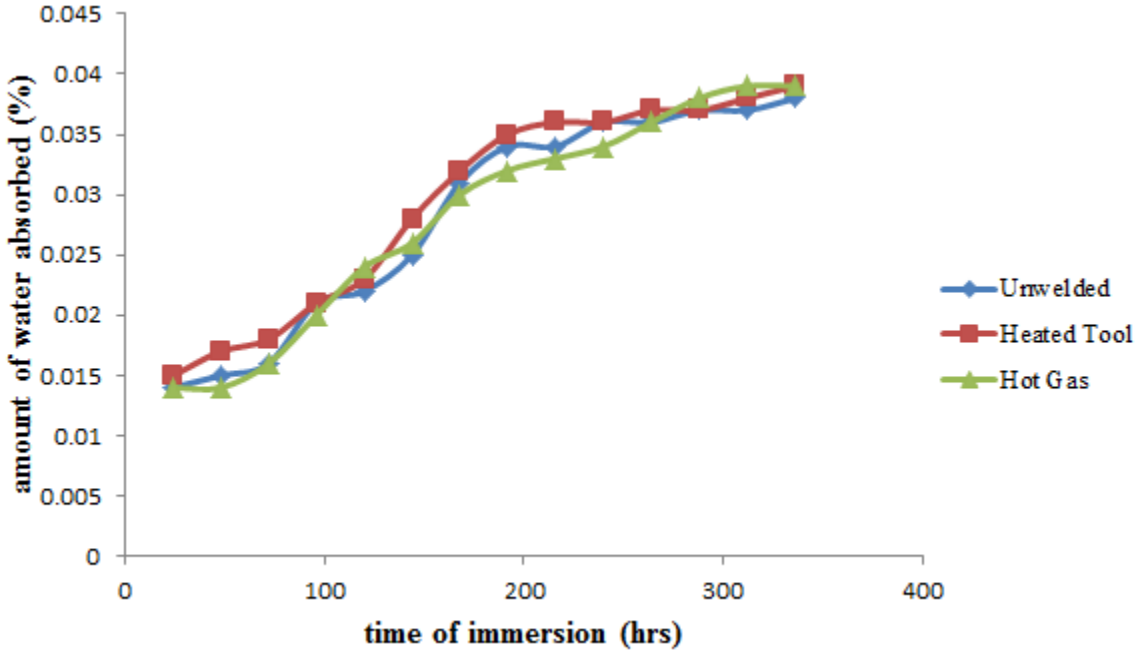


Figure 4.2b: Variation of water absorbed with time of immersion of PP/5% bone composite

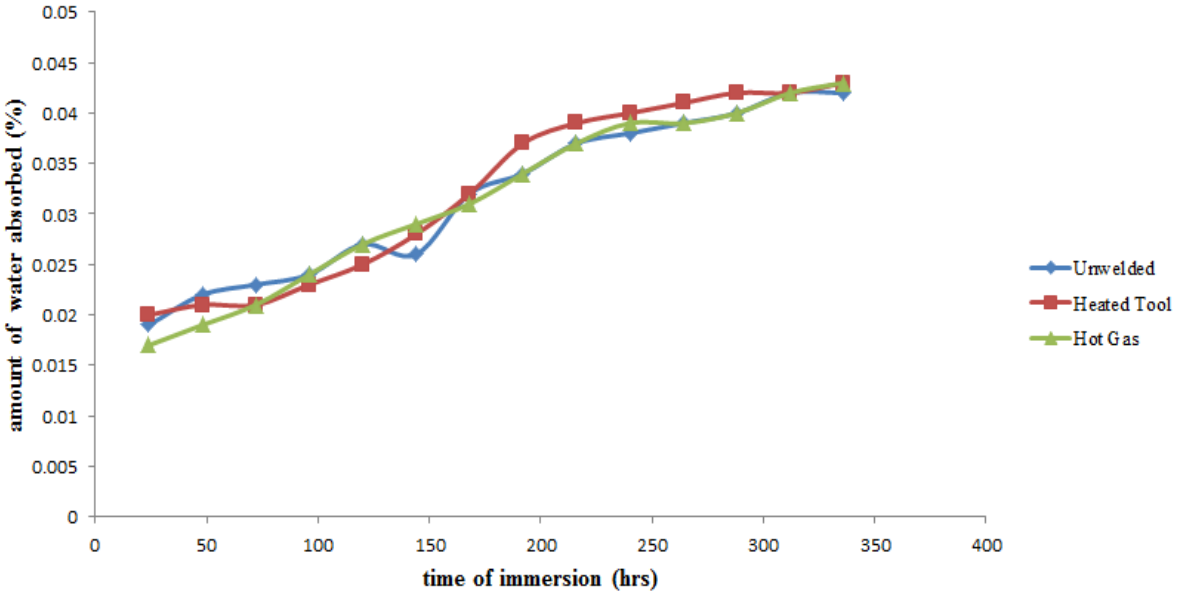


Figure 4.2c: Variation of water absorbed with time of immersion of PP/10% bone composite

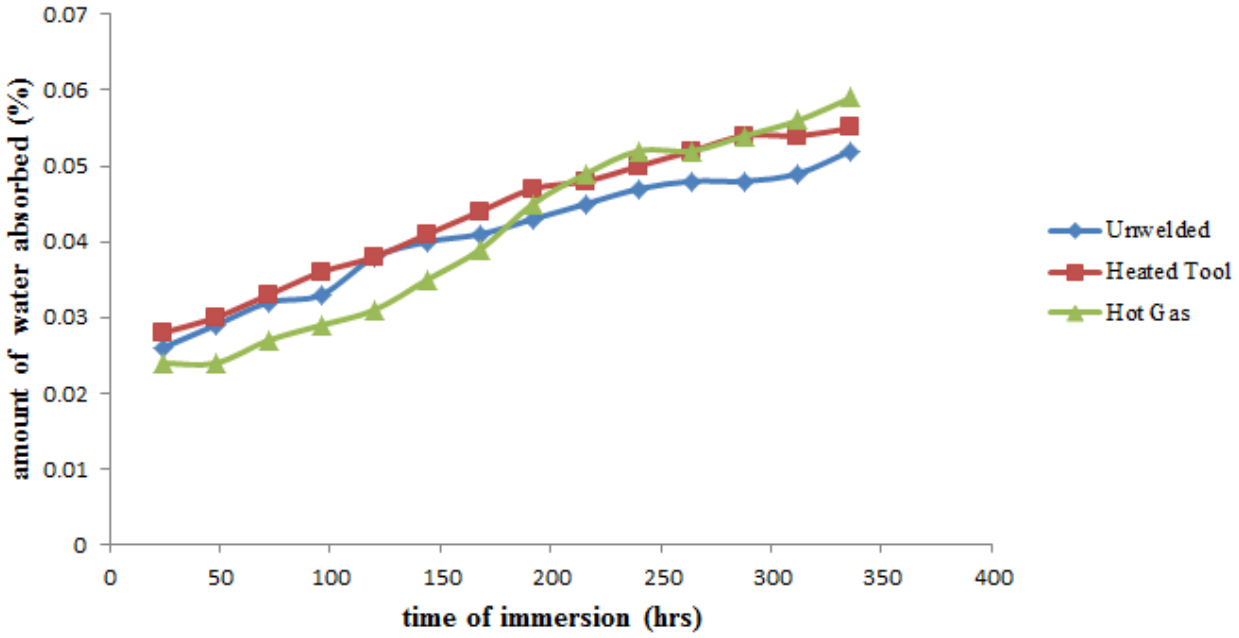


Figure 4.2d: Variation of water absorbed with time of immersion of PP/15% bone composite

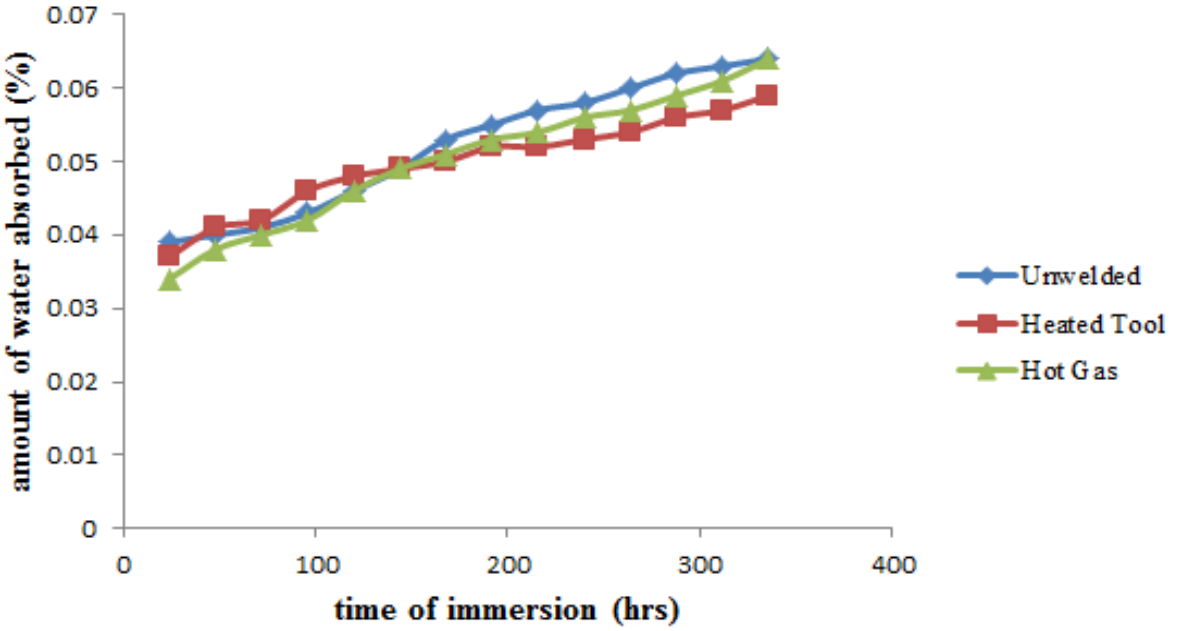


Figure 4.2e: Variation of water absorbed with time of immersion of PP/20% bone composite

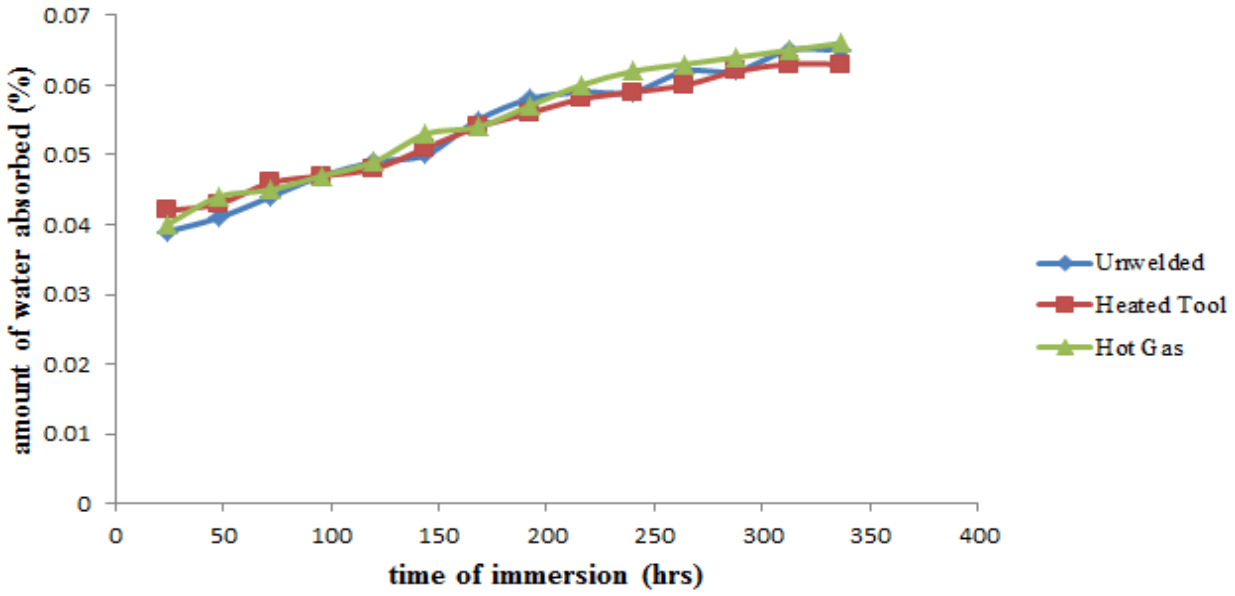


Figure 4.2f: Variation of water absorbed with time of immersion of PP/25% bone composite

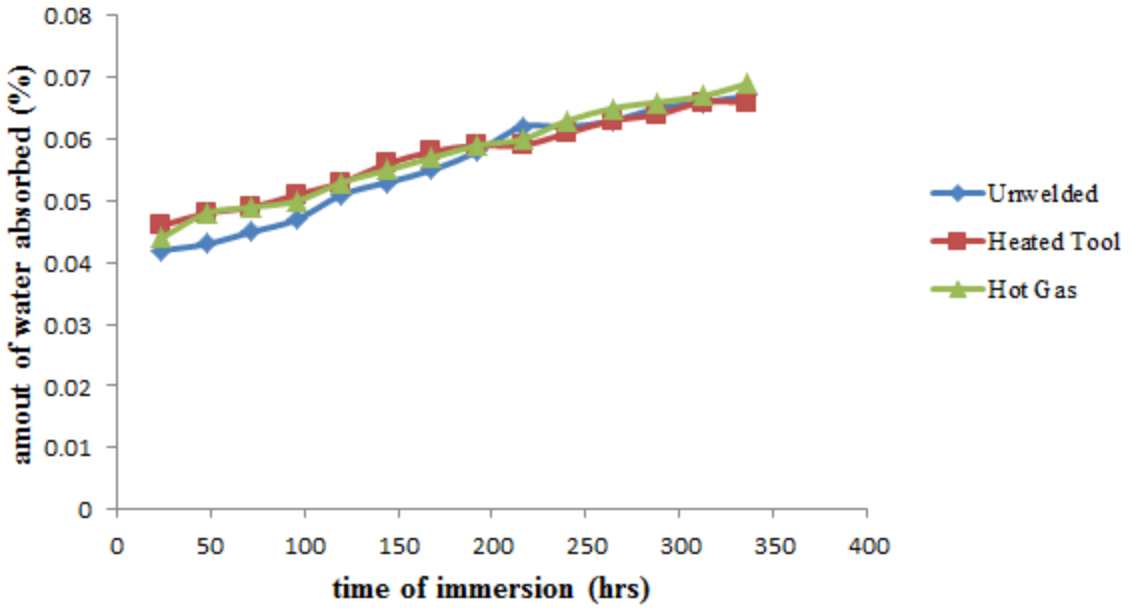


Figure 4.2g: Variation of Water Absorbed with time of immersion of PP/30% bone composite

With reference to Figures 4.2(a to g), it can be seen that there is an increase in the amount of water absorbed as the period of immersion proceeded. After 192 hrs of immersion, PP with 0% reinforcement appeared to have gotten saturated as seen in Figure 4.2a. However, as the percentage of bone powder increased, there were increases in water uptake. This is because, the matrix (PP) and the reinforcement (bone) are not in any way soluble in one another, and interfacial bonding between the bone and PP may not be perfect. Further addition of bone powder might thus have weakened the bonding further; this coupled with bone's hydrophilicity resultedin higher water absorption as observed with increasing filler content. Despite this increase with increasing bone % and period of immersion, there were no significant differences among UWC, HTC and HGW samples. Similar trends were observed by Zhang *et al.* (2014) and Ghasemi and Behzad (2009).

4.4Tensile Strength

Figure 4.3 shows the variation of composite's tensile strength, with increasing reinforcement percentage.

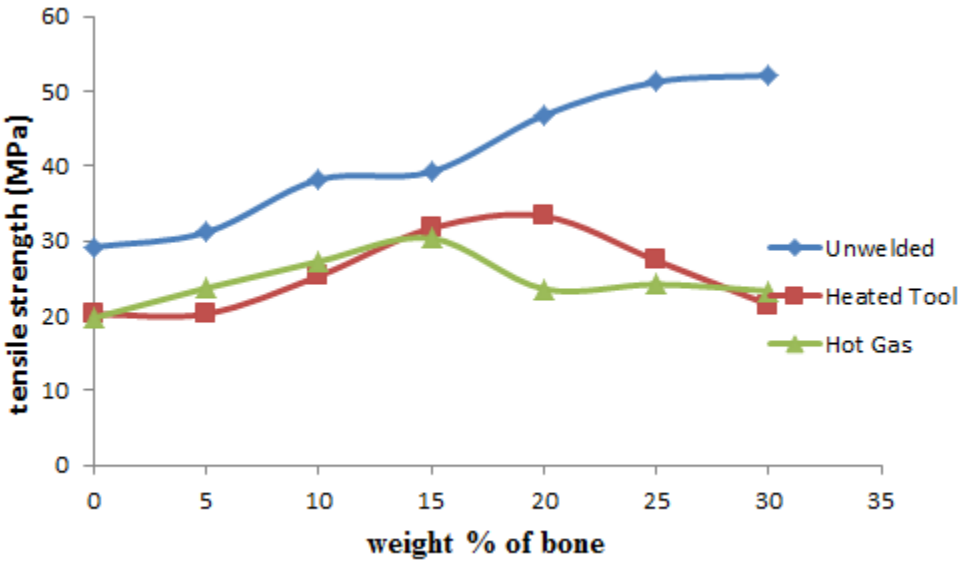


Figure 4.3: Variation of tensile strength with increasing percentage of reinforcement

As the weight percentage(wt%) of reinforcement (strong particles) increased, there is a corresponding increase in strength (Figure 4.3); this may be due to strengthening effect of a strong and hard reinforcing phase (bone). As the volume fraction of the reinforcement increased, so also the strengthening effect increased. UWC saw a corresponding increase up to 30% (57.02 MPa) of reinforcement addition.The increase in tensile strength with increase in filler contents is

in agreement with the findings of Singha and Kumar (2008), Nwanonenyi *et al.* (2013) and Zhang *et al.* (2013), and the unique properties of bone as reported by Javed *et al.* (2012) and Rauf (2014). Relatively lower strength values were obtained from HTW and HGW samples when compared to UWC. This may be explained using the fact that weldment has been reported to be a region of weakness (Czigány and Kiss, 2011; Matthews and Rawlings, 2005) and may act as stress riser.

Despite the overall improvement in tensile strength, drop in property after 15% (equivalent to 40.91 MPa), and 20% (equivalent to 41.54 MPa) in Heated Tool Welded (HTW), and Hot Gas Welded (HGW) samples respectively were recorded. These values showed (when compared with UWC) that, at 15% of reinforcement addition, HTW had strength value 16.70% lower than UWC of the same composition (15% bone). On the other hand, at 20% of reinforcement, the strength value of HGW was found to be 23.23% lower than UWC of the same composition. Furthermore, at 15% of bone addition, HTW has a strength value of 9.59% higher than UWC with 0% reinforcement, while HGW was found to be 11.28 % higher than UWC of 0% reinforcement. This is to say that, the goal of reinforcement is not defeated by the welding processes. Similar trend was observed by Czigány and Kiss, (2011).

4.5 Percentage Elongation

Figure 4.4 shows the variation of composite's elongation at yield with increasing reinforcement percentage.

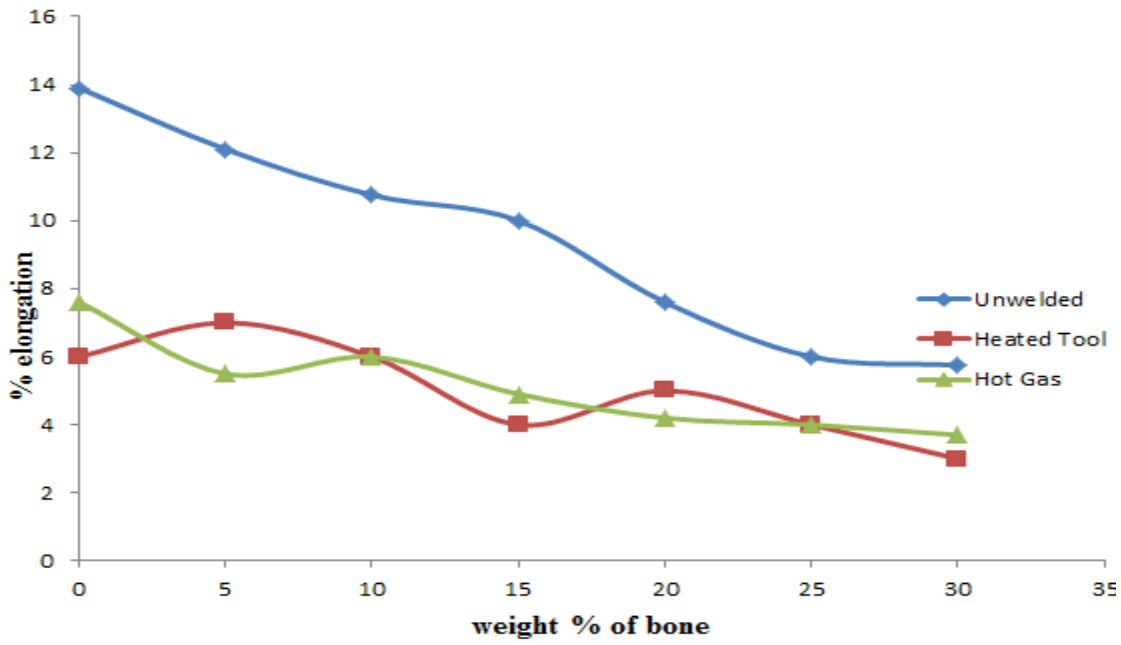


Figure 4.4: Variation of % elongationwith increasing percentage of reinforcement

The effect of bone particles on composites' elongations are shown in Figure 4.4; as the percentage of bone powder increases, % elongation decreases. This is because there could be loss in ductility in the composite (due to strengthening effect) with increase in percentage of reinforcement. This result is also in agreement with other findings, such as Nwanonenyi *et al.* (2013) and Samir (2012). The lower elongations observed in the welded samples may be attributed to the presence of weldments. These weldments may serve as a stress riser and thus preferentially deformed to failure before the parent composite. As such, the extent of straining prior to failure of the welded samples was relatively lower than that of the unwelded composite.

4.6Flexural Strength

Figure 4.5shows the variation of composite's flexural strength with increasing reinforcement percentage.

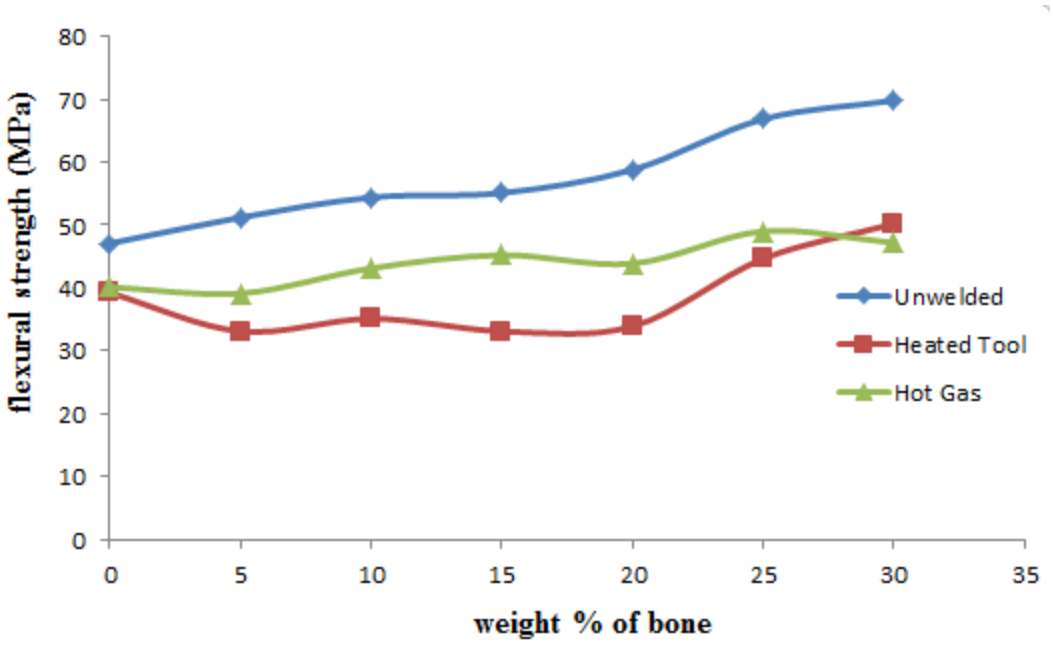


Figure 4.5: Variation of flexural strength with percentage of reinforcement

It was observed that (Figure 4.5), as the weight of calcined bone increased in the composite, the flexural strength also increased. Bearing in mind that bone is strong (Rauf, 2014) and can stiffen (Javed *et al.*, 2012); as a consequence, increase in the volume fraction of bone powder yielded a corresponding increase in flexural strength of composites. The relatively lower flexural strength values obtained in HTW and HGW, when compared with UWC may be due to presence of weldment (stress riser), which has been reported as weak region by Czigány and Kiss (2011), and Matthews and Rawlings (2005). This may be the reason why both HTW and HGW failed at their respective weldments, under bending conditions.

4.7 Impact Energy

Figure 4.6 shows the variation of composite's impact strength with increasing reinforcement percentage.

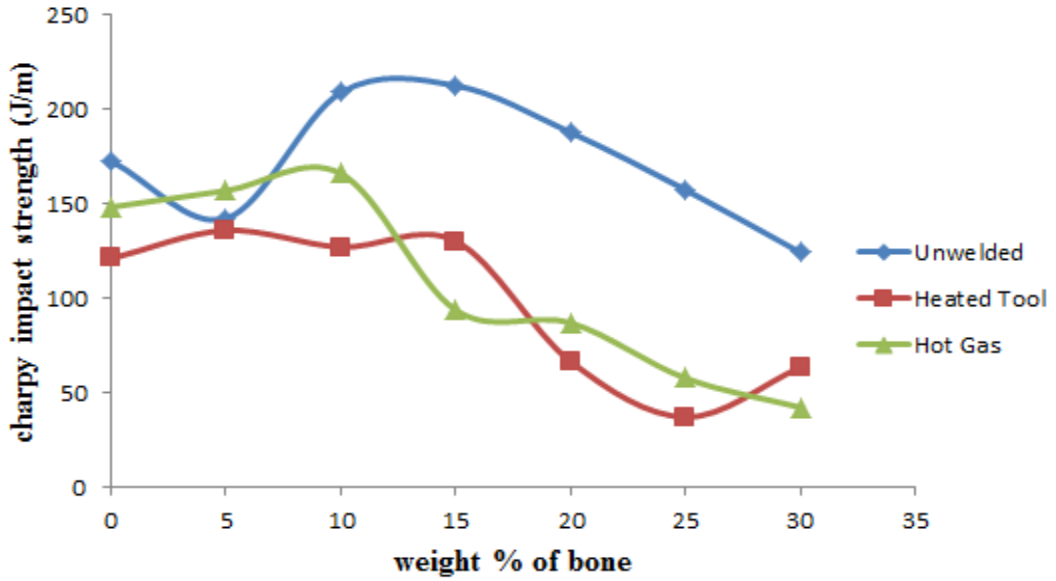


Figure 4.6: Variation of impact energy with increasing percentage of reinforcement.

From the result of impact test (Figure 4.6), it was found that as the wt% of bone increased, the energy absorbed on impact decreasedand then increased and finally dropped. The initial stage upon which it dropped may be due to introduction of little amount of particles (bone powder). The rise in the amount of energy absorbed may be due to the amount of particles within those ranges, sufficient to result in an inter-particle spacing, which often tends to slowdown the initiation of cracks by absorbing some fraction of energy. Further addition may lead to particle-particle interaction, possible clogging of particles and hence, reduce the ability of composite to withstand impact. This trend is in line with the initial reports by Nwanonenyi *et al.* (2013).

4.8Hardness

Figure 4.7shows the variation of composite's flexural strength with increasing reinforcement percentage.

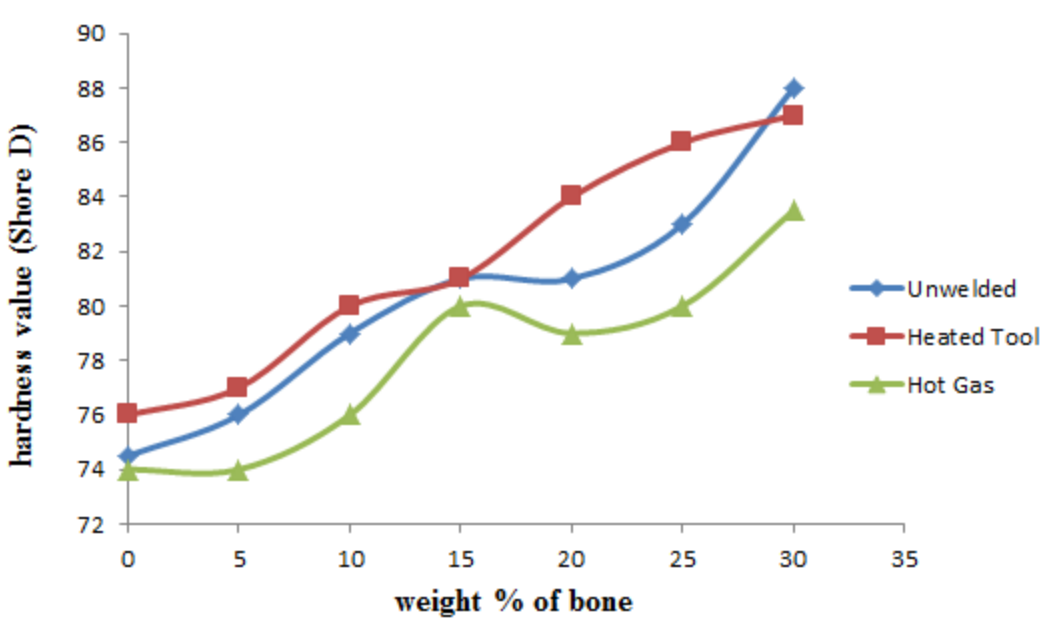


Figure 4.7: Variation of shore hardness value with increasing percentage of reinforcement.

From the graph of hardness: Shore D (Figure 4.7),hardness value of the composite increased as a result of increase in hard and strong phase(bone powder) in the PP matrix.This may be attributed to the fact that the addition of bone powder into plastic stiffened and improved the matrix surface resistance to indentation. This result is in agreement with many research findings such as: Nwanonenyi *et al.* (2013) and Vallo (2000). When welded, HTW was relatively harder than HGW samples. This may be due to the forging pressure used in Heated Tool Welding, which was not present in Hot Gas Welding; as such, this could result to more mechanical interlocking between the matrix and the reinforcement, and hence, superior properties.

4.9Soil Burial Test

The Figure 4.8shows the variation of composite's weight loss with increasing bone percentage for the period of two weeks.

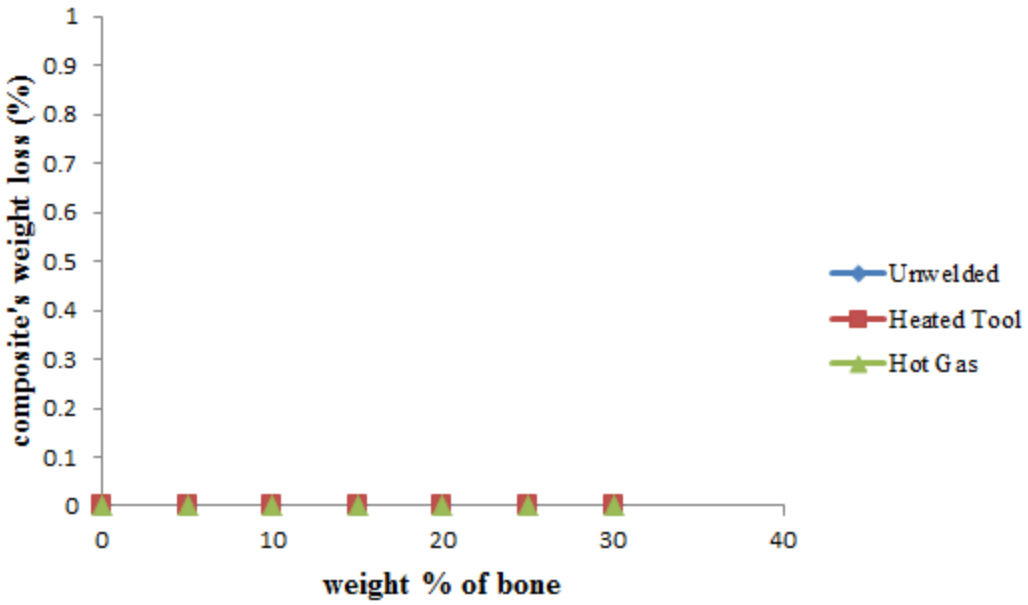
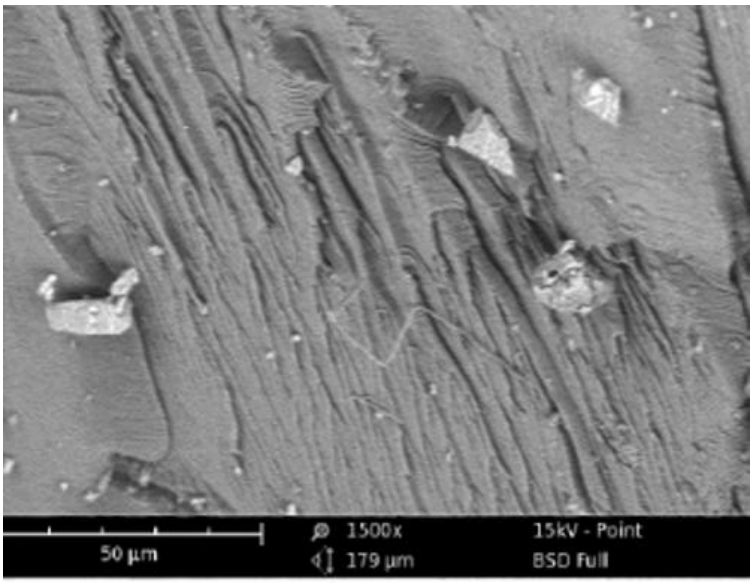


Figure 4.8: Variation of weight loss in soil burial with increasing percentage of reinforcement.

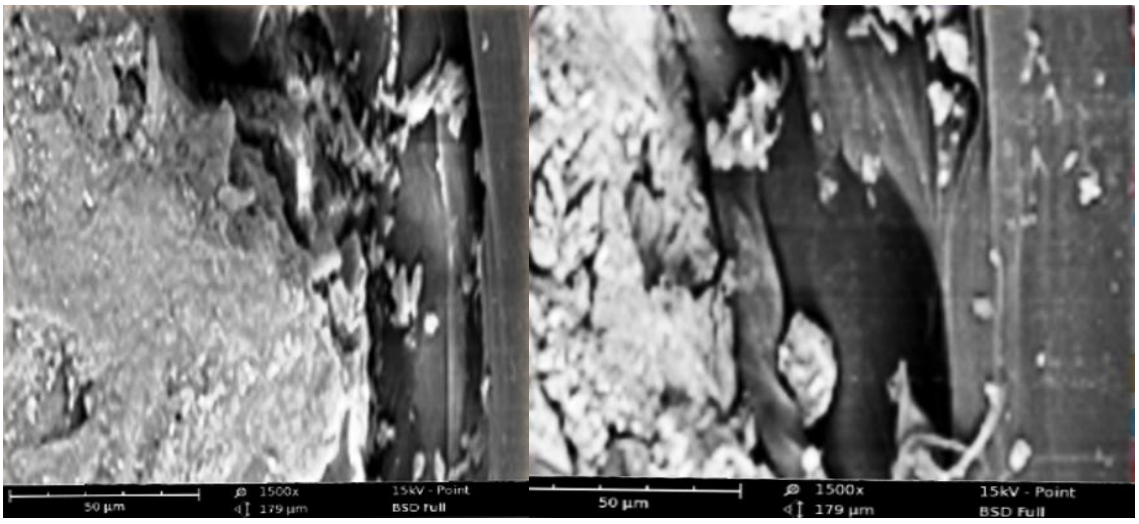
From the Figure 4.8,there was no loss in weight of the composite as function of exposure time. This may be due to "relative inertness of polymers" especially over very short period of time. Hence,it can be deduced thatno degradation was recorded in the composite due to burial in natural soil for the period of two weeks (336 hrs) covered by the test.

4.10Scanning Electron Microscopy (SEM)

The micrographs obtainedfrom morphological studies using SEM are presented in Plate 1, Plate 2 and Plate 3 below:-

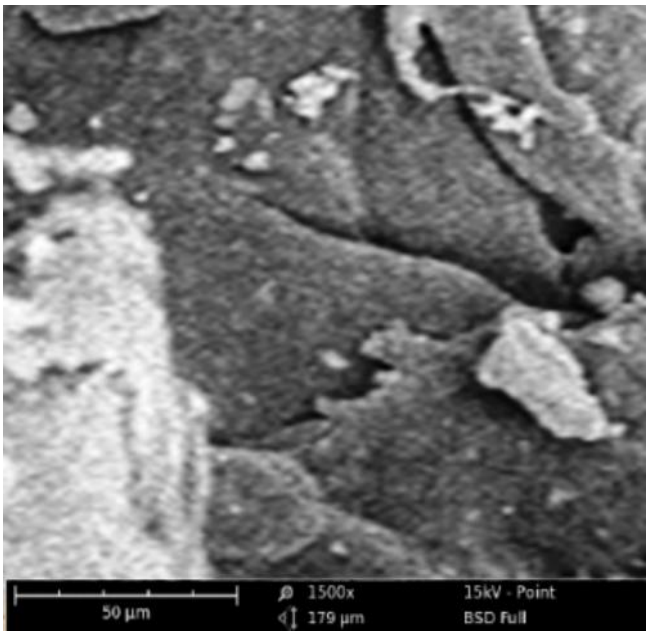


(a)

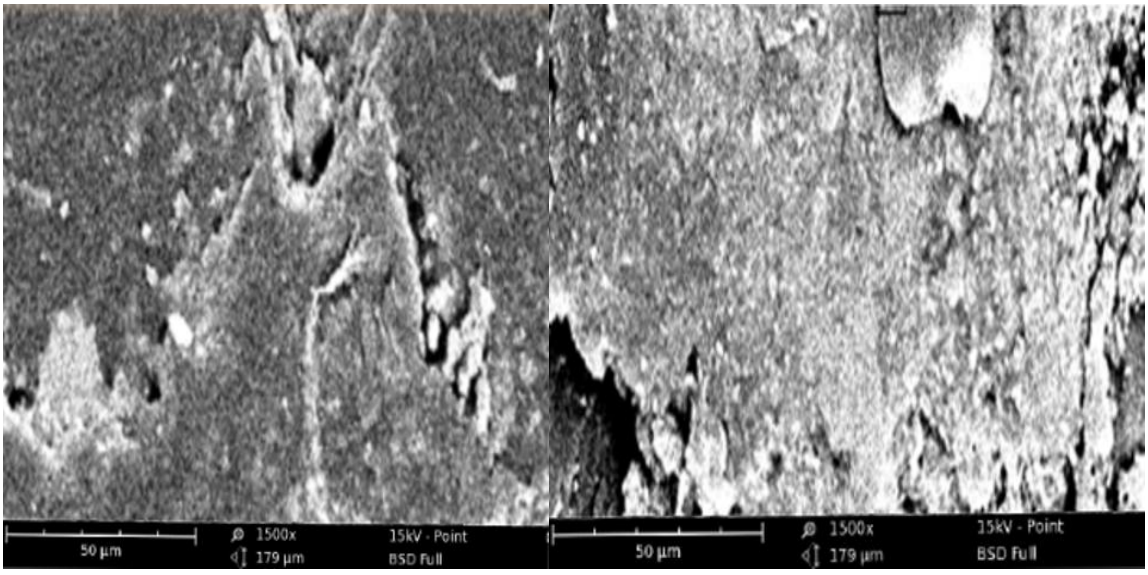


(b) (c)

Plate 1: SEM micrographshowing morphology of PP with no reinforcement. a) In unwelded condition. b) Hot gas welded region showing welding effect. c) Heated tool welded region showing welding effect. (x 1500)

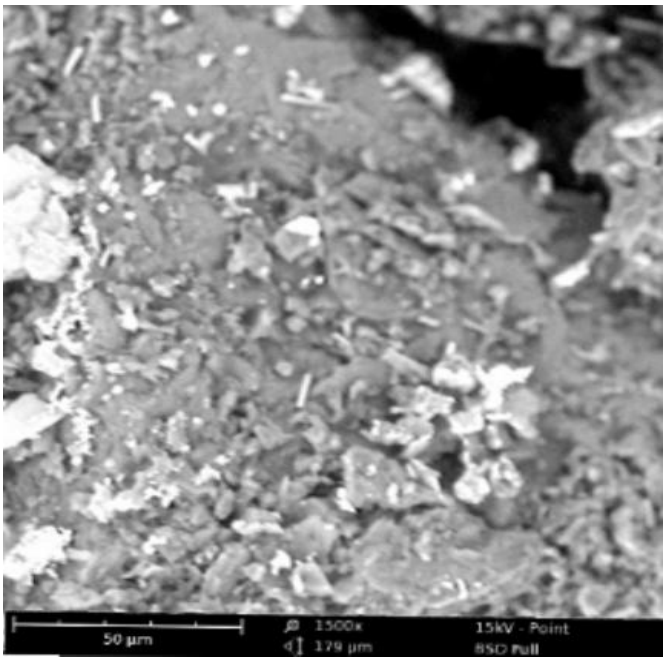


(a)

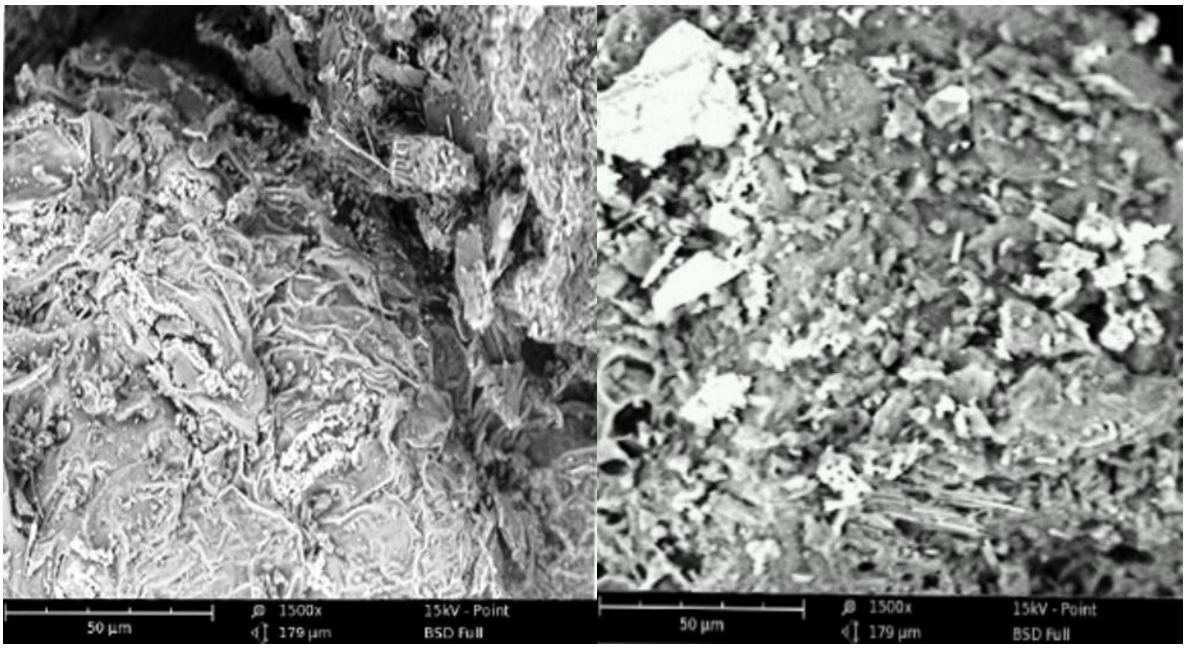


(b) (c)

Plate 2: SEM micrograph showing distribution of reinforcement in PP containing 15% reinforcement.a) In unwelded condition. b) Hot gas welded region showing welding effect. c) Heated tool welded region showing welding effect. (x 1500)


50 µm
1500x
179 µm
15kV - Point
BSD Full

(a)


50 µm
1500x
179 µm
15kV - Point
BSD Full
50 µm
1500x
179 µm
15kV - Point
BSD Full

(b) (c)

Plate 3: SEM micrograph showing distribution of reinforcement in PP containing 30% reinforcement.a) In unwelded condition. b) Hot gas welded region showing welding effect. c) Heated tool welded region showing welding effect. (x 1500)

It can be seen from the SEM images presented above (Plate 1, Plate 2 and Plate 3)that on the basis of comparison, there are some level of distortions in the weldments, relative to the unwelded structure. This may be the cause of relatively lower values in tensile strength, flexural strengthand impact energy but allowed for more level of water to be absorbed due to loose structure.

At higher percentage of bone powder, it was evident that more fraction of polypropylene (PP) was replaced by the bone particles. This might have led to more particle-particle interaction which reduced the ability of the matrix to encapsulate the filler, and adversely lowered the ability of the composite to withstand loading.Further additions of filler up to 30% revealed more particles with more pronounced discontinuities. This may be another reason for further decline in tensile, flexural and impact strength level.

CHAPTER FIVE

5.0 CONCLUSIONS AND RECOMMENDATIONS

5.1. CONCLUSIONS

From the research findings, it can be concluded that:

- Thecow limb's bone was procured, treated and used for the production of calcined bone powder of -75µm particle size. Subsequently, the powder was used to reinforce polypropylene and hence, polypropylene/bone particulate composite produced.
- It is possible to weld polypropylene/bone particulate composite with up to 30% of calcined bone powder into a butt joined structure by Heated Tool and Hot Gas Welding Techniques.
- Polypropylene/bone composites with up to 15% and 20% can respectively be welded using Heated Tool and Hot Gas Welding Techniques without drop in tensile strength.
- Compared to the individual properties of the constituents forming the composite, desirable properties of the composite are enhanced due to utilization of properties of interest in both the matrix and the reinforcement. More so, specific properties are enhanced.
- Water absorbed by the composite (in all conditions considered) increases as both the function of days of immersion and the amount of filler. Though polypropylene appear to be saturated after 192 hrs of immersion, the reinforced (with bone power) continue to absorb water as more bone powder was added.
- From the research findings, the welding processes have no significant effect in the composite's density and water absorption behaviour.
- The welding process can be used to achieve assembly/repair of the polypropylene/bone composite. The processes will be especially effective where hardness is the parameter of interest. In cases where tensile properties are of key interest, the welding processes used should not be encouraged. More so, the composite will be especially vital in applications

such as automobile interior and other dry applications. This is due to increased hydrophilicity displayed by the composite.

5.2 . RECOMMENDATIONS

In future research, the following can be considered:

- The possibilities of conditioning the reinforcing phase (bone powder) used should be investigated. Subsequently, the effect of such treatments be ascertained. This is because, a number of reinforcing phases were reported to have displayed better reinforcement when treated than when used untreated.
- The use of coupling agents, compatibilizers and other additives that increase hydrophobicity be considered. This can serve as an attempt to lower the composite's water uptake.
- Having used IWE welding parameters as specified for polypropylene and other olefins, in future, welding parameters be investigated with regard to the processes and the material being handled. This is to establish (if any) the optimum condition upon which such a weld can be produced.
- Root penetration, Heat Affected Zone(HAZ) etc, should be investigated.
- The composite's properties as a function of water absorbed be established. This is to establish its reliability, considering its hydrophilicity.More so, soil burial test be conducted for a much longer period. This is to ascertain the composite's degradability by microbes.

5.3 CONTRIBUTIONS TO KNOWLEDGE

From the outcome of this study, the following contributions have been made:

- Another material (PP/Bone Composite) of high specific properties has not only been developed, but also fabricated by welding.
- Being a joining process, the success recorded will go a long way in converting this composite into a real engineering material.
- From the outcome of this research, possibility of repairing this composite and other related composites have been brightened.
- From the design point of view, a point of caution has been drawn with respect to composite formulation, that while reinforcement is appreciated to improve the propertiesof interest, other vital properties such as weldability are often compromised.

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%20Fiber%20Reinforced%20Polymer%20Composites%20for%20Large-scale/Th11-4-AF0685.pdf.

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APPENDIX 1: LIST OF TABLES

TABLE 1: Result of Density determination

S/NO	Wt % of bone powder	Density of UWC (g/cm^3)	Density of HTW (g/cm^3)	Density of HGW (g/cm^3)

1	0		0.92	0.90	0.90
2	5		0.97	0.950.94	
3	10		1.00	1.101.00	
4151.121.131.11					
5201.171.161.18					
6251.20		1.211.17			
7301.231.261.20					

TABLE 2: Result of water absorption test

Time of immersion in hrs	Water absorbed by respective UWC (x 10^{-3}%)	Water absorbed by respective HTW (x 10^{-3}%)	Water absorbed by respective HGW (x 10^{-3}%)
	0 5 10 15 20 25 30	0 5 10 15 20 25 30	0 5 10 15 20 25 30
24	12 14 19 26 39 39 42	13 15 20 28 37 42 46	12 14 17 24 34 40 44
48	14 15 22 29 40 41 43	13 17 21 30 41 43	48 14 14 19 24 38 44 48
72	17 16 23 32 41 44 45	15 18 21 33 42 46 49	17 16 21 27 40 45 49
96	20 21 24 33 43 47 47	17 21 23 36 46 47 51	20 20 24 29 42 47 50
120	22 22 27 38 46 49 51	19 23 25 38 48 48 53	23 24 27 31 46 49 53
144	26 25 26 40 49 50 53	25 28 28 41 49 51 56	26 26 29 35 49 53 55
168	30 31 32 41 53 55 55	28 32 32 44 50 54 58	29 30 31 39 51 54 57
192	32 34 34 43 55 58 58	31 35 37 47 52 56 59	33 32 34 45 53 57 59
216	33 34 37 45 57 59 62	32 36 39 48 52 58 59	34 33 37 49 54 60 60
240	32 36 38 47 58 59 62	32 36 40 50 53 59 61	33 34 39 52 56 62 63
264	33 36 39 48 60 62 63	32 37 41 52 54 60 63	34 36 39 52 57 63 65

288 3337 40 48 62 62 65 32 37 42 54 56 62 64 34 38 40 56 59 64 66

312 33 37 42 49 63 65 66 32 38 42 54 57 63 66 34 39 42 56 61 65 67

336 33 38 42 51 64 65 67 32 39 43 55 59 63 66 34 39 43 59 64 66 69

TABLE 3: Result of tensile test

S/NO	Wt % of bone powder	Tensile strength of UWC (MPa)	Tensile strength of HTW (MPa)	Tensile strength of HGW (MPa)
1	0	29.1520.1719.67		
2	5	31.1120.2123.65		
3	10	38.1625.2627.21		
4	15	39.2131.6530.31		
5	20	46.7633.2623.54		
6	25	51.2227.3324.16		
7	30	52.1021.4323.26		

TABLE 4: % Elongation at yield

S/NO	Wt % of bone powder	% Elongation of UWC (%)	% Elongation of HTW (%)	% Elongation of HGW (%)

1	0	13.9067.6
2	5	12.1075.5
3	10	10.7566
4	15	1044.9
5	20	7.654.2
6	25	644
7	30	5.7533.7

TABLE 5: Result of Flexural strength test

S/NO	Wt % of bone powder	Flexural strength of UWC (MPa)	Flexural strength of HTW (MPa)	Flexural strength of HGW (MPa)
1	0	47.139.2140.14		
2	5	51.2333.1139.13		
3	10	54.4135.1343.14		
4	15	55.1233.1245.23		
5	20	58.8133.9143.89		
6	25	66.8744.6649.01		
7	30	69.8150.2147.18		

TABLE 6: Result of Charpy Impact Energy test

S/NO	Wt % of bone powder	Impact Energy of UWC (J/m)	Impact Energy of HTW (J/m)	Impact Energy of HGW (J/m)

1	0	172.73121.21148.46
2	5	142.43136.06157.11
3	10	209.09127.29166.11
4	15	212.72130.3693.92
5	20	187.8866.6787.11
6	25	157.5837.3157.88
7	30	124.4263.6442.45

TABLE 7: Result of Shore D hardness test

S/NO	Wt % of bone powder	Hardness Value of UWC	Hardness Value of HTW	Hardness Value of HGW
1	0	74.57674		
2	5	767774		
3	10	798076		
4	15	8181.580		
5	20	818479		
6	25	83.58680		
7	30	88.5	8783.5	

TABLE 8: Percentage loss in weight after 336 hrsof Soil Burial

S/NO	Wt % of bone powder	Wt loss of UWC (%)	Wt loss of HTW (%)	Wt loss of HGW (%)
1	0	0.00 0.00	0.00	
2	5	0.000.000.00		
3	10	0.000.000.00		
4	15	0.00 0.000.00		
5	20	0.00 0.00	0.00	
6	25	0.000.000.00		
7	30	0.00 0.000.00		